ANNEX VII.  ION BEAM SYNTHESIS AND MODIFICATION OF SILICON CARBIDE

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VII-1. Scientific Scope of the Project

Silicon carbide (SiC) is a very promising candidate material that is only surpassed by diamond in performance for high-power microelectronic device applications. However, traditional fabrication methods cannot meet requirements of large scale, high purity, good crystalline quality and easy further processing for modern applications. Ion beam synthesis (IBS) of SiC is a new technique to respond to the challenge. High-fluence carbon implantation into silicon wafers in combination with subsequent high-energy ion beam annealing is a material-saving and high-efficiency method to form polycrystalline or epitaxial cubic SiC (β-SiC) layers in silicon. Multiple-energy carbon ions are implanted into silicon wafers at elevated temperatures to form a broad carbon-distributed amorphous layer. Subsequent high-temperature thermal annealing can recrystallize SiC but the required temperature may be as high as nearly 1,400°C which is industrially difficult or high costly. Swift heavy ion beam annealing (SHIBA) of C-implanted Si is an alternative for the annealing purpose to recrystallize Si-C. Because the process involves two effects contributing to the recrystallization, energy deposition induced temperature elevation and atomic recoiling motivated atomic mobility. The scientific scopes of the project are

— Conditions of C-ion implantation in Si to result in formation of crystalline Si-C
— Conditions of C-ion energy and fluence in multiple-energy ion implantation to form an applicable broad near-rectangular-shape implanted carbon layer
— Temperatures elevated during C-ion implantation to cooperate with subsequent high-energy ion beam bombardment for optimizing recrystallization of Si-C
— Conditions of swift (high-energy) heavy ion beam such as ion species, energy and fluence for recrystallization of Si-C
— Phases of primarily implanted carbon layers in Si substrate
— Concentration distribution or depth profile of primarily implanted carbon ions in Si substrate by multiple ion implantation
— Microstructures or phases after swift heavy ion beam annealing
— Model of the ion beam synthesis of Si-C
VII-2. Experiments


(1) **Samples:** Two-inch p-type silicon wafers with the resistivity of \(~30 \ \Omega\text{-cm}\) and a (100) surface.

(2) **Ion beam conditions:**
   - Energy: 80 keV; Fluence: \(2.7 \times 10^{17}\) ions/cm\(^2\); Beam current: \(2 - 5 \ \mu\text{A/cm}^2\); Temperature: room; Implanter: Varian ion implanter at Chiang Mai University.
   - Energy: 40 keV; Fluence: \(6.5 \times 10^{17}\) ions/cm\(^2\); Beam current: \(2 - 5 \ \mu\text{A/cm}^2\); Temperature: 400°C; Implanter: Danfysik ion implanter at Uppsala University.

(3) **Implantation direction:** Samples were oriented at 7° from normal.
(4) **Post-implantation treatment:** Si wafers were cut into pieces with a size of about \(1\times1\) cm\(^2\) and annealed in a vacuum furnace at temperatures of 800, 900 and 1000°C for 60 minutes, respectively.

VII-2.2. Multiple-energy Carbon Ion Implantation in Silicon

**Samples:** Two-inch p-type silicon wafers with the resistivity of \(~30 \ \Omega\text{-cm}\) and a (100) surface.

- **Ion beam conditions:**
  - Two energy sequences: 600-500-400-300 keV and 300-400-500-600 keV; Fluence for each energy: \(3.2 \times 10^{17}\) ions/cm\(^2\); Beam current: \(2 - 5 \ \mu\text{A/cm}^2\); Temperature: room; Implanter: 1.7 MV tandem accelerator at Chiang Mai University.

- **Implantation direction:** Samples were oriented at 7° from normal.

VII-2.3. Analysis of Carbon Depth Distributions

VII-2.3.1. Rutherford Backscattering Spectrometry (RBS)

- **Accelerator:** 1.7 MV tandem accelerator at Chiang Mai University.
- **Beam conditions:** Analyzing ion species: He\(^{++}\); Energy: 2.13 MeV; Beam current: \(~20 \ \text{nA}\).
- **Detection conditions:** Scattering angle: 160°; Detector: Si surface barrier detector; System resolution: 24 keV FWHM, corresponding to a depth resolution of some 60 nm in Si.
- **Conversion from energy spectrum to depth profile:** SIMNRA and self-developed programs

VII-2.3.2. Elastic Recoil Detection Analysis (ERDA)

- **Accelerator:** Tandem accelerator at the National Tandem Accelerator Facility in Uppsala, Sweden.
- **Beam conditions:** Analyzing ion species: \(^{127}\text{I}^{7+}\); Energy: 29 MeV.
- **Data acquisition and analysis system:** FAST ComTec GmbH.
— **Conversion from energy spectrum to depth profile**: Program CONTES.

**VII-2.4. Characterization**

— Infrared spectroscopy (IR)
— Raman scattering analysis
— RBS/Channeling analysis
— Glancing incidence X-ray diffraction (GIXRD)
— Transmission electron microscopy (TEM)

**VII-2.5. Swift Heavy Ion Beam Annealing (SHIBA) of C-ion Implanted Si**

**VII-2.5.1. I-ion beam annealing**

— **Facility**: 5-MV tandem accelerator at the National Tandem Accelerator Facility in Uppsala, Sweden.
— **Beam conditions**: Ion species: Iodine (multiply charged); Energy: 10, 20, 30 MeV; Fluence: 1, 2, 5 ×10¹² ions/cm²; Fluence rate: 1×10¹⁰ ions/cm²/s.
— **Temperature**: 90, 400, 800°C

**VII-2.5.2. Xe-ion beam annealing**

— **Facility**: 2-MV Van de Graaff ion accelerator at the Ion Beam Center, Surrey, UK.
— **Beam conditions**: Ion species: Xenon (Xe⁺⁺); Energy: 4 MeV; Fluence: 5×10¹³ ions/cm², 1×10¹⁴ ions/cm².
— **Temperature**: 500°C

**VII-2.6. Repairing of the Varian Ion Implanter**

The Varian ion implanter at FNRF would be the main working force for the project. However, the machine, model 200-DF5, is about-two-decade old and thus various technical problems often occur. After great efforts in fixing many problems in such as vacuum systems and electronic control systems, we were still unable to extract ions out of the source. IAEA sent Dr. Russell Gwilliam, Surrey Ion Beam Center, University of Surrey, UK as the IAEA Technical Cooperation Expert to us to carry out the mission of “To assist in the restoration of the Varian ion implanter”. Mr. Gwilliam worked at FNRF from November 20, 2006 to December 1, 2006. He succeeded in repairing the implanter. Before the last day of his work at FNRF, the machine finally worked. But, unfortunately at the last day some part in the ion source was suddenly burnt and within only half a day it could not be fixed.

**VII-2.7. Construction of a Sample Holder Heater System**

A halogen-lamp-based sample holder heater system has been constructed and completed. The system consists of a temperature controller, a halogen lamp, a transformer, a thermocouple, a magnetic switch, and a dimmer. We used an analog Digicon DG-8 temperature controller to
read and control the temperature up to 599°C. The lamp is a 250-W 24-V OSRAM halogen lamp that can heat the filament up to 500°C. A step-down 250-W 24-V transformer is used to supply the power to this lamp. A K-type thermocouple measures the temperature up to 500°C suitable for this system. The dimmer is used to increase or decrease the intensity of the lamp heating. The temperature controller is used for control the temperature required.

**VII-2.8. Preliminary Attempt to Investigate Luminescence of SiC Nanocrystallites**

Nanocrystalline SiC has been reported to emit pure blue or ultraviolet luminescence which is particularly useful for display. We have recently started some preliminary investigations on this issue.

— **Etching of the C-ion implanted Si:** The sample was Si implanted with C-ions at energy of 40 keV and fluence of $6.5 \times 10^{17}$ ions/cm². Chemical etching was done with dipping the sample in solutions of HF (50%) and KOH (50%), respectively; the former for etching SiO₂, while the latter for etching Si. The solution temperature was 80°C (boiling) with stirring. The etching time was varied from seconds to minutes.

— **Ionoluminescence (IL) analysis:** The analysis was done with the 1.7-MV tandem accelerator at FNRF using H⁻ at energy of 25 keV and current of 1 µA. The analyzing time duration was about 2 seconds.

— **Photoluminescence:** A commercial compact equipment of photoluminescence was used with varied input wavelength from 300 nm to 350 nm.

**VII-3. Results and Discussion**

**VII-3.1. Depth profiles of single-energy implanted carbon in Si**

— A fluence higher than about $6 \times 10^{17}$ ions/cm² is necessary for a stoichiometric implantation;

— Neither elevated-temperature ion implantation nor post-implantation thermal annealing broadens the C depth profile;

— High-fluence implanted C does almost not diffuse in Si, instead may already be combined with Si;

— Our self-developed RBS method works for analysis of light elements in heavy matrix.

**VII-3.2. Characteristics**

— Elevated-temperature C-ion implantation favors formation of SiC;

— Post-implantation thermal annealing favors SiC formation, and the higher the annealing temperature the better;

— The type is cubic silicon carbide, i.e. 3C-Si, or β-SiC, polycrystalline.

**VII-3.3. Swift heavy ion beam annealing (SHIBA)**

— Swift heavy ion beam annealing (SHIBA) works, resulting similarly to thermal annealing;
The higher the ion fluence and energy (in certain ranges), the better;

Advantages of ion beam annealing compared with thermal annealing:

- Lower temperature (n×100°C compared with ~1000°C)
- Shorter treatment time (2 min compared with 1 hour)
- Easier recrystallization
- No Si-oxide top layer;
- Conditions to be optimized;
- Mechanism involved should be electronic stopping.

VII-3.4. Multiple-energy C-ion implantation in Si

- Multiple-energy C-ion implantation broadens the carbon profile.
- For the energy reducing sequence, the carbon depth profile is considerably broader than that from the energy increasing sequence, which is well agreed with the program-simulated profile. It is a puzzle. Anyway, this result is indeed informative for us to obtain a broad SiC layer buried in Si.

VII-3.5. Luminescence

- Direct observation of luminescence is impossible, as SiC in the sample is covered by SiO₂ and Si.
- We have not yet succeeded in removing the top layers by simply dipping in the chemical solutions. The electrochemical etching is suggested to be required for the purpose. Ion beam sputtering can be also a choice.
- Photoluminescence experiment was not yet successful. No expected luminescence was observed at the expected wavelength. Reasons might be lack of skill and experience in operation of the photoluminescence equipment or others, probably from the material itself.

VII-4. Overall Conclusions

- The ion beam synthesis technique developed, i.e. high-fluence C-ion implantation in Si wafer at elevated temperature followed by vacuum thermal annealing or swift heavy ion beam annealing, can form buried high-quality polycrystalline nano-grained β-SiC layer in Si wafer.
- Swift heavy ion beams may act as favorable annealing of SiC, governed by electronic stopping process, and the effect depends on ion energy and fluence.
- Neither elevated-temperature ion implantation nor post-implantation thermal annealing can broaden the implanted high-concentration carbon profile.
- Multiple-energy C-ion implantation in Si in the energy sequence from high to low energy favors to obtaining a broad buried SiC layer.
- Certain methods are needed to expose the ion-beam-synthesized SiC nanocrystallites for luminescence.