Structural study of metal-SiC, -Si, contact using soft X-rays: PEEM and SXES

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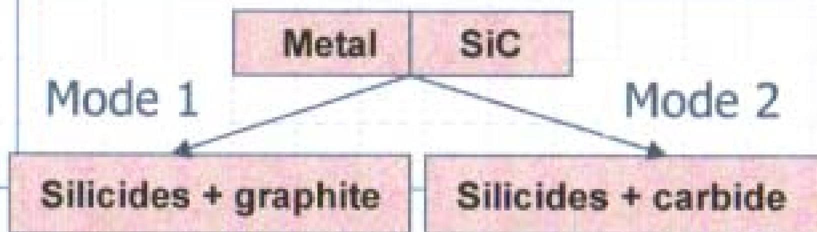
- ***Introduction***
- ***Objective of the study***
- ***PEEM study***
- ***SXES study***
- ***Conclusions of the study***

1. Introduction:

Transition Metal/SiC systems



- **Silicon Carbide (SiC) features:**
  - ***Wide energy bandgap***
  - ***High breakdown electric field***
  - ***High saturated electron drift velocity***
  - ***High thermal conductivity***

Ti/SiC system (Mode 2)

- *Ohmic and Schottky contacts*
- *Ti-silicides*
  - *Lower contact resistance*
- *Ti-carbide-*
  - *Improves Ohmic behavior*

- ***Most characterizations on this system in destructive methods***
  - ***TEM, Ar- sputtered depth profiling***
- ***No SXES and PEEM studies yet reported on Ti/SiC***

Objective of the study:

- ***To conduct a non-destructive spectro-microscopy characterization on the surface morphology and interface structure of the heat-treated Ti(film)/SiC(substrate) by***
  - ***Photoemission electron microscopy (PEEM)***
  - ***Soft X-ray emission spectroscopy (SXES)***

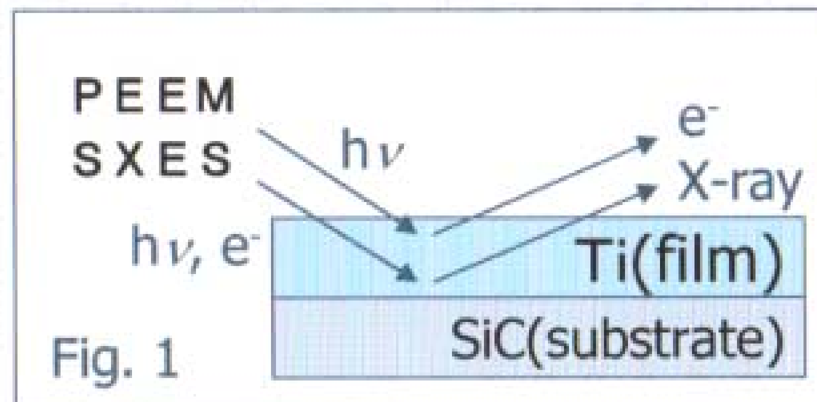


Fig. 1

2. Photoemission electron microscopy (PEEM)

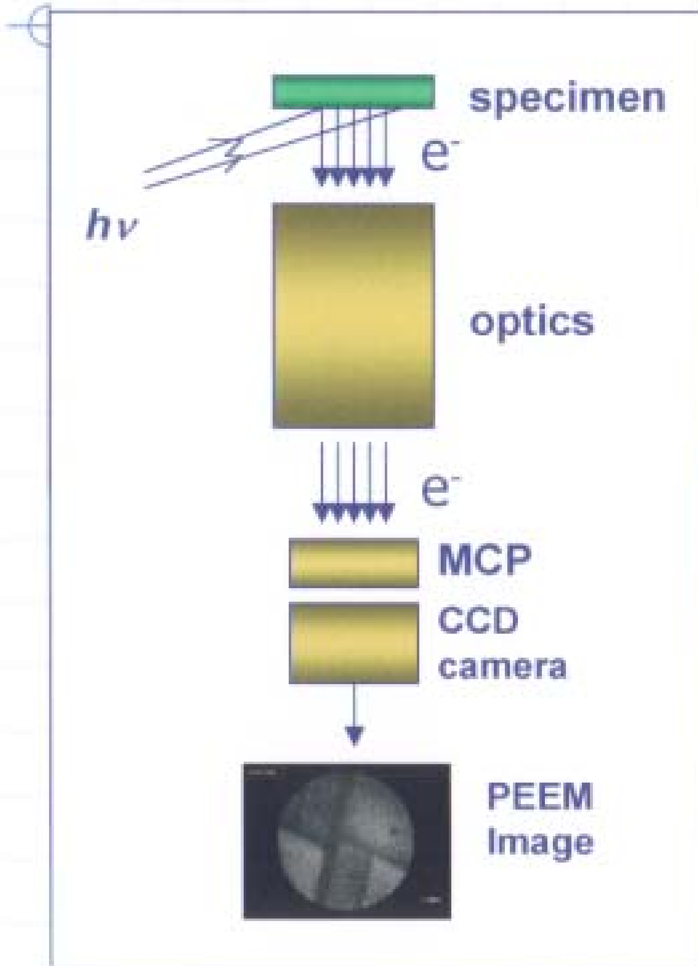


Fig. 2

PEEM image is based on the lateral photoemission intensity distribution from a solid sample surface.

i. Contrast mechanisms in PEEM

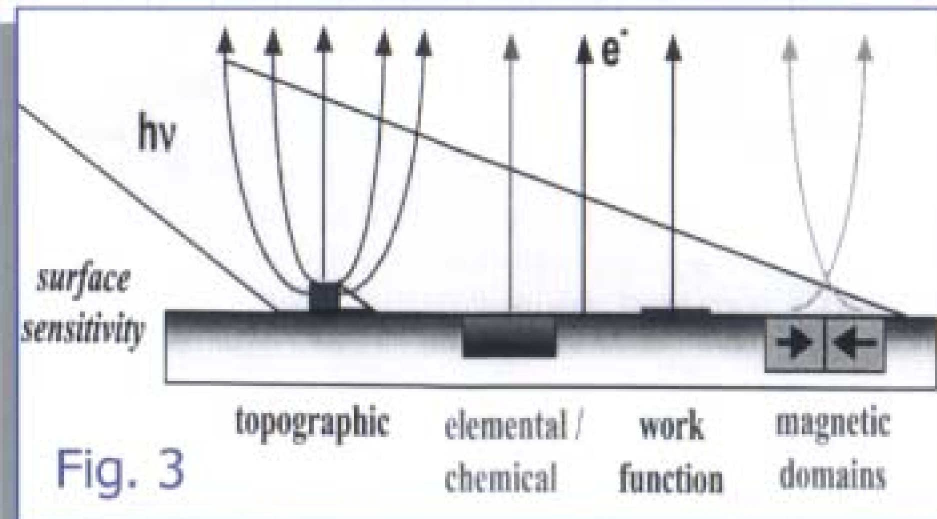
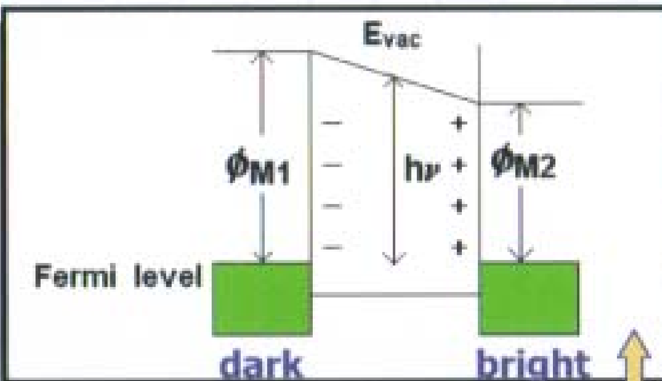


Fig. 3

- ***Real-time observation***
  - ***In-situ annealing***
  - ***Surface oxidation***
  - ***Chemical element mapping***
  - ***Chemical state mapping***

ii. Contrast mechanisms in PEEM utilized in this study:

Fig. 4. Contrast due to work function difference; ($\phi_{M1} > \phi_{M2}$)



For ($\phi_{M1} > h\nu > \phi_{M2}$), electrons will be photo-emitted from region M_2 (bright) but not from M_1 (dark).

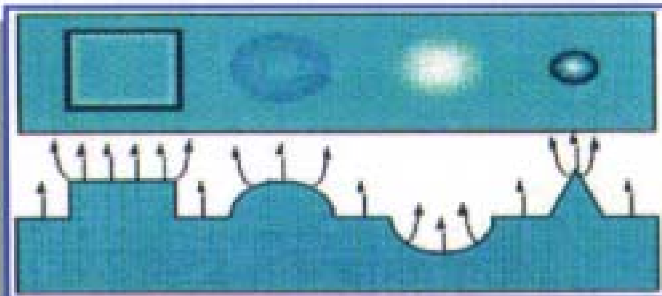
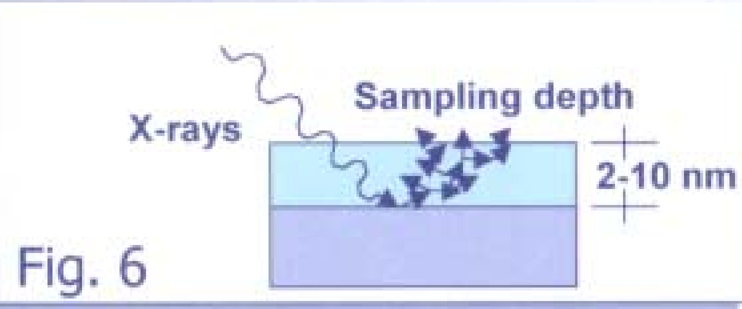


Fig. 5 Topographical contrast

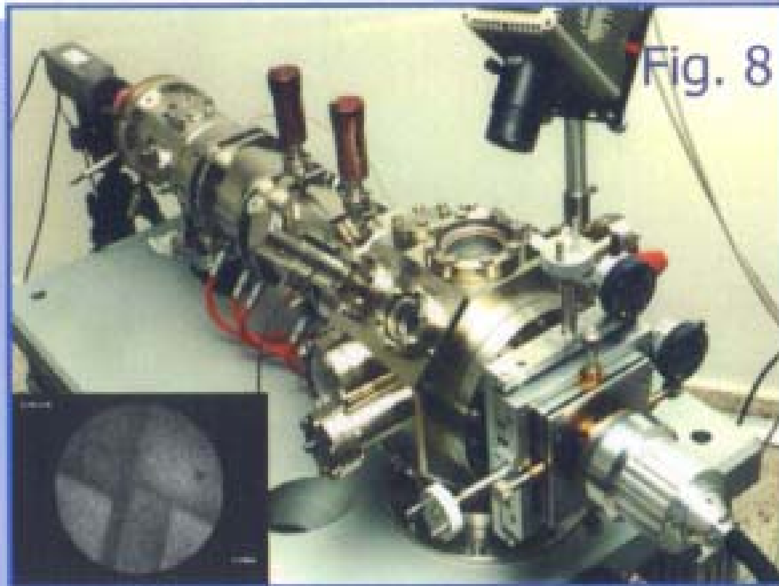


(A) Hg lamp *(B) 90 eV photons (dark Si surface)* *(C) 130 eV photons (bright Si surface)*

Fig. 7 PEEM images of Ti/Si(100); Imaging by varying the photon energy:

Using 130-eV photons, which is slightly above Si $L_{2,3}$ electron, Si-rich surface would have a higher photo-electron yield. Thus, Si surface then would appear brighter.

iii. PEEM microscope in HiSOR:



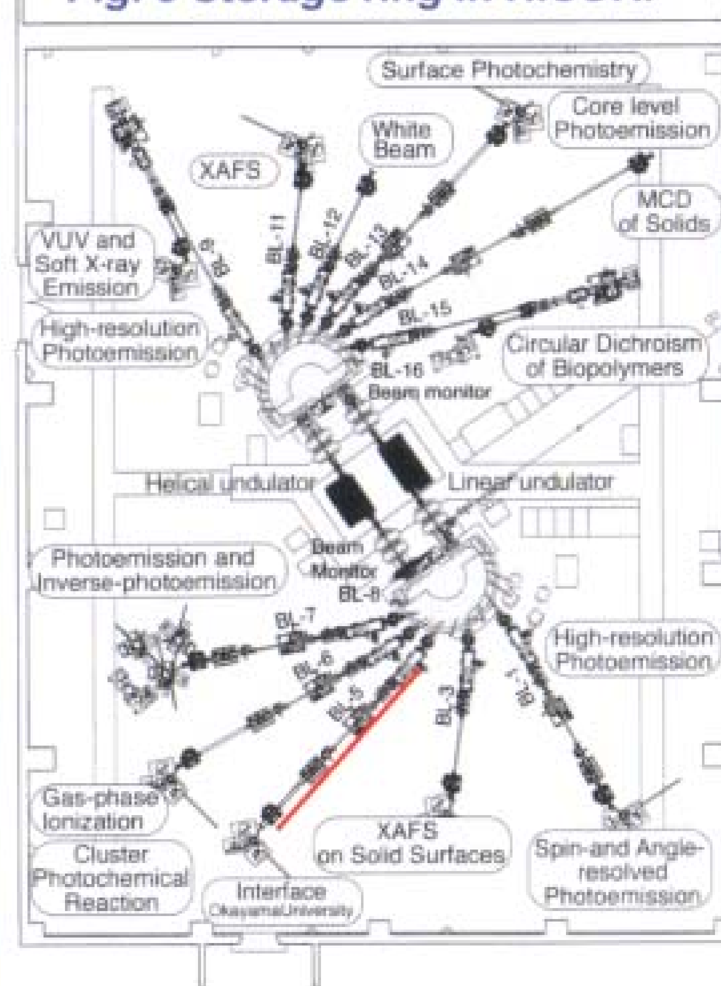


Fig. 9 Storage ring in HiSOR.

i.) ELMITEC PEEM III system

ii.) Lateral resolution: 15 nm

iii.) Field of View: 2-150 μm

iv) Sample temperature: up to 1500°C

v.) Light sources: Hg lamp and synchrotron radiation

vi.) Attached to BL-5, HiSOR

iv. PEEM images of Ti film on Si(100):

- *Ti film: 40nm*
- *Work functions:*
  - *Ti(4.33eV)<$TiSi_2$(4.53eV)<*
  - *hν(5.1eV)<Si(5.2eV)*
- *Phase transition at ~700°C:*
  - *Si diffuses to Ti layer and C49 phase of $TiSi_2$ is nucleated*
- *Formation of nano-structures (white dots) at 700°C*
- *Field of view (FOV) = 50 μm*

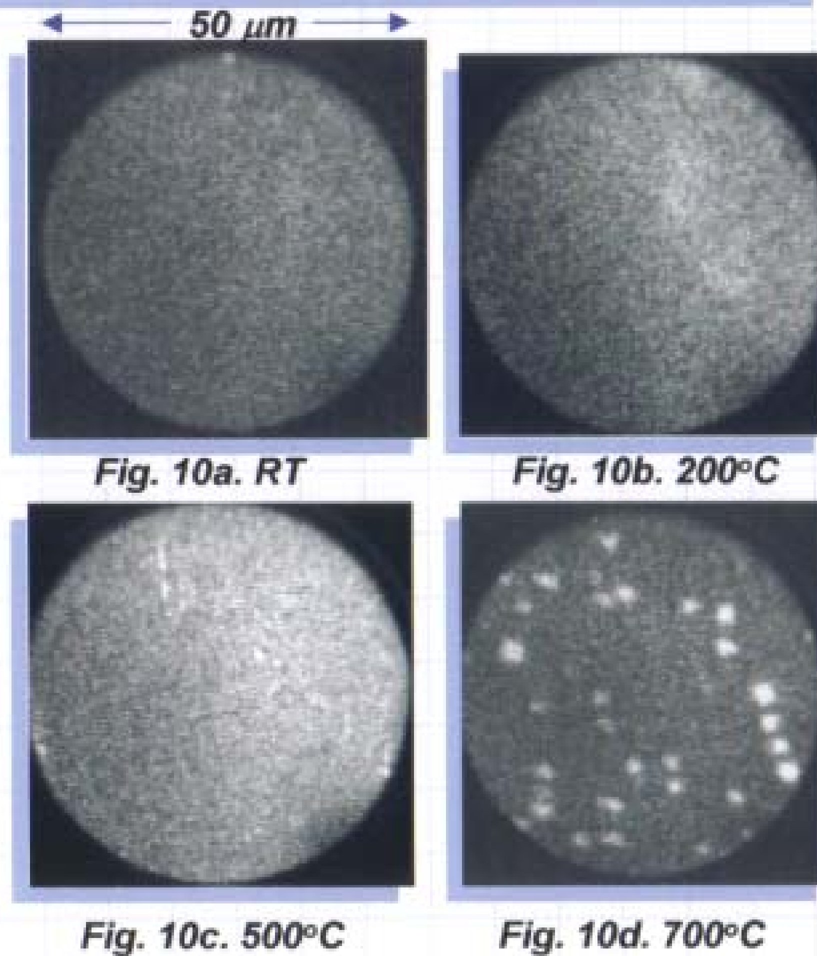


Fig. 10a. RT

Fig. 10b. 200°C

Fig. 10c. 500°C

Fig. 10d. 700°C

v. PEEM images of Ti(50nm) film on 4H-SiC: FOV = 150 μm

- *Ti film: 50nm*
- *Light source: Hg lamp*

- *Dark image at RT due to an oxydized Ti surface*
- *Brightening of surface at 550°C due to oxygen desorption.*
- *Formations of some white island-like structures at 850°C.*

Fig. 11a. RT

Fig. 11b. 550°C

Fig. 11c. 650°C

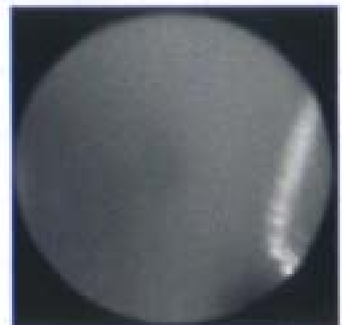


Fig. 11d. 700°C

Fig. 11e. 800°C

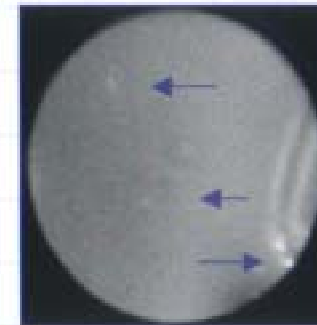


Fig.11f. 850°C

vi. PEEM images of Ti(40nm) square film on 3C-SiC: FOV = 150μm

- *Ti film: 40 nm in deposited in square shapes (600 μm x 600 μm in sizes)*
- *Light sources: Hg lamp and 130-eV photons from SR*

- *Dark image at RT due to oxidized Ti surface.*
- *Formations of some white island-like structures at 850°C.*
- *Four different layers are observed at the edges of 40 nm thick Ti squares on 3C-SiC/Si(100) after annealing to 850°C, with the topmost layer composed of unreacted Ti and Ti-silicides.*

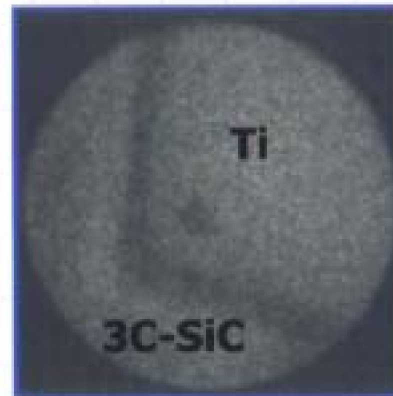


Fig. 12a. RT, Hg lamp

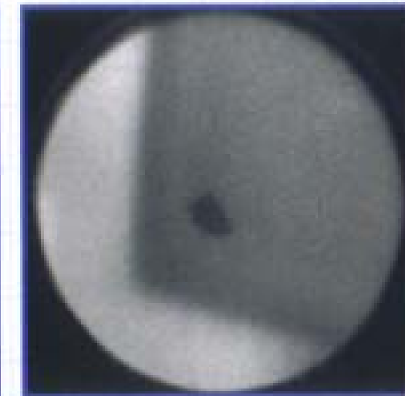


Fig. 12b. RT, 130 eV photons

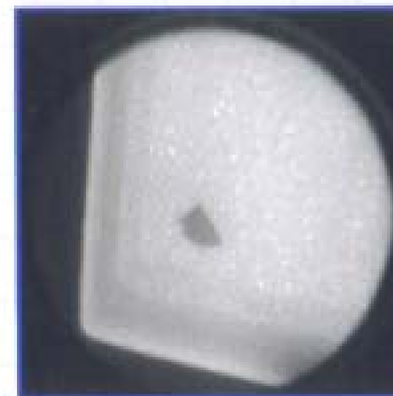


Fig. 12c. 850°C, Hg lamp

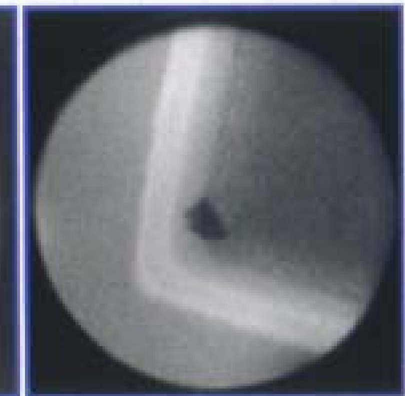


Fig. 12d. 850°C, 130 eV photons

vii. PEEM images of 40 nm Ti square film on 3C-SiC: FOV=50μm

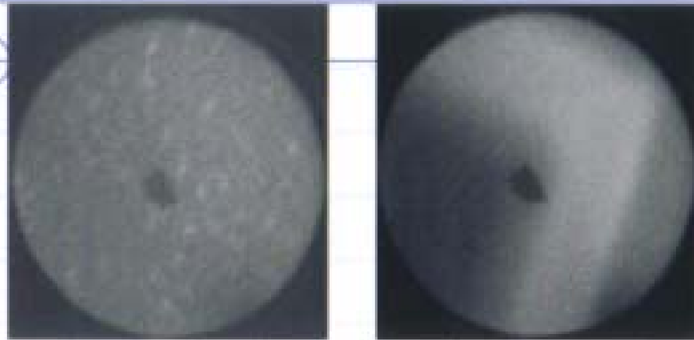


Fig. 13a. Topmost surface, Hg lamp **Fig. 13b. Corner, 130 eV-photons**

viii. Schematic of PEEM images of Ti(40nm) square-patterned film on 3C-SiC:

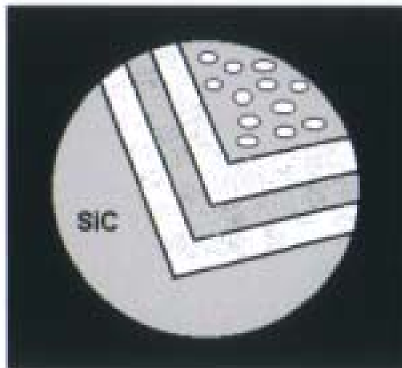


Fig. 14a. Top view

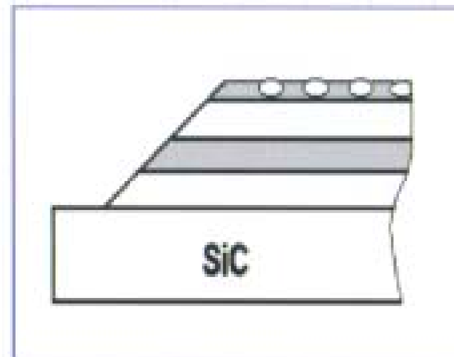


Fig. 14b. Side view

ix. AES of Ti(5 nm)/3C-SiC/Si(100)

- **Annealing condition: 600-900°C, 5 min in UHV**

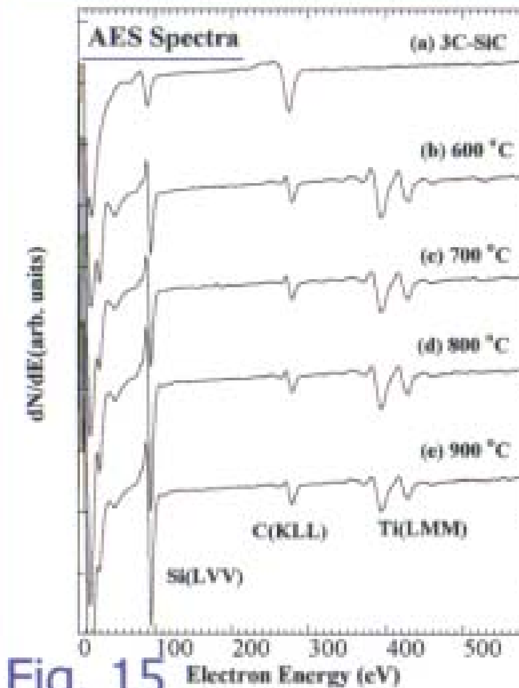


Fig. 15

- ***Significant increase*** ***in Si LVV/C KLL peak-to-peak height ratio after annealing.***
- ***The surface becomes Si-rich*** ***after high thermal annealing.***

3. Soft X-ray emission spectroscopy (SXES)

- ***Non destructive bulk and buried layer characterization technique***
- ***SXES spectra reflects valence band density of states***
  - ***Si $L_{2,3}$ emissions***
    - ***Si s and d states***
  - ***C K emissions***
    - ***C p states***
- ***Formed layer in interface is analyzed by the changes in SXES spectra.***
- ***Soft X-rays induced by***
  - ***Synchrotron radiation (selective fluorescence excitation)***
  - ***High energy electron (electron excitation)***

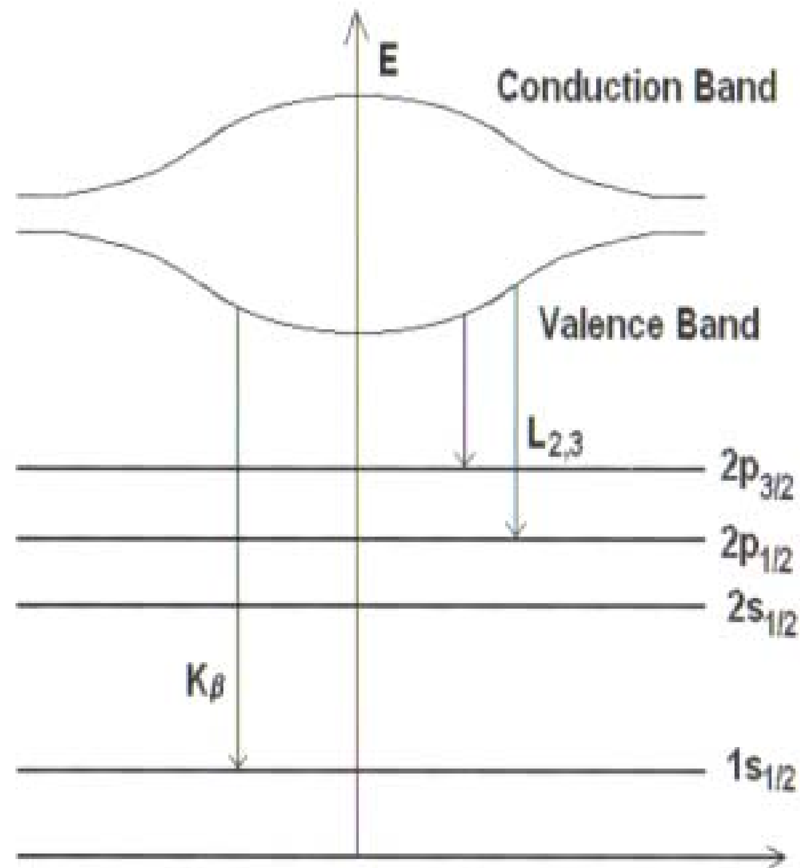


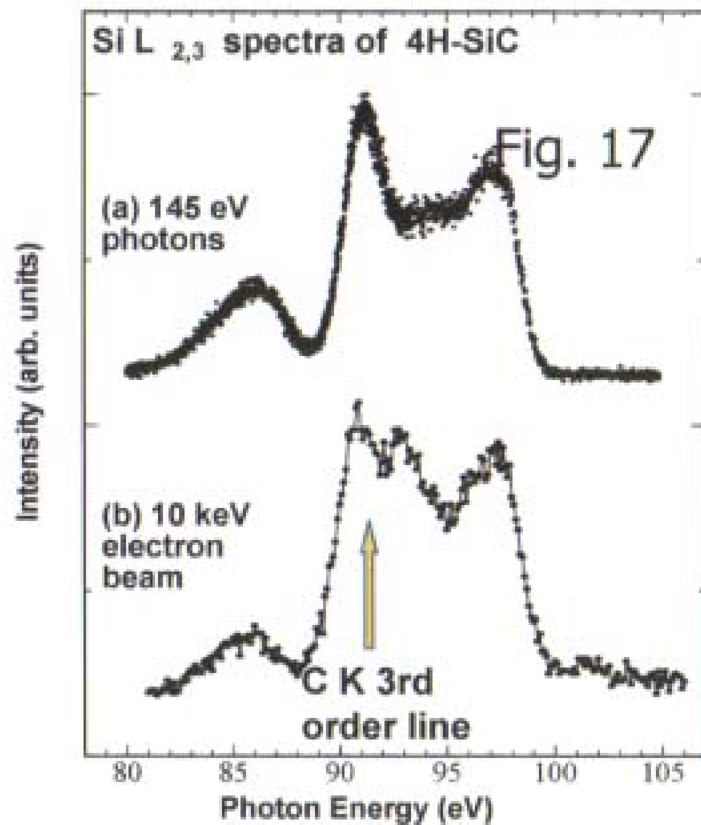
Fig. 16

X-ray intensity:
$I(E) \sim N(E)P(E)$.

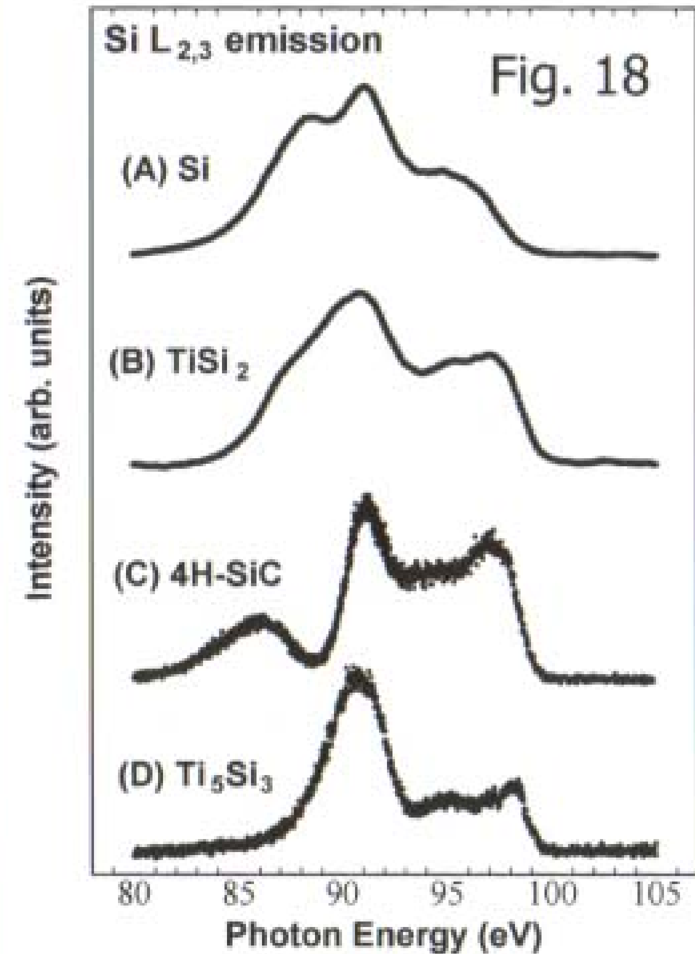
Selection rule:
$\Delta l = \pm 1; \Delta j = 0 \text{ or } \pm 1$

a. Selective fluorescence excitation

- ***No overlapping of Si $L_{2,3}$ and 3rd order diffraction line of C:***
  - ***Si $L_{2,3}$: 145 eV photons***



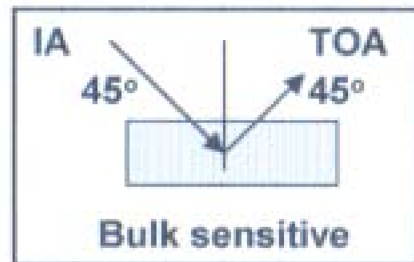
b. Si $L_{2,3}$ emission spectra of different species



c. Soft X-ray fluorescence spectra of Ti(50nm)/4H-SiC(0001)Si-face

- ***Annealing condition: <u>800-1000°C</u>, 5 min in UHV***
- ***Si $L_{2,3}$ induced by 145 eV photons at BL-19B, Photon Factory, KEK***

50nm Ti
SiC(0001)



- *Lowering of peak intensities at 86 eV.*
- *Lowering of terrace from 93-98 eV.*
- *No peak at 86 eV of 1000°C annealed sample*

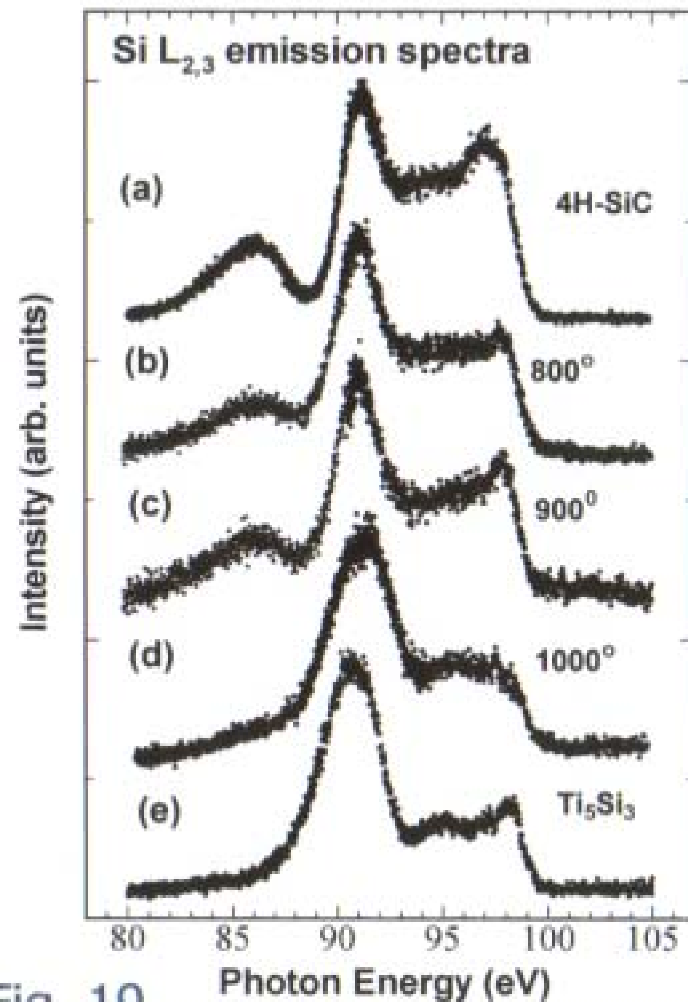


Fig. 19

d. Synthesized Si $L_{2,3}$ spectra from 4H-SiC and Ti_5Si_3.

Synthesized Spectra		
Sample temp.	Ti_5Si_3	4H-SiC
A. 800°C	~40%	~60%
B. 900°C	~45%	~55%

i.) Synthesized spectra are overlapped on the obtained spectra.

ii.) Synthesized spectra generated from Ti_5Si_3 and 4H-SiC spectra in the ratio of

(sample A-800°C)- 0.40:0.60

(sample B-900°C)- 0.45:0.55

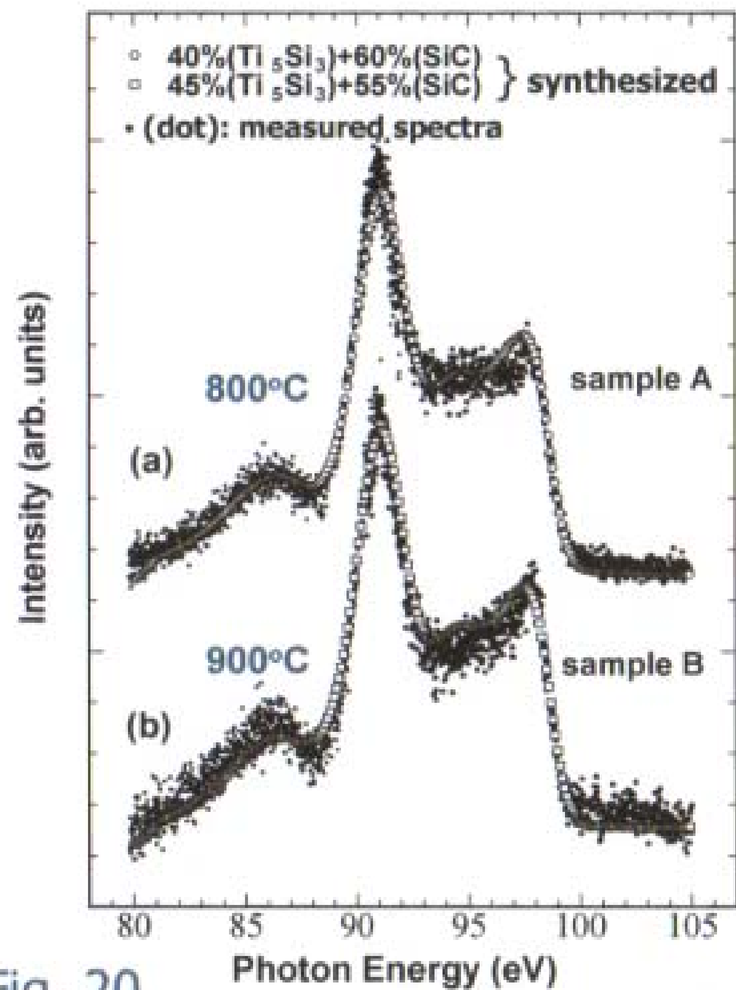
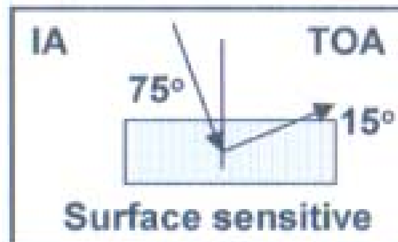


Fig. 20

e. C K fluorescence spectra of Ti(50nm)/4H-SiC(0001)Si-face

- ***Annealing condition: 800-1000°C, 5 min in UHV***
- ***C K induced by 350 eV photons***



I.) 800°C: (a) Existence of free C atoms within the Ti_5Si_3-TiC reacted region, (b) the occurrence of TiC in amorphous state, (c) and/or TiC with dissolved Si atoms.

II.) 900°C: (a) Mixture of TiC and 4H-SiC spectra.

III.) 1000°C: (a) High crystalline TiC and/or (b) TiC with C atoms in ternary silicide bonding (possibly Ti_3SiC_2).

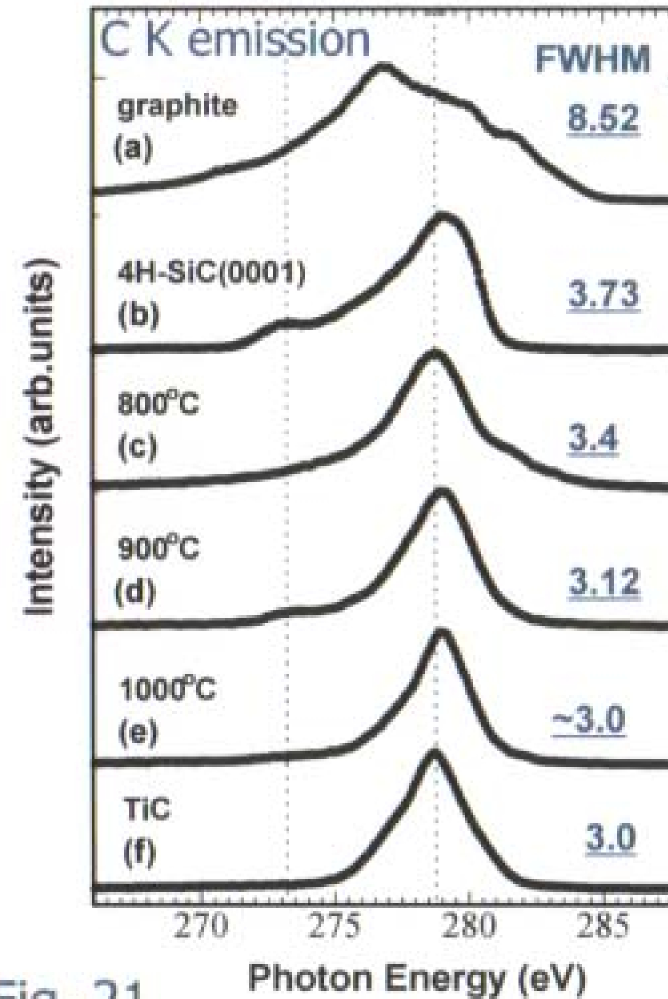


Fig. 21

f. Reacted layer in Ti(50nm)/4H-SiC(Si-face)

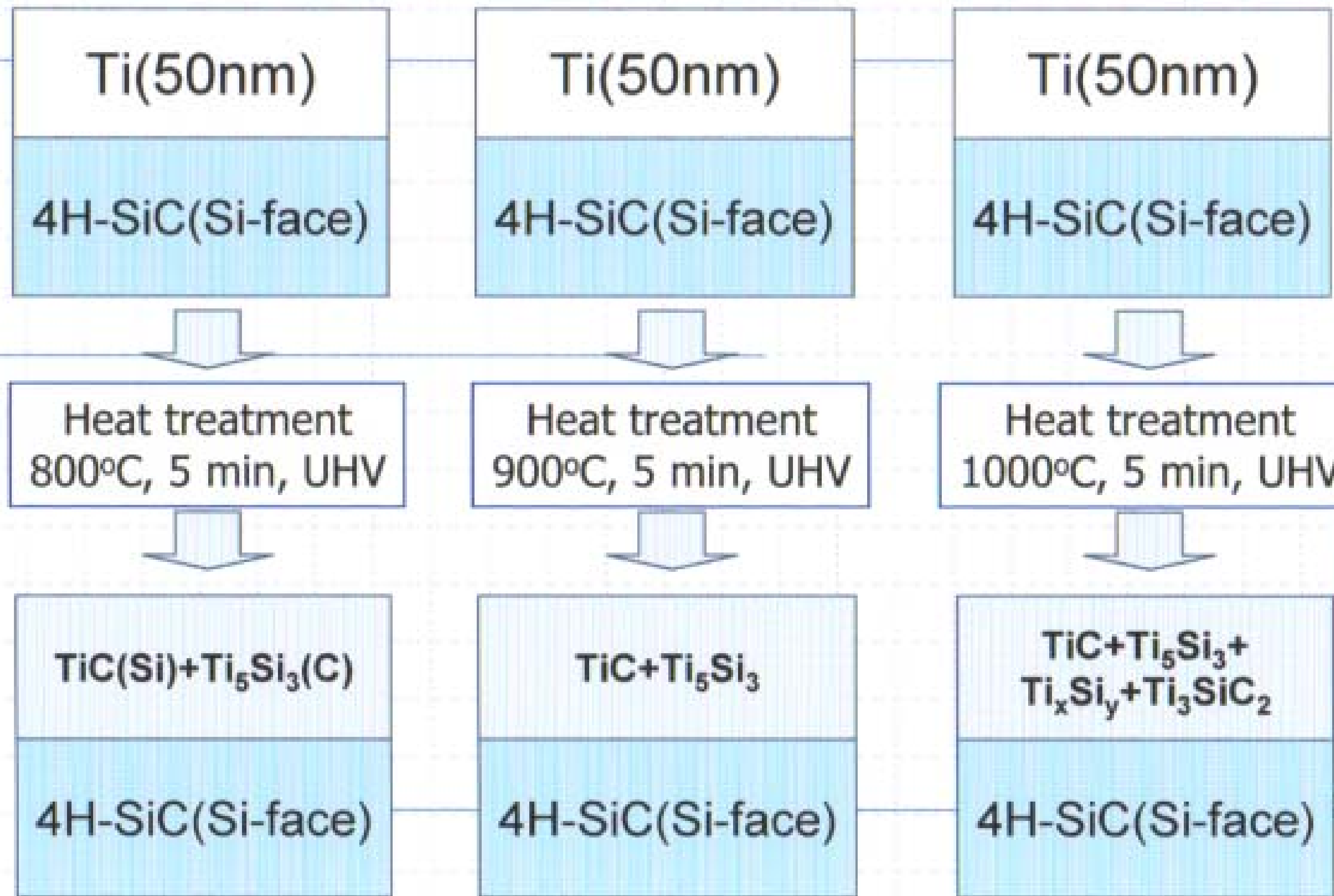


Fig. 22

4. Conclusions on PEEM and SXES study of Ti(film)/SiC:

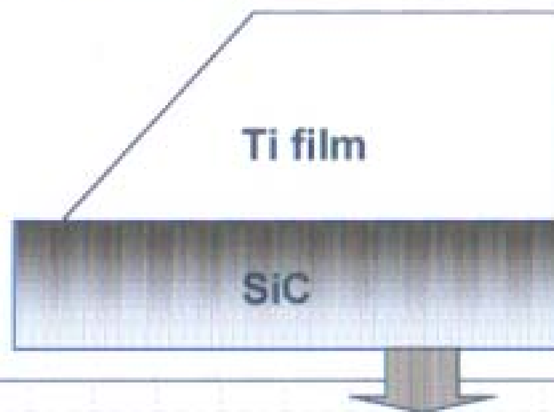


Fig. 23a

Heat treatment

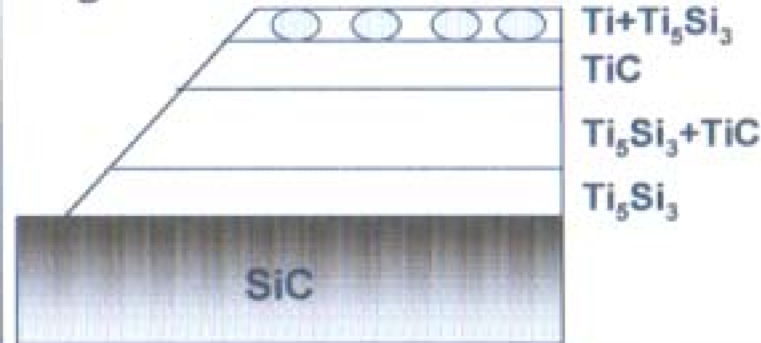


Fig. 23b

1.) SXES and XRD characterizations on the interface of Ti/SiC reveal the formations of Ti_5Si_3 and TiC as the majority formed species in the reacted zone.

2.) Island-like structures were observed on Ti(50nm)/4H-SiC(0001) were at~ 850°C.

3.) Four-layer gray regions were observed on edges of the 40 nm thick Ti square film on 3C-SiC/Si(100) after annealing to 850°C, with the topmost layer composed of unreacted Ti metal and Ti-silicides(in island-like structures).