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Ion beam assisted synthesis of nanoprecipitates by dual beam irradiation

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Motivations

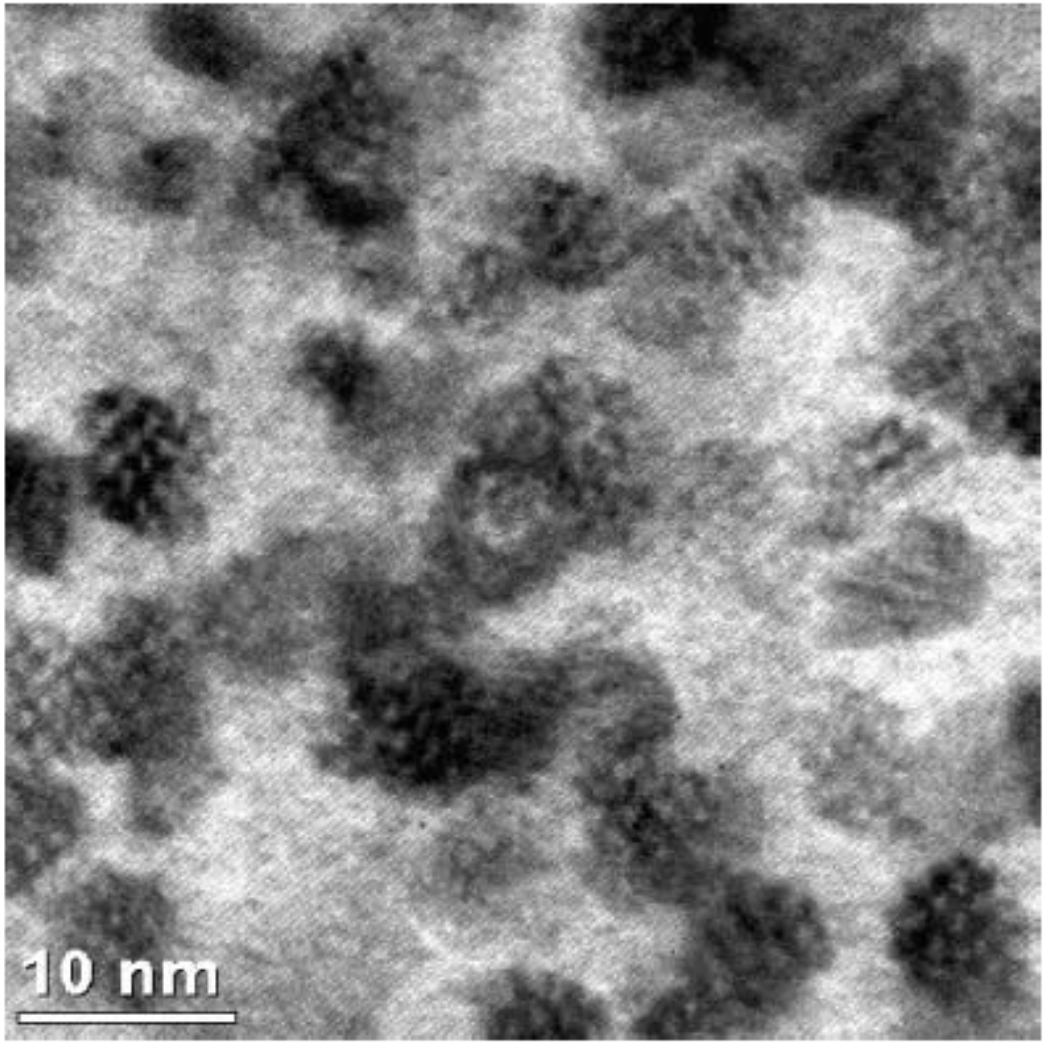
- **From a fundamental point of view: To improve our understanding of the growth mechanisms of nanophases assisted by ion beam (co)irradiation**

- **From an application point of view: Ceramic nanoprecipitate dispersions in metallic matrices are known to enhance both the mechanical properties and the radiation tolerance**

Two pionneering works

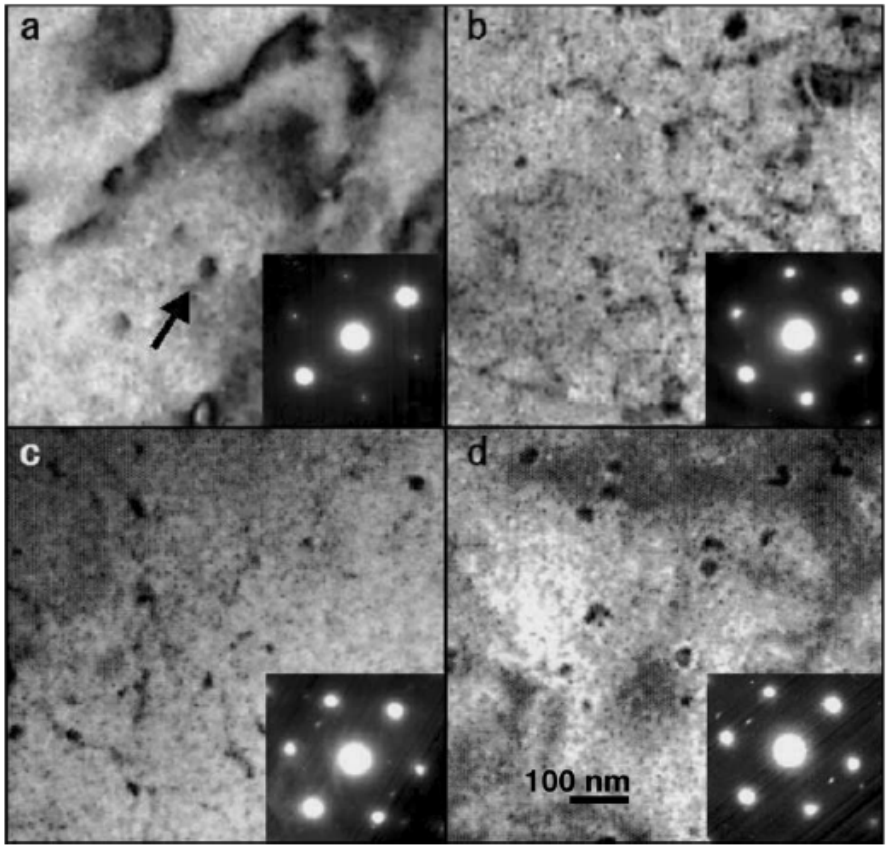
- R. Kögler, Appl. Phys. A76, 2003, 827 and Nucl. Instrum. Meth. Phys. Res. B206, 2003, 989
- SiC in Si by 360 keV C^+ and 1.5 MeV Si^+ co-implantation at 450 C
- 8.41 x 10^{16} C/cm^2 and 2 x 10^{16} Si/cm^2
- AES, XRD and TEM

- D. Sakuma, J. Nucl. Mater. 329-333, 2004, 392
- Y_2O_3 in Fe alloy by 400 keV Y^+ and 83 keV O^+ co-implantation
- 1.0 x 10^{16} Y/cm^2 and 1.5 x 10^{16} O/cm^2
- Post-annealing 300 – 1300K
- TEM

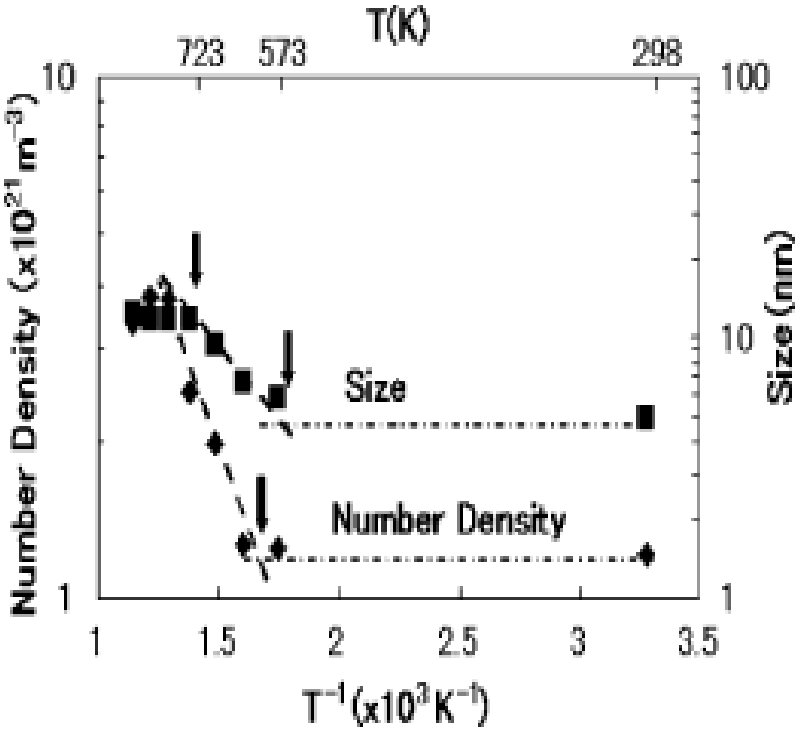


R. Kögler

HREM image of the depth position x = 0.75 µm (C range above the first damage layer) of sample C + SiHE, showing a high density of SiC precipitates with a Moiré pattern



Nano-oxide particle structure during in situ annealing: a) before annealing, b) annealed at 573 K, c) 623 K and d) 673 K.



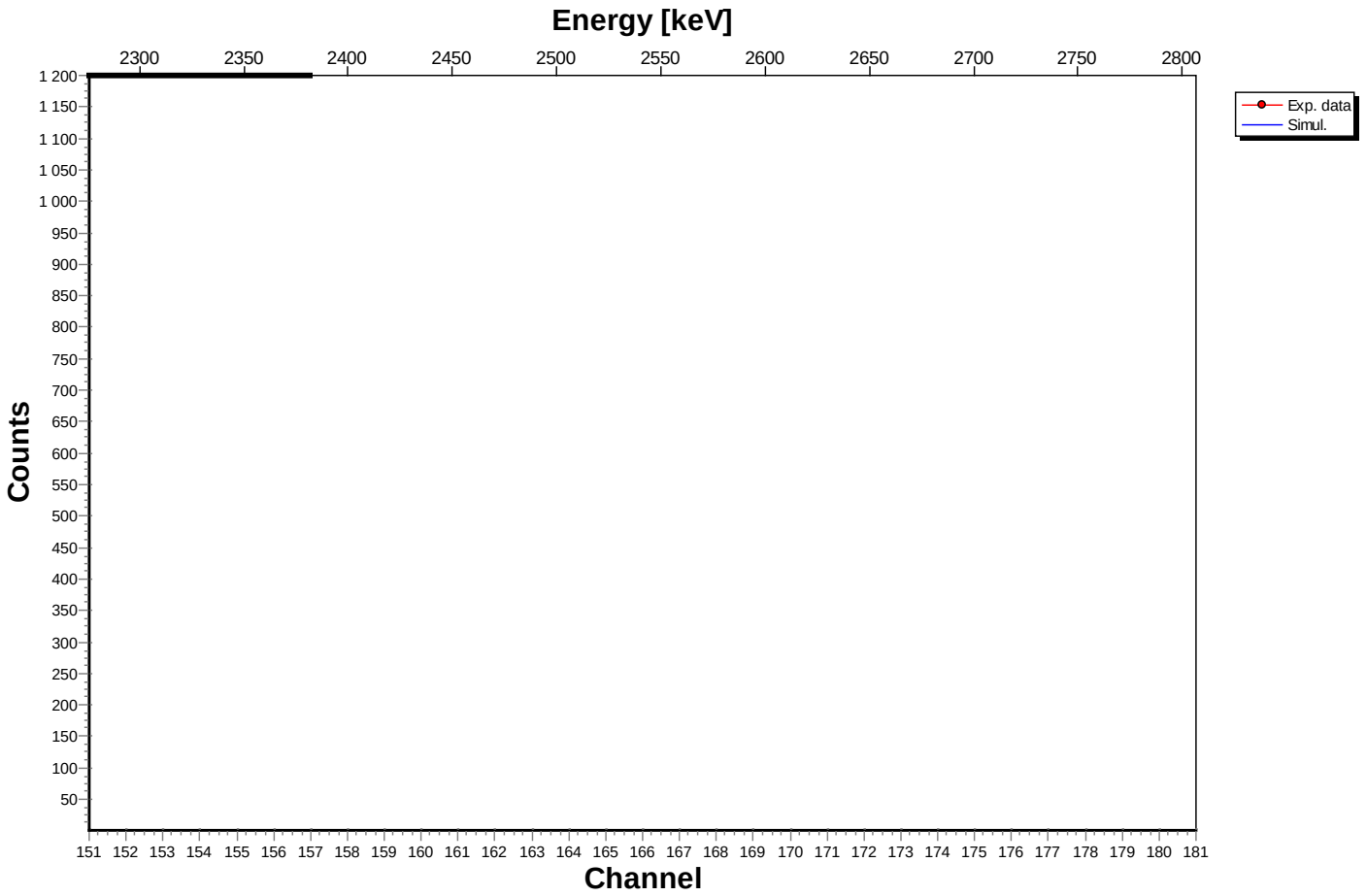
The particle number density and average size for thin specimens as a function of the in situ Temperature.

D. Sakuma

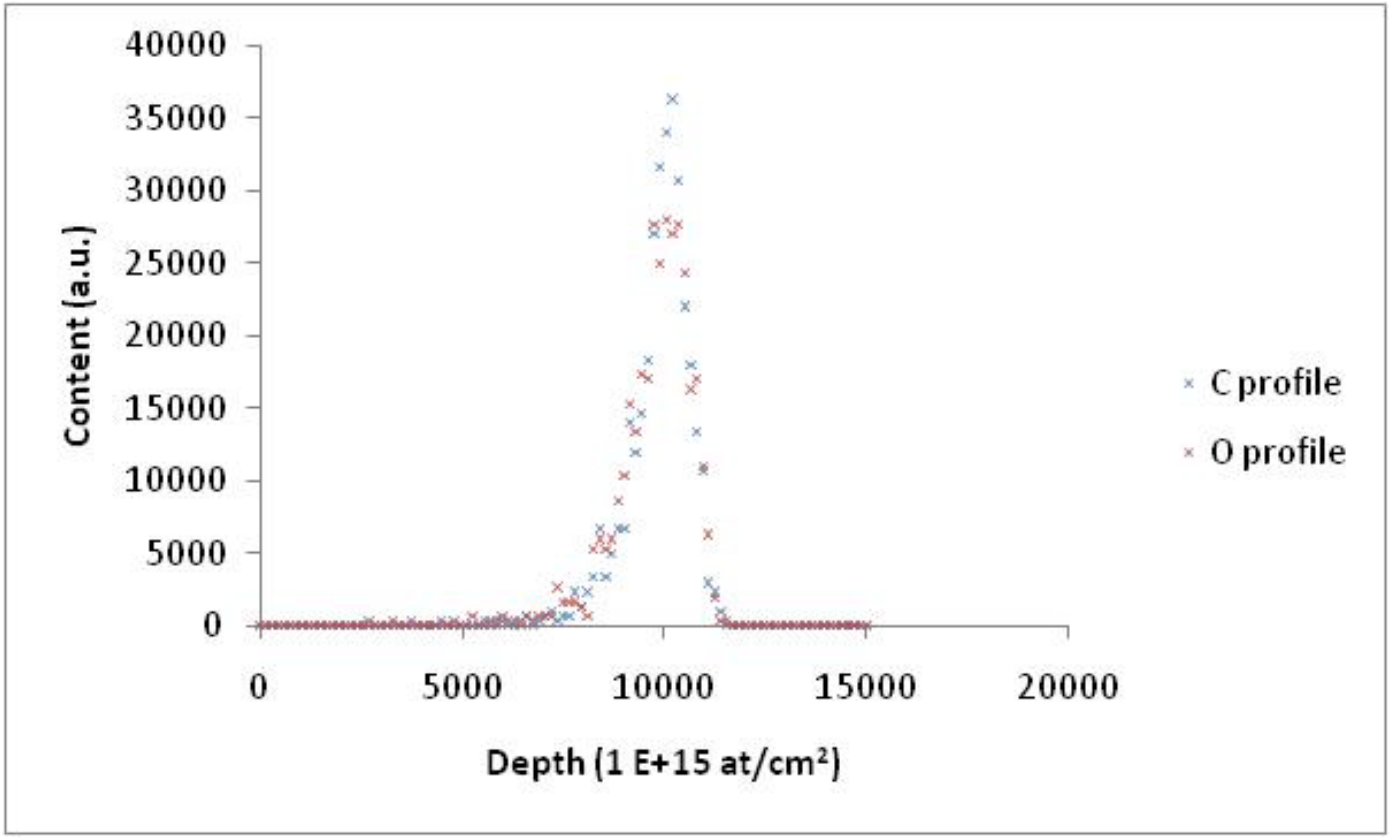
This work

- **SiC in Si (100) by 1.5 MeV C^+ (Épiméthée) and 2.5 MeV Si^+ (Japet) co-implantation and SiO_2 in Si(100) by 1.7 MeV C^+ (Épiméthée) and 2.5 MeV Si^+ (Japet) co-implantation at RT**
- **3.0 x 10^{16} C or Si/cm^2 – 2.5 x 10^{16} Si/cm^2 – 5.0 x 10^{16} O/cm^2**
- **Post annealing at 1000 C for 3 hours under Ar**
- **Characterization task: XRD, NRA and Raman microspectrometry**

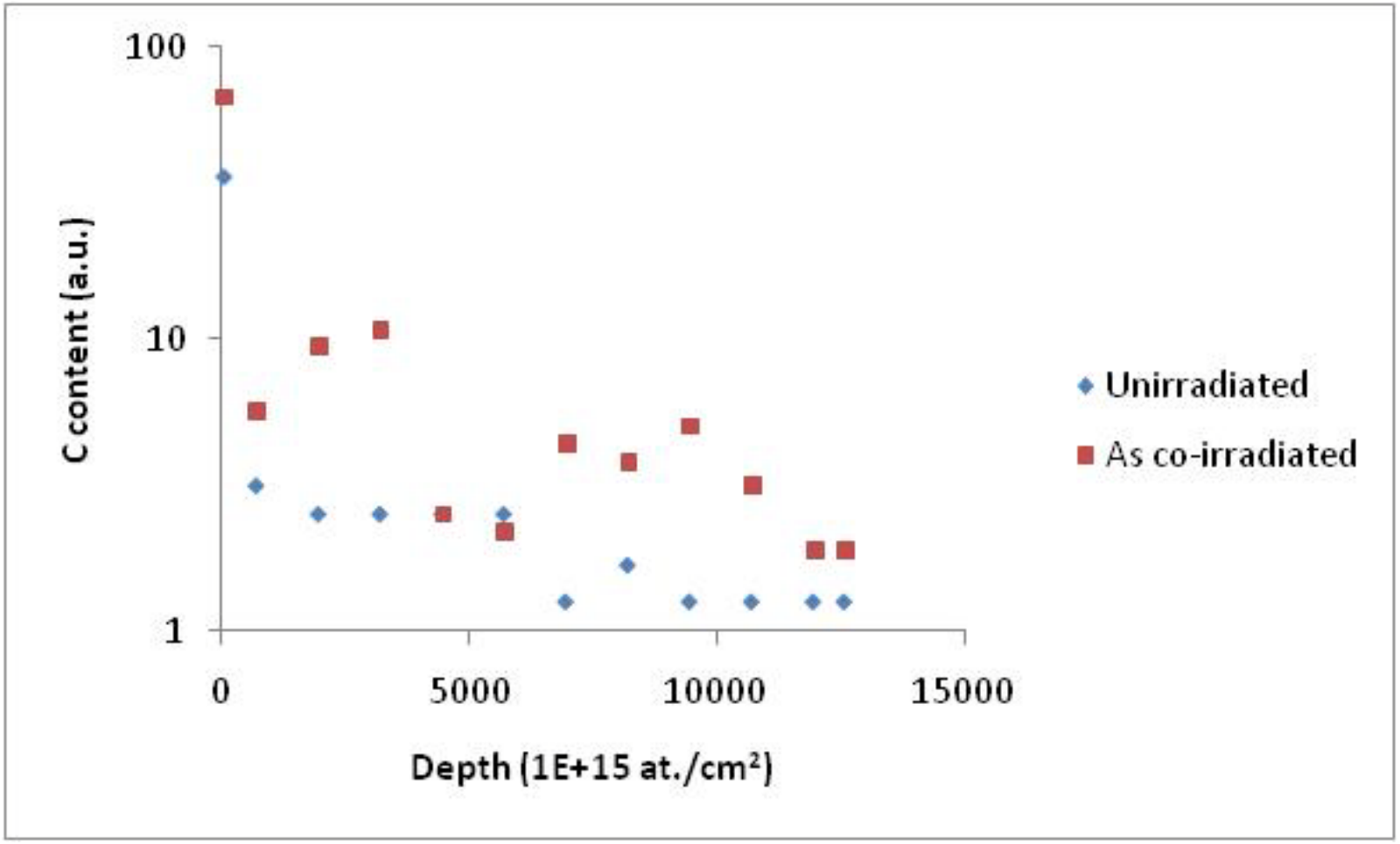
First results (1): NRA



Example of SIMNRA fitting: Si + C co-irradiated Si before annealing



Theoretical C and O depth profiles extracted from SRIM calculations



Experimental C depth profiles extracted from SIMNRA fitting

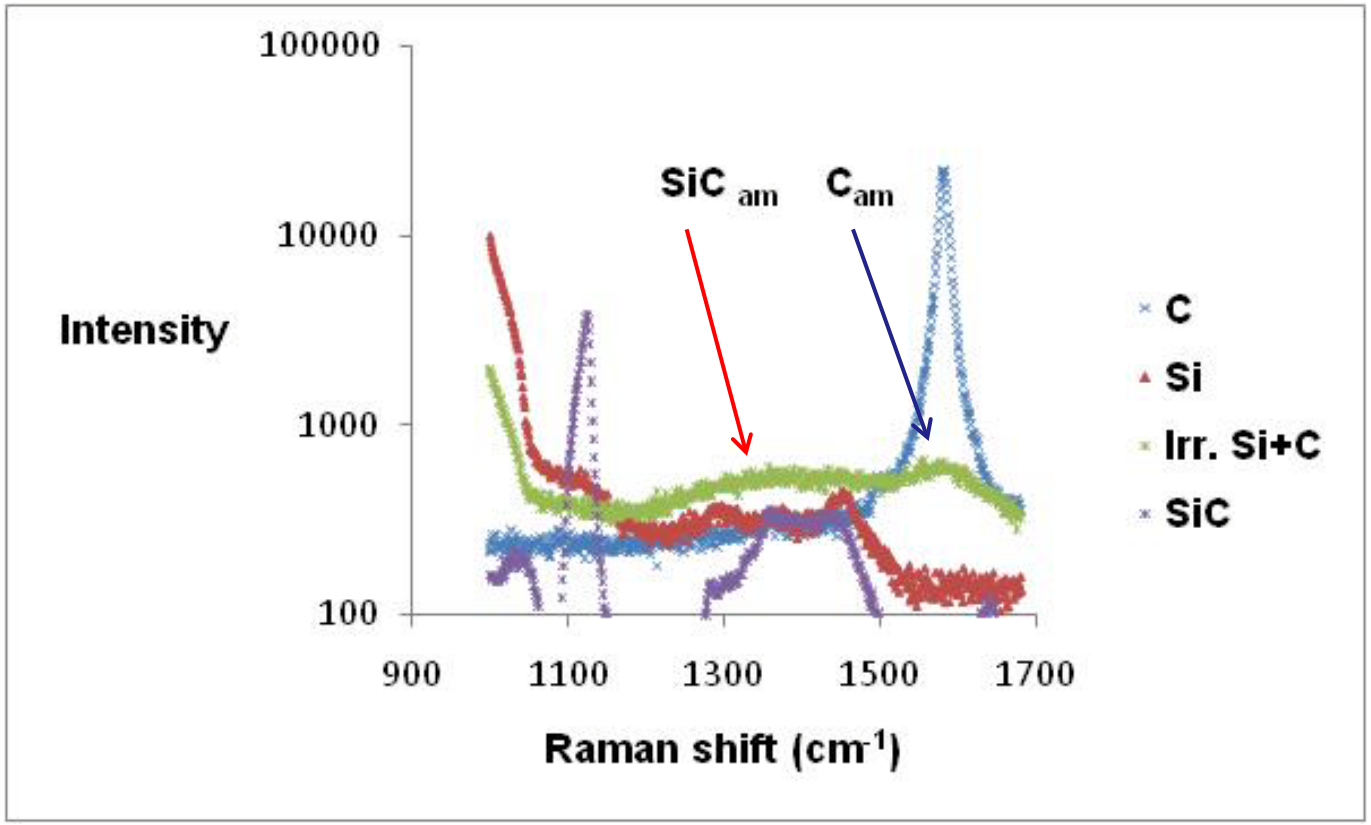
Main observations

- **Carbon atoms (contamination) present on the surface of Si(100) have been incorporated within the substrate**

- **The carbon depth profile obtained before annealing (SIMNRA fitting) is much more larger than the predicted one (SRIM calculation)**

- **Concerning the Si + O co-irradiated substrate, a redistribution of O atoms also occurs during the co-irradiation**

First results (2): Raman microspectrometry



Main observations

- **The characteristic Raman bands for SiC_{am} and C_{am} are detected on the Si + C co-irradiated Si (100) substrate**

- **Comparing these data with those from pure graphite, polycrystalline SiC and virgin Si (100) substrate, we can assess the formation of both amorphous SiC and C within Si after the co-irradiation ($|\Delta G_f SiC| \sim 0.64$ eV)**

- **In the case of the Si + O co-irradiation, the Si – O Raman bands are not visible on the spectrum registered just before annealing ($|\Delta G_f SiO_2| \sim 8.91$ eV)**

What is still to be performed?

- **Co-irradiation Si + C:**

  ☺ Post-annealing characterization by NRA and Raman microspectrometry

  ☺ TEM examinations

- **Co-iiradiation Si + O**

  ☹ As co-irradiated characterization by NRA (Si NRA contributions)

  ☺ As co-irradiated characterization Raman microspectrometry

  ☺ Annealing treatment

  ☺ Post-annealing characterization by NRA and Raman microspectrometry

  ☺ TEM examinations

Future works

- **TiC or TiN in UHP Fe or model Fe-xCr alloy (with $5 \leq x \leq 20$) by Ti/C or TiN co-implantation ($|\Delta G_f TiC| \sim 1.88$ eV, $|\Delta G_f TiN| \sim 3.22$ eV)**

- **Parametric study: R_p, dose rate, total dose, co-irradiation T, annealing conditions**

- **Extension to nanoprecipitate assisted synthesis for other carbide or nitride with a low enthalpy formation as WC ($|\Delta G_f WC| \sim 0.45$ eV)**

Post-doctoral work by Gihan VELISA at JANNUS Saclay

(from November 2011 to October 2013)