| ESRF | **Experiment title:** Microstructural study of Fe-implanted SiC : Towards a diluted magnetic semiconductor | **Experiment number:** HS-3341 |
|---|---|---|
| **Beamline:** BM02 | **Date of experiment:** from: 04-OCT-2007 to: 08-OCT-2007 | **Date of report:** 20-FEV-2008 |
| **Shifts:** 12 | **Local contact(s):** **Jean-Paul SIMON** (INP-Grenoble) | *Received at ESRF:* |


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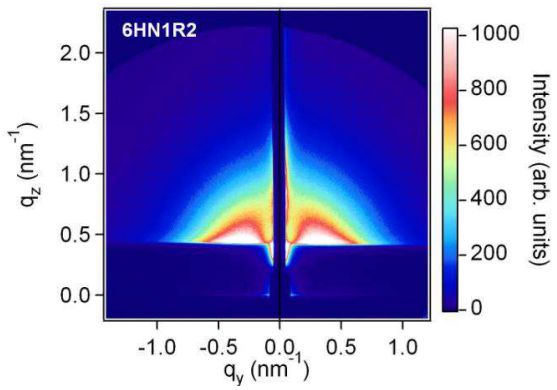
## Report:

**The goal of this project was to use GISAXS and XRD to characterize the morphology and the crystalline structure of magnetic nanoclusters formed by furnace annealing of Fe-implanted monocrystalline (0001) 6H-SiC samples.** The allocated beam time (12 shifts) allowed us to study all the 10 planned samples. The majority part of the allocated beam time was used for GISAXS experiments and it was possible to explore XRD of almost all samples by performing quick (θ, 2θ) scans.




GISAXS from sample annealed at 1000°C / 85'

**Figure 1**

| Samples | **GISAXS** Diameter of scattering particles | **SR XRD** Diameter of diffracting particles (Scherrer formula) | **SQUID/VSM** Ms in $\mu_B$/At.Fe at 10K (300K) |
|---|---|---|---|
| **As-implanted** | ~ 1-2 nm | - | Limit of detection |
| **Annealed 900°C/1h** | ~ 5 nm | 5.4 nm | 0.85 (0.5) |
| **Annealed 1000°C/85'** | 5-6 nm | 6.1 nm | 1.6 (0.75) |
| **Annealed 1300°C/4'** | > 6 nm | Not measurable | 1.5 (0.9) |

**Table 1**

GISAXS experiments revealed scattered intensity corresponding to scattering objects of at least 5 nm in diameter in annealed samples (Figure 1 and Table 1). As shown on Figure 2 and Figure 3, XRD experiments revealed extra 6H-SiC peak(s) in the range 2θ ~ 52.2° - 52.6° in the annealed samples.

Recent TEM observations of annealed samples (Figure 4) (at the LMP-Poitiers – D. Eyidi) have shown the presence of 5 to 10 nm dots with heavy element(s) (Z-contrast) and moiré patterns corresponding to crystallographic planes with an interreticular distance d ~ 0.2 nm parallel to the basal plane (0001) of the SiC matrix. The interreticular distance d ~ 0.2 nm corresponds to the Bragg peak observed in XRD at 2θ ~ 52.2° - 52.6° in the annealed samples.

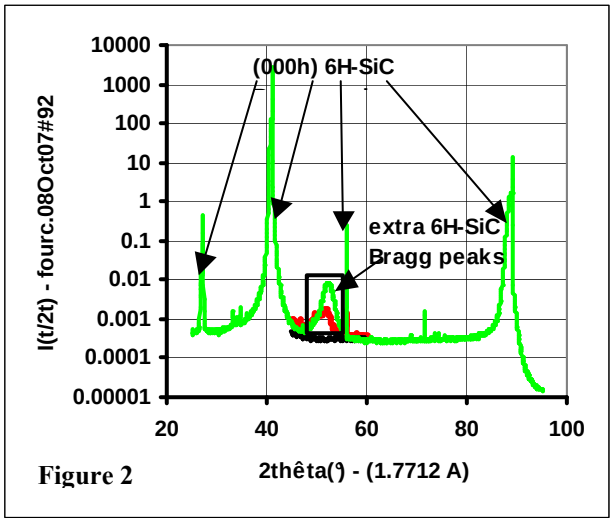



Figure 2

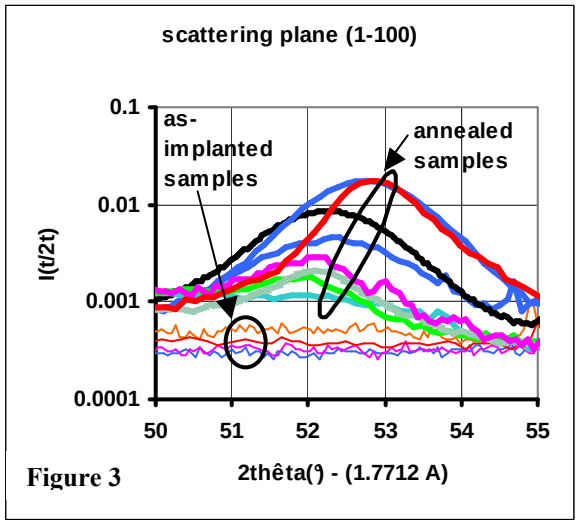
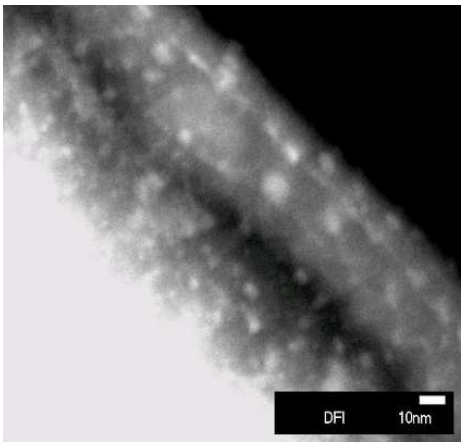



Figure 3



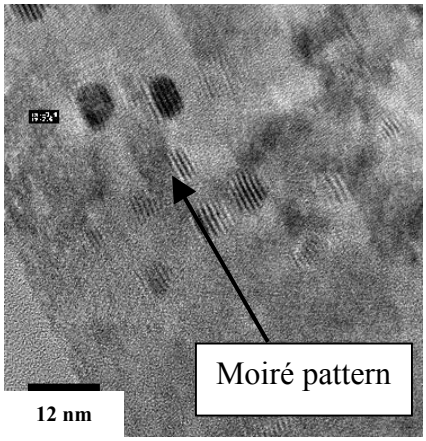
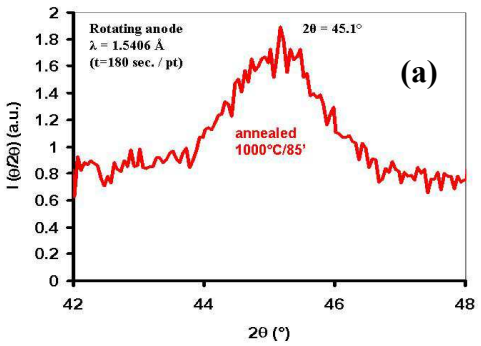




**Figure 4 :** 5 to 10 nm dots with heavy element(s) ($Fe_3Si$-like nanoparticles ?)

These results are coherent with $Fe_3Si$-like ($d_{220}$ ~ 0.2 nm) nanoclusters in epitaxial relationship with the SiC matrix in the annealed samples, as recently suggested in the literature [1]. Nevertheless, more work is needed to conclude about the exact nature of the observed Fe-rich nanoclusters (d ~ 0.2 nm may correspond to pure iron and $Fe_3C$ lines too).

Thus, using **7-circle Diffractometer facility of the BM02 beamline** allowed detection of very low concentration of diffracting Fe-rich nanoclusters in such samples, with a high sensitivity compared with our laboratory experimental set-up, as shown on Figure 5.



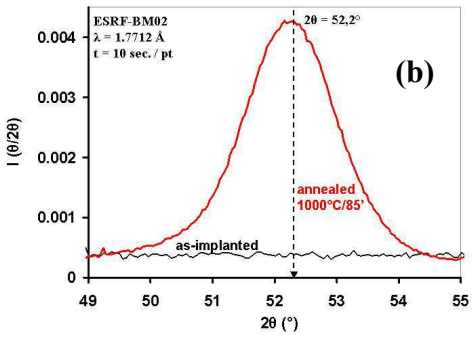




**Figure 5** : XRD curves of Fe-implanted 6H-SiC at 380° C and subsequently annealed at 1000°C for 85 min., highlighting the presence of $Fe_3Si$-like diffracting particles.
**(a)** laboratory set-up
**(b)** 7-circle Diffractometer facility of the BM02 beamline.

Moreover, using the **$\lambda \approx 1.77$ Å** radiation allows to avoid iron fluorescence (which is excited in our XRD laboratory set-up).

[1] F. Stromberg, W. Keune, X. Chen, S. Bedanta et al. , J. Phys.: Condens. Matter **18** (2006) 9881.