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Crystal distortions and structural defects at several scales generated during the growth of silicon contaminated with carbon

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Title: X-ray imaging analysis of crystal distortion and crystalline defect generation during solidification of silicon contaminated with carbon

Summary (field limited to 40 words):

Carbon is a major impurity when one wants to produce silicon for efficient photovoltaic solar cells. Its impact on the generation of distortions and defects was studied by X-ray imaging at ESRF both *in situ*, during solidification, and *ex situ*.

Main text (around 700 words):

For all fabrication processes of the photovoltaic (PV) industry based on crystalline silicon (Si), grain boundaries, dislocations, and impurity contamination control during solidification remains a major challenge to improve the electrical properties of the final Si material. Carbon (C) is one of the most frequent impurities in directional solidification processes. It can be introduced due to the industrial furnace environment, crucible and starting feedstock, in particular in the case of recycled materials. C can be at the origin of shunts, several-scale defects and distortions in the crystal that can all contribute to a degradation of the final performance of the solar cells.

The simultaneous combination of X-ray radiography and X-ray white beam Bragg diffraction imaging (topography) achieved *in situ* during Si solidification in the GaTSBI furnace (Growth at high Temperature observed by Synchrotron Beam Imaging), which is presently run at beamline ID19 at ESRF [1] allowed us to characterise the dynamics of the growth mechanisms involved in the formation of the grain structure and of defects, related to the presence of C. *Ex situ* Rocking Curve Imaging (RCI) experiments conducted at beamline BM05 at ESRF was used to obtain a more precise analysis of the defects including sub-grains and to quantify their associated distortion fields and misorientations. Moreover, the section RCI mode allowed to confirm that the defects observed are not limited to the surface of the samples and ingots but are also present within the bulk of the samples.

The effect of C impurity contamination on the formation of grains and structural defects during solidification for both reference and industrial samples was studied. In the presence of C, we show that the grain structure resulting from solidification is constituted by a higher proportion of high-order twin (δ in **Figure 1**) and of random angle grain boundaries for C-contaminated samples compared with the case of pure samples. This is due to the higher probability, in the presence of C and related precipitates, of grain nucleation not all related to the energetically $\Sigma 3$ twin most favourable nucleation in pure samples. Such twin boundaries are at the origin of grain-scale (mm to cm) distortions in the crystal that

persist and are accentuated after cooling down, can be at the origin of dislocation emission and act as preferential sites for the segregation of impurities in general.

Moreover, as revealed both *in situ* (ϕ in **Figure 1**) by White beam X-ray Bragg diffraction imaging (topography) and *ex situ* by rocking curve imaging (**Figure 2**), local distortion regions of the crystalline structure associated to sub-grains are distributed all over the samples. They are observed during solidification of different types of samples containing C (mono-crystal, industrial ribbons and multi-crystalline sample) and are retrieved after solidification. We propose a mechanism in which these features are formed during solidification in the presence of SiC precipitates at the level of the solid-liquid interface. The growing silicon just above the precipitates is misoriented compared to the solid-liquid interface of silicon growing from the seed. When both regions meet, sub-grains result and compete with each other due to the presence of precipitates all over in the sample volume. Bunches of dislocations can also be directly related to the presence of C precipitates thanks to the *in situ* topography imaging.

These results show the main impact on the formation of the grain structure and crystalline defects at different scales of the presence of C while growing silicon. These growth features that remain after solidification have a severe impact on the electrical properties of Si and further on the performance of photovoltaic solar cells. The results briefly described above and presented in [2] could only be derived from the correlation of both *in situ* and *ex situ* diffraction imaging techniques (**Figures 1 and 2**) implemented at ESRF.

Principal publication and authors

Ouaddah H. (a), Regula G. (a), Reinhart G. (a), Périchaud I. (a), Guittonneau F. (b), Barrallier L. (b), Baruchel J., Tran Caliste T. N., Mangelinck-Noël N. (a), Crystal distortions and structural defects at several scales generated during the growth of silicon contaminated with carbon, *Acta Materialia* **252**,118904 (2023); doi: /10.1016/j.actamat.2023.118904

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References

[1] Becker M. *et al.*, *Journal of Applied Crystallography* **52**.(6), 1312-1320 (2019).

Figures

Fig. 1: Sequence of topographs during solidification of a Si reference sample contaminated with C (1.5×10^{17} at/cm³) for the $\bar{1}1\bar{1}$ (top) and c) $\bar{1}1\bar{3}$ (bottom) diffraction spots. a) and f) t_0 : start of cooling-down at -1 K/min; b) and g) $t_0 + 6$ min; c) and h): $t_0 + 9$ min; d) and i): $t_0 + 11$ min; e) and j): $t_0 + 14$ min.

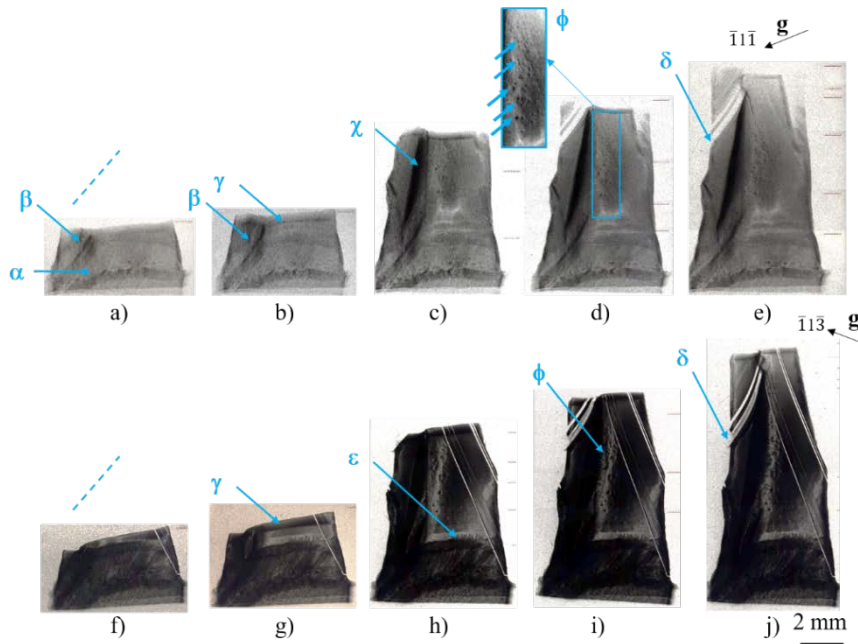
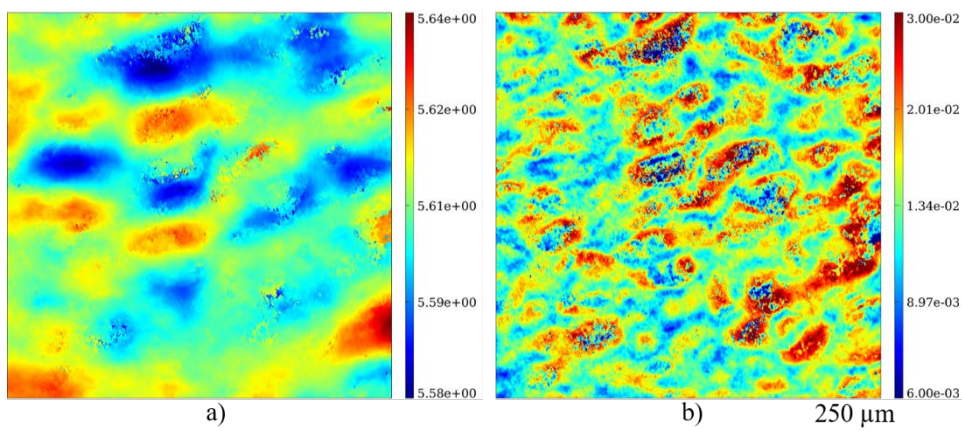
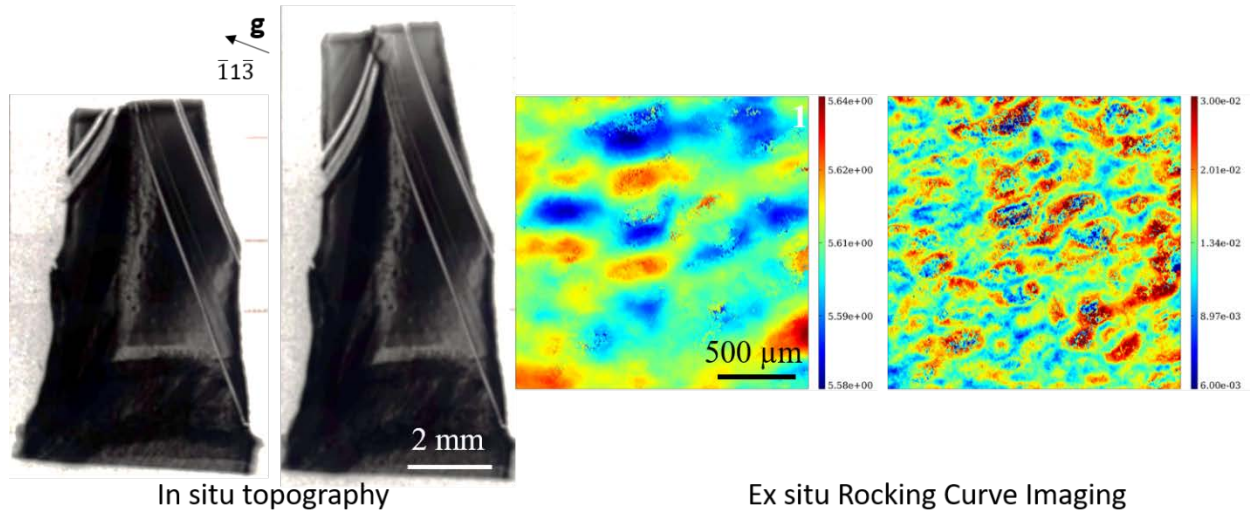


Fig. 2: Same sample as in Figure 1 after solidification: projection RCI ($\{111\}$ diffraction spot) results with a pixel size of $0.75 \mu\text{m}$, a field of view of $1.5 \times 1.5 \text{ mm}^2$ and a Bragg angle : $\theta_B = 5.6^\circ$. a) Peak-Position (PP) map. The scale gives the position of the peak in degree. b) Full Width Half Maximum (FWHM) map. The scale gives the misorientation value in degree.



Feature Image for the ESRF home page [optional]



Feature image.

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Beamline(s) on which experiments were carried out: BM05 and ID19.