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# Quantitative HRTEM investigation of nanoplatelets

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ABSTRACT

High resolution transmission electron microscopy experiments were performed to investigate nanoplatelets induced by ion implantation into a germanium wafer. Atomistic models were used for image simulation in order to get quantitative information from the experimental images. The geometrical phase shift analysis technique was also employed to measure the strain field induced by such defects. We discuss the limits and artefacts imposed by each approach and show how these approaches can be combined to study the atomic structure of such defects and the strain field they induce.



## 1. Introduction

The term "Platelets" is commonly used in the literature to denote two-dimensional defects. Platelets are encountered in diamond-type structure and structures derived from it: sphalerite and wurtzite. Their presence was reported since the early 60s and extensively studied in diamond (Evans and Phaal, 1962; Barry, 1991). In other covalent crystals such as Si, Ge, GaAs, InP or GaN platelets can be created by a high dose incorporation of hydrogen or helium into a wafer via implantation or diffusion (Terreault, 2007; Fichtner et al., 1999; David et al., 2007; Nomachi et al., 1997; Hayashi et al., 2004; Moutanabbir et al., 2008). Helium platelets have also been widely studied in metals, in the framework of nuclear applications (Evans et al., 1981). In all cases, these defects are at the origin of strong surface or sub-surface modifications (blistering, exfoliation, layer splitting) that triggered out a huge amount of studies for materials improvement in the field of nuclear technology and for defect engineering in semiconductors.

The questions related to platelet description concern the geometrical parameters (width and lateral extent), the composition (presence of vacancies, interstitials or impurities) and the lattice displacement they induce. Whatever the system, direct experimental methods are required to give an accurate description of the atomic structure of the platelets. Their analysis is a long-standing and challenging problem for transmission electron microscopy (TEM), closely related to the analysis of Guinier–Preston zones in CuAl alloys.

Since the pioneer works from Evans and Phaal (1962) in type I diamond, many TEM approaches were employed to measure the strain field surrounding platelets and to probe their atomic structure. The estimation of the strain field was mainly conducted by using a Moiré fringe technique (Bursill et al., 1981) or based on the refinement of the intensity in images obtained under two-beam conditions (Humble, 1982; Muto et al., 1995). Measurements were also performed by large angle convergent beam electron diffraction on large platelets (Cherns et al., 1997). Naturally, the determination of their atomic structure was tackled by matching the experimental HRTEM contrast with simulated images (Barry, 1991; Bursill et al., 1978; Humble et al., 1985), with various levels of accuracy depending on the performances of the microscopes used and on the level of discrepancy between the atomic models to be discriminated. These last experiments were mainly performed on relatively large platelets (viewed edge-on) to ensure that they thread the electron microscopy sample, in order to compare experimental and simulated contrasts.

The level of knowledge concerning the structure of hydrogen-induced platelets (HIPs) in semiconductor can be summarized by the following assessment: the determination of the strain field and

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atomic structure of platelets have never been performed on the same defects. Indeed, the strain field measurements were mostly obtained for embedded platelets whereas the atomic structure refinements were extracted from platelets from which edges were cut (so that they thread the sample) (Neethling et al., 1988). This point could be considered as minor detail, but if one considers that strain relaxation occurs during the specimen preparation (especially by releasing the stress at edges), then one can easily admit that the images would be affected by removing the edges of a defect. The situation becomes critical in the case of gas-filled defect from which the possible trapped gas would have been released by cutting the edges. This is a pitfall in the understanding of the nucleation and evolution of these defects, as the two approaches reported above can hardly be compared. Bringing together strain field measurements, atomic structure refinement and results obtained from theoretical calculations is expected to be very fruitful. Such an approach has recently allowed establishing that platelets in diamond were related to the presence of nitrogen (Goss et al., 2003).

In the case of nanometric HIPs (nanoplatelets), as a consequence of the scale of investigation, HRTEM is the only relevant approach. An elegant and accurate method for evaluating the displacement field around defects in crystals from HRTEM images was proposed by Hÿtch et al. (1998). This geometrical phase shift analysis (GPA) method was successfully employed to measure displacement field around dislocations in silicon with accuracy better than 0.003 nm (Hÿtch et al., 2003). Recently, an attempt to measure the displacement field around nanoplatelets, produced by boron implantation in silicon, by a combination of weak-beam dark field imaging and GPA analysis was reported (Cherkashin et al., 2005). However the authors did not mention whether the defects studied by HRTEM were embedded in the thin electron microscopy sample or not, which is a critical point for HRTEM image interpretation. Moreover, in the case of gas-filled cavities, as the strain field along the electron beam path is inhomogeneous, the contrast variations with observation conditions (crystal thickness, defocus, ...) might be unpredictable (Cherns et al., 1997; Spence, 1982). Otherwise speaking, one cannot assess that the atomic columns coincide with white (or black) dots in the whole image, especially close to the core of the defect, where the displacement field could be relatively high. Such effects could notably distort the results given by the GPA method. Thus, probing the exact stoichiometry and the atomic structure of nanoplatelets from quantitative HRTEM experiments seems to remain challenging.

It appears essential in such a case to combine image simulation based on atomistic models with the GPA method. In this paper we discuss the limits and artefacts of such an approach applied to embedded defects. We finally show that the combination of the HRTEM contrast matching, displacement field analysis via the GPA method and DFT calculation leads to a reasonable description of the structure of nanoplatelets and their associated strain field.

## 2. Technical details

HIPs were obtained by high fluence hydrogen implantation in a n-type (0 0 1) germanium wafer (details are given in David et al., 2007). HRTEM experiments were performed in the cross-section geometry along the [1 $\bar{1}$ 0] zone axis. The TEM sample was mechanically polished with a tripod polisher down to 10 μm and subsequently thinned by ion milling in a GATAN-PIPS apparatus until electron transparency. Low energy (2.5 keV) and low incidence (±8° and ±6° for the final step) were used to minimize irradiation damage. This procedure ensures the completion of wide thin areas required to perform quantitative HRTEM (Q-HRTEM). HRTEM images were recorded by using a JEOL 3010 operated at 300 kV ($LaB_6$, $C_s$ = 1.2 mm, 0.19 nm point-to-point resolution). Focus series were acquired either on photographic film or with an ORIUS SC 1000 CCD camera at nominal magnifications of 400 and 600 K. Photographic films were further digitized either at 1200 dpi (dot per inch) or 2400 dpi providing an adequate sampling of atomic columns for GPA analysis. Despite the high voltage used for these experiments, we did not observe the formation of irradiation-induced extended defects. HRTEM pictures were then analyzed with the GPA method to estimate the strain field surrounding the platelets. The HRTEM contrast was simulated using the JEMS package (Stadelmann, 1987) in the multislice framework. For all calculations, the Debye-Waller factors were set to 0.005 $nm^3$ (which is highly questionable for the atoms located at the core of the defect); details can be found in Barbot et al. (2008). Sample thickness $t_s$ and objective defocus $\Delta f$ were determined by image matching of a strain free region, located several tens of nanometres away from the studied defects, with thickness/defocus maps calculated using both Bloch wave approach and multislice method. Input models for multislice simulations of the contrast of the defects were obtained from atomistic simulations performed with the Tersoff inter-atomic potential (Tersoff, 1989) Different configurations of the core of the platelets were considered: the number of removed Ge-atoms layers was varied as well as the shift $\delta$ between the core atoms along the [0 0 1] direction (incremental step was set to 0.01 nm) (David et al., 2009). The atomic positions surrounding the fixed core atoms were then relaxed until atomic forces became negligible. The contrast simulation was then carried out following two main steps: first we considered a defect threading the sample, and afterwards we built a model in which the defect had a finite size $t_d$ along the electron beam path (embedded in a matrix, see Fig. 1). The contrast matching between experiments and simulations was then optimized by varying $\delta$ (0.01 nm step), the position $h_d$ and the thickness $t_d$ of the defect (by 0.8 nm steps). Total thickness $t_s$ and objective defocus $\Delta f$ were also optimized by fine tuning around the values previously determined from a strain free region. Threefold astigmatism influence was deliberately not considered in this study (Merkle et al., 1998), since the lattice fringes imaged here are the same across the image, which would not imply an artificial expansion of the core of the defect (the situation significantly differs from the case of an asymmetric grain boundary). Complementary to this classical approach in Q-HRTEM, the simulated images were also analyzed using the GPA method in order to compare the phase images obtained from experimental micrographs and simulated pictures. These results were finally checked against density functional theory (DFT) calculations; details concerning the latter are given elsewhere (David et al., 2009).

## 3. HRTEM experiments on nanoplatelets

Fig. 2a shows the deeper part of the buried defect layer produced by H implantation in Ge. (0 0 1) platelets which sizes range from 5 to 10 nm are observed. On this picture, it is worth

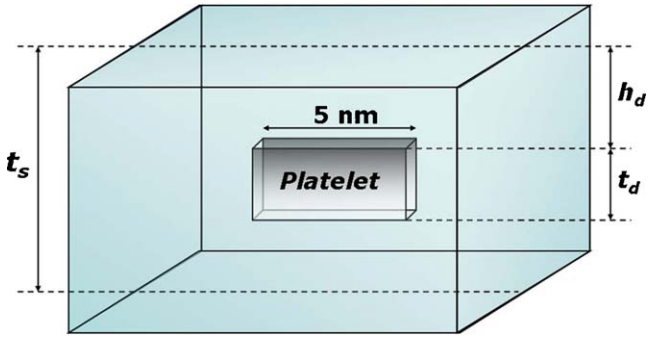


**Fig. 1.** Sketch of a nanoplatelet embedded in a TEM sample depicting the geometrical parameters used for our calculations.