TEM study of homoepitaxial diamond layers scheduled for high power devices: FIB method of sample preparation

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Abstract

Homoepitaxial diamond structure observation by transmission electron microscopy (TEM) is still a very hard job due to the difficulty in preparing electron transparent samples for the further observation. The present contribution details the experimental operations with their respective conditions step by step. Finally high resolution TEM (HREM) observations of a CVD grown epilayer on a unintentionally doped HPHT (001) oriented substrate are present to show the high quality of the sample preparation method.

Keywords: homoepitaxial diamond, electron microscopy; FIB dual beam.

1. INTRODUCTION

Despite diamond is one of the most promising candidates among the wide bandgap semiconducting materials for extreme conditions of service, the technology is still not ready for implementation of this material in industrial opto- and microelectronic devices. High mechanical properties combined with its highly attractive optical, electronic (carrier μ~4000cm^2/Vs) and thermal properties and the possibility to range from an insulating to semiconducting [1] or metallic [2] and even superconducting [3] behavior should be good reasons to push investigation toward industry. Up to date diamond has demonstrated some capability to yield high power, short wavelength or high velocities devices. Indeed, Schottky diodes [4], UV-LEDs [5] and delta-doped layers [6] fabrication were reported. Nevertheless, only in very few cases was this material evaluated at the nano- and microstructural level in terms of defects and chemical distribution [7], although the electronic devices could certainly be improved optimizing their layers structures, doping profiles or defects distribution. In this context, Transmission Electron Microscopy (TEM) [8-10] studies are necessary, but such observations require electron-transparency of the samples. In the case of homoepitaxial diamond, the conventional method of preparation is not adequate as no mechanical thinning can be applied before the final ion milling.

This high mechanical hardness turns difficult not only the sample preparation for TEM but also to cleave the sample to make local analysis versus depth or to make laser mirrors. The commercialization of FIB-Dual Beam [10] (Focused Ion Beam coupled to a SEM column) turns now easier the sample preparation for TEM studies although this equipment is still only introduced in a few laboratories. This allows replacing the mechanical preparation operations by another abrasion technique. This solution for hard materials allows, in addition, a very precise choice (below 100nm in the semiconductiong Si industry) of the region for TEM observation and also a fairly small waste of material while preparing for the TEM preparation (area around 20x20µm).

2. EXPERIMENTAL PROCEDURE

The methodology proposed in this work, starts with Pt [11] deposition using an electron beam and finishes in the same SEM chamber with the slimming of the sample until electron-transparency is reached under Ga ion-beam abrasion. The paper will describe this procedure in details and will conclude with a typical TEM study.
The FIB-Dual Beam used is a FEI Quanta 3D, with 52º between the electron (thermoionic W gun) and ion columns (Ga ions). TEM observations are carried out with a Jeol 2010F microscopes working at 200 keV beam energy allowing high coherence and small spot size for scanning transmission electron microscopy (STEM).
Samples are grown by chemical vapour deposition (CVD, 2µm thick epilayer) on Sumitomo (001) oriented HPHT substrates. The specificity of the sample preparation on the FIB is here discussed because the related chamber volume and number of injectors is reduced respect to standard FIB.

3. RESULTS AND DISCUSSION

In the following, the FIB-related operations of TEM preparation are described step by step. In Fig.1 SEM micrographs, the two first operations are described: (i) 5µm thick Pt deposition and (ii) the dig of two trenches at each side of the latter (see inset Fig1b). The Pt deposition is located just above the region chosen for the TEM observation. Its duty is to protect, from the ion bombardment, the region of TEM observation. This deposition is performed with an ion current of 0.3nA at a beam energy of 30keV, with the Pt injector located at around 10µm from the deposition region. It is worth noting that, for the case of diamond, electron assist deposition, induces diamond etching probably due to ionization of the $Pt(CO)_6$ gas producing oxidation of the diamond with generation of $C0_2$. The principle of the deposition below the ion beam is identical to CVD and the occurring reactions are comparable to laser induced CVD, reaching in the present case much better resolutions. Indeed, the physics of this deposition consists in cracking, below the high energetic Ga bombardment, the organometallic molecule to obtain metallic Pt. The residual molecules are then evacuated by the vacuum pumps. For the present work, using a FEI Quanta 3D FIB, Pt deposition with the ion beam at 52º, respect to the surface to be soldered, is not possible. The incident beam has to be always perpendicular to the surface of deposition.
Then, on both sides of this Pt deposition, trenches are trimmed to define the region that will be then observed. This method has been previously used for Si and labelled lift-out [12]. The depth of trenches depends on how deep the observations should be performed. Indeed, the angle of the stairs in the inset is nearly 52º and can not be changed because the further cut off of the lamella (see inset Fig.2) is done using the ion beam. Therefore, the deeper is required the TEM preparation, the wider should be the trench. They are machined by different successive operations of etching making then this stair-like shaped shown in the inset.

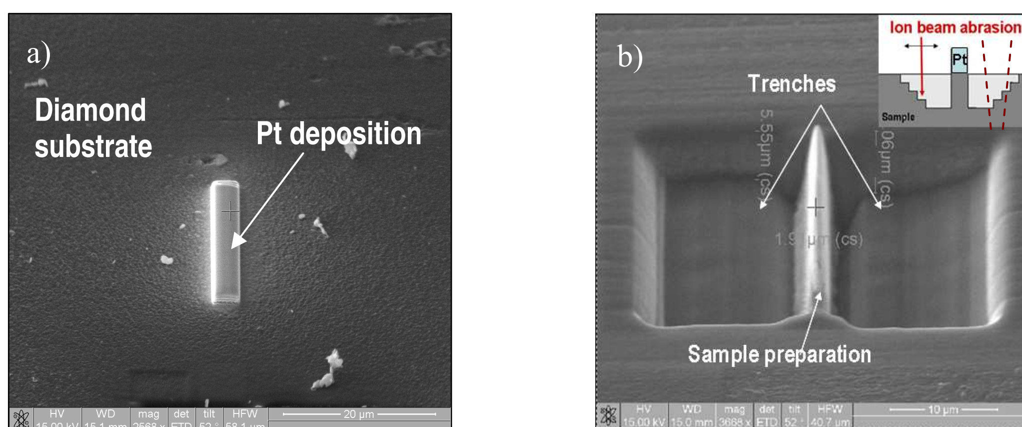

Fig.1: (a) SEM micrograph of diamond sample surface with the Pt deposition. (b) SEM micrograph after the trenches were dug by the ion beam at each side of the region chose for the sample preparation.

The etching is performed here under 20nA ion beam current, as also used for other materials [13]. Once the trenches are obtained a second operation to reduce the bottom sample thickness is carried out tilting the sample ±7º respect to the ion beam direction as indicated by the dashed lines in inset. This thinning is achieved in both side of the defined lamella. The ion beam current varies from 7 nA to 3 nA during this tilt milling. The ending current, is fixed at that value to make as mild as possible the final etching. The process of milling is executed with help of $MgS0_47H_20$ (labelled SCM: Selected Carbon Milling) injected near the ion beam spot to avoid redeposition of the removed materials. This process is always executed when the ion beam is used as etching tool. Cracking this molecule induces oxygen that combine then with the carbon forming volatile CO_2. Other authors simply reduce the current and/or dwell time at the expense of a longer process time or use the help reactive gases, as for example fluor, to binds the sputtered atoms [14]. For diamond, the solution is easier as it highly reactive with oxygen. Once this phase is finished, the resulting lamella should be retired from the bulk diamond and fixed to a Cu grid for TEM observations. In Fig. 2a, the image shows the process of cutting the thin lamella from the bulk material using the Ga ion beam. Only a reminding hold is kept, as shown at the right hand of the micrograph. It will be removed once the sample is fixed to the micromanipulator tip (Fig.2b). This will enables to remove the lamella from bulk material. The cut process is realised at 30kV/3nA ion beam with the geometry shown in the inset i.e. with a tilt of the lamella plane of 59º respect to the ion beam (see inset Fig.2a). To have the angle above 52º allows the electron observation of the cut.
In Fig.2b, the extraction of the preparation from the bulk material is showed. This is performed after the soldering the lamella to the tip of the micromanipulator followed by the ion milling of the hold reminding at the right hand of fig2a. The soldering process is achieved at an acceleration voltage of 30 kV and an ion beam current of 0.3 nA. This is

performed with ions impinging on the lamella with an angle of 59º, using C deposition, on both tip and Pt lying on the top of the lamella. This means with the electron beam at 7º allowing a simultaneous observation. Other authors used W, Pt or even just held by electrostatic forces [15]. Here, Pt deposition is possible only with perpendicular incidence of ions and this operation must be done with the sample in a horizontal position to retire then the lamella by downloading the sample.

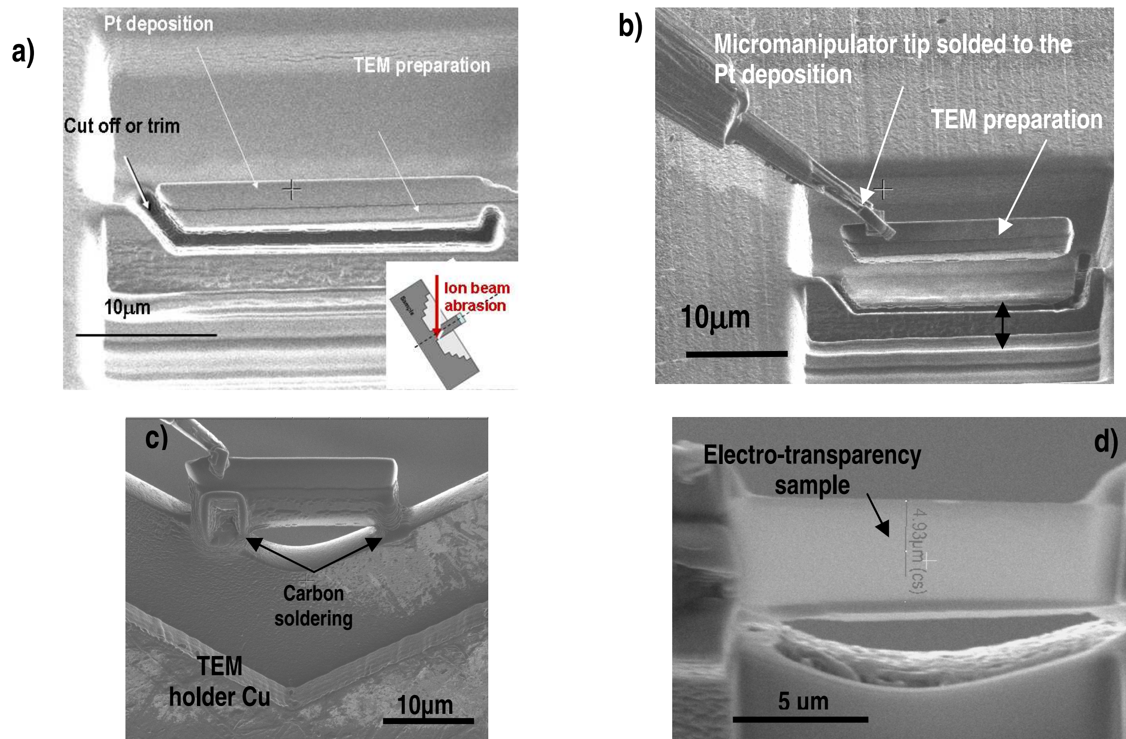

Fig.2: (a) SEM micrograph of the sample preparation after etching around the machined platelet. (b) SEM micrograph of the specimen in the moment of the lamella extraction from the bulk sample. The adhesion/soldering of the lamella to the tip of micromanipulator using C on Pt deposition can be observed (see arrow). (c) Micrograph of the sample fixed to Cu grid before cutting the tip with the ion beam. (d) After fixing the sample preparation to the grid and cutting the tip from the sample, a thinning and cleaning is applied as shown in the SEM micrograph.

Thus, with the FEI Quanta·3D model, the soldering must be performed with carbon. The tip approach operation is very delicate as the tip must touch the Pt. This step can be followed by simultaneous SEM observation and, when it touches, the sample move slightly. Once the lamella is moved to the grid, it should be fixed on it. The figure 2c shows the soldering of the lamella to the TEM grid using C (as Pt can be achieved only with a perpendicular incidence of the ion, here the sample is at 52º respect to ions and 90º respect to e-beam) just before the final thinning. This step requires to use a low ion beam current density with an orientation perpendicular to the sample surface plane. This geometric configuration allows holding the lamella to TEM grid without superficial damage [13].

Finally a thinning and cleaning of the TEM preparation should be applied. This final process is performed in two steps. First, the sample preparation is oriented parallel to the ion beam. Thus, when the ions are milling flush on the surface of the sample preparation, the electron beam allows to roughly evaluating at the same time, by transparency, the instantaneous thickness. These mild millings are achieved sequentially at one side of the sample and then at the other applying a short tilt of $\pm$2º upon each side sequentially. The ion beam acceleration voltage is fixed first at 30 kV and a current of 1nA. The operation is repeated until a thickness of 500 nm is reached. Then those conditions are reduced progressively to final values of 5kV and 0.07nA and tilts of $\pm$ 6º to soften the etching and avoid the ion implantation [15]. The final thickness should be around 50 nm, and then the TEM observations can be performed. In Fig.3, the low magnification TEM micrograph and high resolution TEM (HREM) shows the capability of the technique.

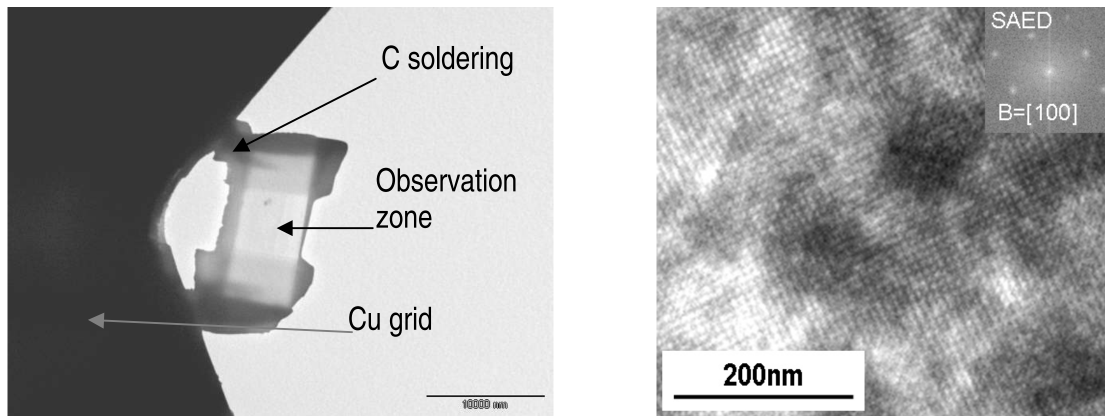

Fig 3: (a) Low magnification TEM micrograph of the diamond sample preparation. The micrograph shows the final result using the FIB-Dual Beam procedure. (b) HREM observation showing the capability of the technique.

4. CONCLUSIONS

The FIB-Dual Beam TEM preparation of diamond homoepitaxial structure is described. The classical routine used for other semiconducting materials is here adapted to diamond material. The main feature introduced here are: (i) For the case of undoped diamond, samples are charging during ion beam operation and SEM position checking should be performed frequently to ensure beam position, (ii) Electron assist Pt deposition is found to etch the diamond and thus only direct ion beam assist deposition is performed, (iii) for the cut off of the lamella, an inclination of 7º respect to the electron beam (i.e. etching at 59º of the ion beam) is applied to allow simultaneous observation by SEM of the ion machining, (iv) soldering is achieved by carbon deposition in spite of Pt. Finally HREM observations certify the high quality of the preparation method.

5. REFERENCES

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