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Scanning near-field cathodoluminescence microscopy of an Si^+ implanted and thermally annealed SiO_2 layer

J. Fauré [a,*], D. Pastré [a], D. Muller [b], M. Troyon [a]

[a] *Université de Reims, Unité de Thermique et d'Analyse Physique, EA 2061, 21 rue Clément Ader, 51685 Reims cedex 2, France*
[b] *Laboratoire PHASE, UPR 292-CNRS, 67037 Strasbourg cedex, France*

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Abstract

A scanning near-field cathodoluminescence microscope (SNCLM) is successfully used to image the cathodoluminescence of an Si^+ implanted and thermally annealed submicronic SiO_2 layer. Owing to the subwavelength resolution of the system a "cross-sectional" cathodoluminescence image was obtained. The intensity image profile shows that sample luminescence results from the whole SiO_2 layer confirming a preceding electroluminescence study. Sample luminescence is attributed to point defects generated into the whole SiO_2 layer during Si^+ ion implantation and thermal annealing. © 1999 Elsevier Science B.V.

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1. Introduction

The luminescence of silicon based structures is strongly investigated since the discovery of visible light emission in porous silicon [1]. Structures based on Si nanocrystals embedded in SiO_2 has led to interesting results [2,3]. Ion implantation of Si^+ ions into SiO_2 is a promising technique to elaborate such materials. Until now the cathodoluminescence (CL) of Si^+ implanted SiO_2 layers have scarcely been studied. The classical cathodoluminescence systems have generally a resolution of the order of 1 μm or even more. Indeed, these systems use mirrors [4] or fibers [5] to collect the photons emitted by the sample in the far-field mode and then the lateral resolution is generally limited by the interaction volume of the electrons and the energy transfer in the specimen [6].

When a better resolution is required near-field detection systems can be used owing to their subwavelength resolution. We have recently developed a Scanning Near-field Cathodoluminescence Microscope (SNCLM) to improve the resolution of cathodoluminescence images [7,8]. Our system is a combination of a Scanning Force Microscope (SFM)/Scanning Near-field Optical Microscope (SNOM) with a Scanning Electron Microscope (SEM) equipped with a Field Emission Gun (FEG). This hybrid instrument allows the conventional imaging by SEM, the investigation by SFM of the local topography and simultaneously the CL imaging.

* Corresponding author: Laboratoire de Microscopies Electronique et Tunnel, 21 rue Clément Ader, 51685 Reims cedex 2, France; e-mail: joel.faure@univ-reims.fr.

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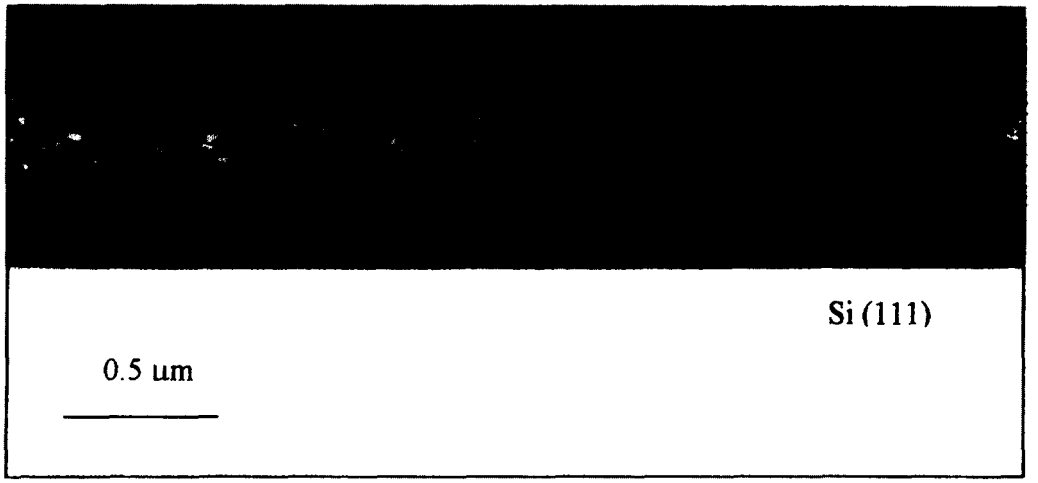

Fig. 1. (220) DF XTEM micrograph of the SiO_2 layer, thermally grown on an Si(111) substrate, after Si^+ ion implantation (160 keV; 10 $\mu A\,cm^{-2}$; $1.2{\cdot}10^{18}$ cm^{-2}) and thermal annealing (1100 °C; 4 h).

In this paper, owing to the subwavelength resolution of our SNCLM, we have investigated the "cross-sectional" cathodoluminescence of a submicronic Si^+ implanted and thermally annealed SiO_2 layer. Concurrently "cross-sectional" TEM (XTEM) observations were performed to investigate the structural state of the sample.

2. Experimental

The SNCLM has already been described elsewhere [7,8]. We are giving here only its main features. The SFM head is able to work as a SNOM owing to an optical fiber placed just upon the silicon nitride tip at a distance of about 50 μm. The photons are generated by a 3 keV electron beam reaching the surface, just under the SFM tip, with a 70° incidence resulting in a maximum penetration depth well below 100 nm. The photons are collected in the near-field mode by the SFM tip and diffracted towards the optical fiber. The great advantage of the near-field collection is that the resolution is not limited by the energy dissipation and energy transfer in the material, since this technique is based on evanescent wave detection. With this system, a 100 nm lateral resolution has been obtained [7]. Because of the high brightness of the FEG, high electron probe current density can be obtained, even at low voltage, that favors the photon yield. Despite these good working conditions, the current detected by the photomultiplier is limited to about 0.3 nA corresponding to a light power collected by the fiber of about $0.3{\cdot}10^{-9}$ W.

The sample was prepared as follows: a low resistivity ($\rho < 8{\cdot}10^{-4}\ \Omega\,cm$) Si (111) n-type substrate was thermally oxidized at 1100 °C to obtain a thick homogeneous SiO_2 layer. The SiO_2 layer was implanted at room temperature with Si^+ ions in the following conditions: ion energy = 160 keV; ion dose = $1.2{\cdot}10^{18}$ cm^{-2}, ion current density = 10 $A\,cm^{-2}$. The implanted sample was finally annealed in a controlled Ar atmosphere at 1100 °C for 4 hours.

For SNCLM and XTEM observations a "cross-section" was prepared. Two pieces were cut on the sample and glued together. From the obtained sandwich a slice was cut with a diamond saw and mechanically polished to a thickness of about 50 μm. Before SNCLM observations the slice surface was cleaned by ion etching with an Ar^+ ion beam under the following conditions: ion energy = 5 keV; ion beam incidence = 10°; ion current density < 3 $\mu A\,cm^{-2}$. For XTEM observations, the slice was finally thinned by ion etching using two aligned Ar^+ ion beams having the previous characteristics. XTEM observations were performed in the "two strong beams" conditions to investigate the crystallographic structure of the sample.

Fig 2 (a)

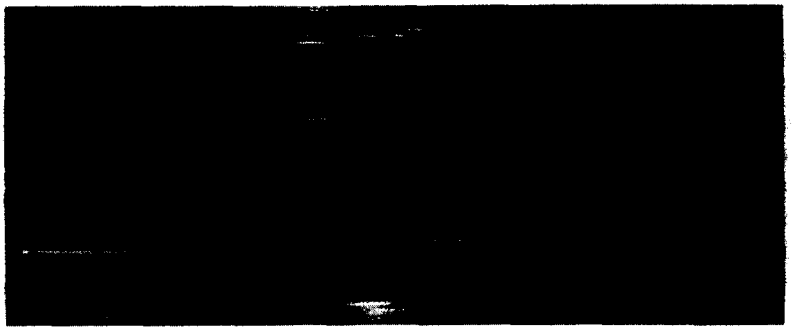

Fig 2 (b)

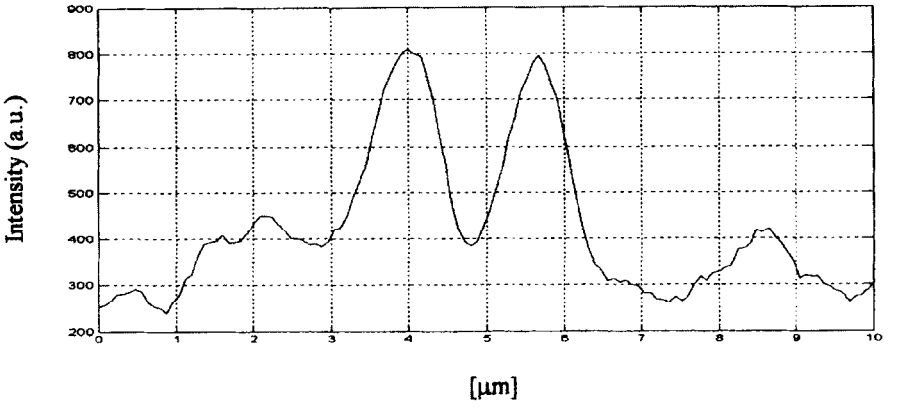

Fig 2 (c)

Fig. 2. (a) SEM micrograph of the sample "cross-section". The two SiO_2 layers are visible on both sides of the central glue line. (b) SNCLM image of the area shown in the SEM micrograph revealing two parallel bright bands associated to SiO_2 layers. (c) CL intensity profile of the above image. The width at half height of each peak is 0.7 μm corresponding to the width of the SiO_2 layers.

3. Results and discussion

Fig. 1 is a (220) dark field (DF) XTEM micrograph of the sample showing a 650 nm thick SiO_2 layer on the Si (111) substrate (black and white respectively). The first half of the SiO_2 layer presents a high density of Si nanocrystals (appearing in white). In the micrograph, individual crystallites with a size widely larger than 10 nm are visible. Microdiffraction investigations reveal that Si nanocrystals are randomly distributed into the amorphous SiO_2 layer. In a preceding paper [9] we related Si-nanocrystals formation to Si^+ ion-implantation and thermal annealing conditions.

Fig. 2 presents a SEM micrograph and the related SNCLM image obtained with a 3 keV electron beam. In the SEM micrograph (Fig. 2a), the central band (about 1 μm wide) corresponds to the glue and on both sides the SiO_2/Si interface is clearly visible at a depth of about 0.7 μm. The CL image (Fig. 2b) presents a central black band (the glue) between two parallel bright bands (the SiO_2 layers). The CL intensity profile of the sample is presented in Fig. 2c and reveals two separated peaks associated to the two bright bands. The luminescence peaks are symmetrical and no luminescence-increase is observed on both sides of the central black band. The bandwidth at half height of the two peaks is estimated to be about 0.7 μm. These observations suggest that sample cathodoluminescence results from the whole SiO_2 layer without Si-nanocrystals contribution. Indeed the symmetry of intensity peaks reveals the absence of CL signal increasing in the first half of the SiO_2 layer.

These results confirm a previous electroluminescence (EL) study [9]. In this paper it is firstly demonstrated that there is no EL signal related to Si-nanocrystals emission (at around 600 nm) and secondly that a blue EL peak is observed at around 470 nm whereas no EL signal is obtained with a non-implanted sample. The luminescence at 470 nm is attributed to point defects ("neutral oxygen vacancies") introduced into the whole SiO_2 layer during Si^+ ion implantation and thermal annealing and the absence of Si-nanocrystals related emission is related to a too-high mean-size of Si nanocrystals.

In summary, we successfully used our SNCLM system to investigate the "cross-sectional" cathodoluminescence of a submicronic Si^+ implanted and thermally annealed SiO_2 layer. Owing to the subwavelength resolution of our SNCLM we obtained a SNOM image showing that sample luminescence results from the whole SiO_2 layer. This result confirms the conclusions of a preceding EL study [9] attributing the sample luminescence to irradiation point defects introduced into the SiO_2 layer during Si^+ ion implantation and thermal annealing. This work finally demonstrates that the SNCLM is a powerful instrument to localize structural defects luminescence on solid surfaces with a high lateral resolution.

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