IL NUOVO CIMENTO Vol. 27 C, N. 5 Settembre-Ottobre 2004
DOI 10.1393/ncc/i2005-10014-8

Investigation of dopant profiles in nanosized materials by scanning transmission electron microscopy

P. G. Merli(1), V. Morandi(1), A. Migliori(1), C. Baratto(2), E. Comini(2)
G. Faglia(2), M. Ferroni(2)(*), A. Ponzoni(2), N. Poli(2) and G. Sberveglieri(2)

(1) *CNR IMM Sezione di Bologna, Area Ricerca Bologna - Via Gobetti 101*
I-40126 Bologna, Italy

(2) *INFM Sensor Lab. - Dipartimento di Chimica e Fisica per l'Ingegneria e per i Materiali dell'Università di Brescia - Via Valotti 9, 25133 Brescia, Italy*

(ricevuto il 20 Gennaio 2005)

Summary. — Scanning electron microscopy is capable to provide chemical information on specimens interesting for the field of materials science and nanotechnology. The spatial resolution and the chemical information provided by incoherent imaging and detection of transmitted, forward-scattered electrons can reveal useful information about the specimen composition and microstructure. This paper discusses the capability and potential of low-voltage Scanning Transmission Electron Microscopy (STEM) for the characterization of multilayered structures and dopant profiles in crystalline materials.

PACS 68.37.Hk – Scanning electron microscopy (SEM) (including EBIC).
PACS 85.40.Ry – Impurity doping, diffusion and ion implantation technology.
PACS 61.72.Ss – Impurity concentration, distribution, and gradients.

1. – Introduction

The basic research in the field of nanosized materials and the effort to fabricate complex nanostructures require a continuous improvement of the characterization techniques. In fact, the capability to investigate the microscopic structure of a material down to atomic level allows one to understand the basic physics mechanism governing the evolution of the nanostructure and to optimize the preparation processes.

Scanning or transmission electron microscopy (SEM or TEM) can provide imaging at very high spatial resolution and even reveal chemical information about the specimen at a comparable nanometric scale. Such a joining offers an unrivalled advantage for the characterization of nanosized materials and particularly in the field of silicon-based

(*) E-mail: matteo.ferroni@ing.unibs.it

© Società Italiana di Fisica

devices, as the shrinking dimension of integrated systems has pushed other measurement and analysis methods such as Rutherford Backscattering spectrometry or Secondary Ion Mass spectroscopy beyond their resolution capabilities [1-4]. Indeed, the reduction of the physical size of semiconductor devices requires a continuous improvement of the spatial resolution capabilities, while the production of shallow junctions that is presently pursued demands an enhanced chemical sensitivity.

Transmission electron microscopy at high beam energy, typically in the 200–400 keV range, attains spatial resolution higher than the one provided by scanning electron microscopy. Presently, the incoherent imaging formed by scanning the TEM nanoprobe through a thin specimen and collecting the transmitted electron through a high-angle annular dark-field (HAADF) detector achieves atomic resolution together with a contrast directly interpretable in terms of chemical features of the specimen. Such a high performance has been enabled by the capability to focus the electron beam into a subnanometric probe. The limitations of the methods arise from the difficulties in preparing a specimen as thin as to limit the beam broadening and from the efficiency of electron collection trough the electro-optical system of the TEM.

Low-energy STEM, *i.e.* the collection of transmitted electrons generated by the 20–30 keV beam of a SEM, offers a complementary approach to incoherent imaging and is finding growing interest in the field of device characterization. Beside the limitation in probe size-state of the art microscopes can achieve probe size lower than 1 nm, low-energy STEM takes advantage of the absence of electromagnetic lenses below the specimen. Thus, such an aberration-free system is capable to achieve high electron collection efficiency.

This article presents the capability of low-energy STEM to investigate compositional profiles in doped silicon, with the aim to highlight the sensitivity and resolution of the technique.

2. – Experimental set-up

The first test specimen was purposely fabricated by molecular beam epitaxy and consists of several AlAs layers equally spaced by GaAs. The width of the first AlAs layers measures 40, 20, 10, 8, 5, 3, and 1 nm, and the layers are separated by 100 nm GaAs. Two ultra-narrow layers, only 2 and 1 AlAs mono-layers in width, were also realized in the specimen. Such a sample features an abrupt compositional variation across the specimen, which allows one to investigate the detection capability of STEM.

For the STEM investigation of a doping profile, a monocrystalline Si wafer was implanted with Sb at an energy of 23 keV, with a dose of 2×10^{15} atoms/cm^2. After implantation, the sample was annealed at 1000 °C for 2 min. The Sb concentration profile was determined by computer simulation [5]. It resulted that the implanted region extended about 40 nm in depth and the maximum concentration of about 2.5% was estimated at about 17 nm below the surface [6].

Both samples were prepared for cross-sectional observation, following the standard procedure for TEM specimen preparation. The specimens were mechanically grinded, polished, and dimpled. Electron transparency was finally achieved by low-angle Ar milling. Particular care was adopted in order to avoid contamination and to reduce the specimen thickness below 100 nm.

STEM observations were carried out in a LEO 1525 field-emission SEM, operated at 20 keV and equipped with a dedicated detector for collection of the transmitted electrons. The experimental set-up is schematically shown in fig. 1. The focusing and stigmation

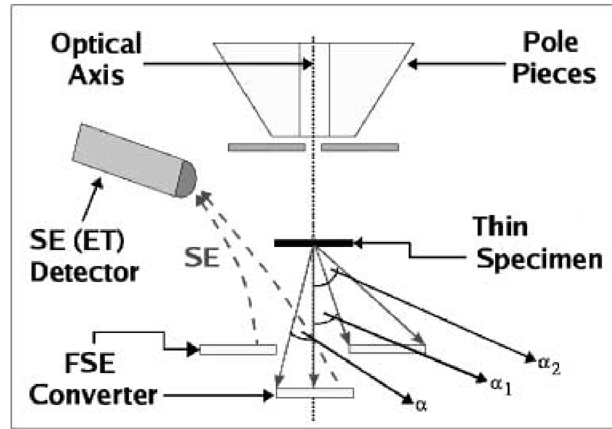

Fig. 1. – Experimental set-up for the detection of transmitted electrons in the scanning electron microscope. The transmitted electrons hit the annular strip covered by magnesium oxide. The off-axis secondary-electron detector measures the intensity of the secondary electrons generated by the transmitted electrons.

of the probe was controlled by optimizing the secondary electron image provided by the in-lens detector of the microscope. The extremely small distance between the thin specimen and the lower polepiece of the electron column allowed imaging of the specimen surface by the in-lens detector and prevented the Everard-Thornley detector, which is located off-axis, from detection of the secondary electrons generated by the specimen. For this reason, this latter detector was used to detect the transmitted beam through a conversion strategy. As visible in fig. 1, the highly energetic electrons of the transmitted beam impinge on a film of magnesium oxide, which yields a number of secondary electrons proportional to the intensity of the beam with conversion efficiency close to unit.

3. – Image formation mechanism

The electron image of the SEM is formed by scanning the finely focused electron beam over the specimen and mapping, point by point, the intensity of the transmitted signal varying as a result of interactions between the beam electrons and the atoms constituting the specimen. Such mechanism of image formation is incoherent, and the output signal or image intensity, $O(r_0)$, pertaining to a detail crossing the specimen as a doped region in a cross-sectioned specimen, is determined by the relation

$$O(r_0) = I(r - r_0) \otimes S(r) ,$$

where r is a position in the sample, $S(r)$ is the specimen response when the probe is located at r_0, $I(r - r_0)$ is the probe intensity at the specimen surface, and $\otimes$ is a convolution over r [7-9].

According to this incoherent sequential imaging procedure, the resolution and contrast of the image are governed by the probe size and by $S(r)$, respectively. Indeed, the resolution is always defined by the lateral dimension of the probe, while the beam broadening affects the contrast, thus determining the feature visibility in the specimen.

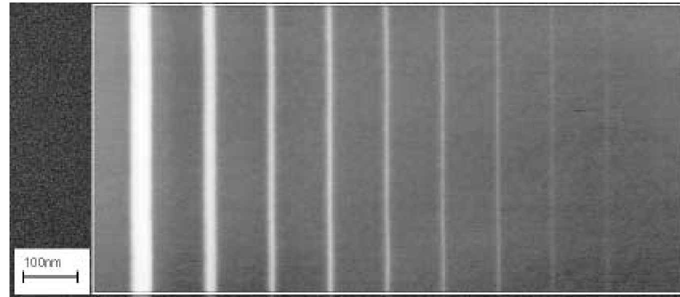

Fig. 2. – STEM dark-field image of the AlAs/GaAs multilayers. The AlAs layers appear brighter than the surrounding GaAs regions because of the higher average Z.

If the spot formed by the beam on the sample surface does not allow atomic resolution imaging, as is the case in low-energy STEM, the image transfer function of the specimen depends on the scattering power averaged over the region from which the mapped signal emanates.

As visible in fig. 1, the geometry of the MgO converter allows an angular selection on the transmitted electrons. Indeed, collection in the 0–α° range determines formation of a bright-field (BF) image as the central, unscattered beam is detected. Differently, the annular detector collects the electrons scattered in the α_1–α_2° range. The dark-field (DF) image formed by these latter electrons features a contrast reversed with respect to the BF image.

4. – Experimental evidence

The imaging of the AlAs/GaAs provides information on the detection capability of the STEM technique. Figure 2 shows the intensity of the electrons transmitted through a very thin region (about 200 thick) of the multilayered specimen. The geometry of the MgO converter provided a DF image. The AlAs layers appear brighter than the surrounding GaAs regions because of the higher average atomic number, Z.

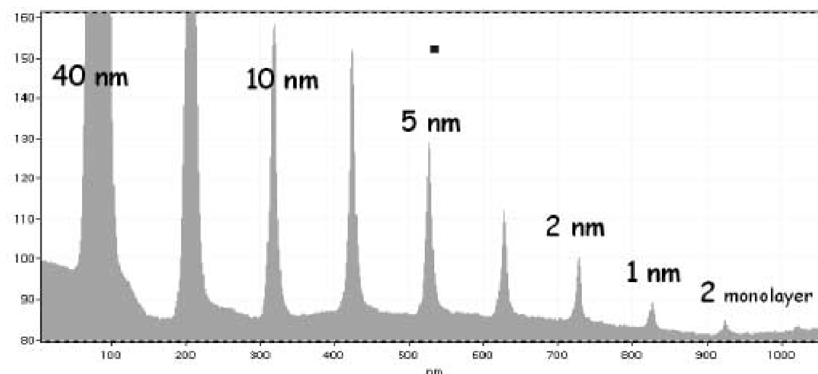

Fig. 3. – Intensity profile of the STEM image shown in fig. 2. The narrowest AlAs layer, which the STEM is capable to detect, is only two atomic layers in width.

Fig. 4. – STEM dark-field image of the Sb-implanted Si specimen. The implanted Sb segregated in small precipitates about 20 nm below the surface. The arrow marks the position of the precipitates.

Figure 3 highlights the contrast variation arising from variation of average Z in the specimen. The narrowest AlAs, which STEM can detect, is only 2 mono-layers in width. The layer appears wider as a consequence of the probe size, about 1.5 nm, which does not affect the detection capability but determines the spatial resolution of the technique.

Such a high performance opens up perspectives of application of the low-energy STEM for characterization of dopants profiles in silicon. Indeed, the present technology of silicon-based devices features chemical non-homogeneities at the nanometric scale. Therefore, most of the information useful for the characterization of a silicon device derive from the investigation of the dopant distribution in areas extending not over a few tenths of nanometers.

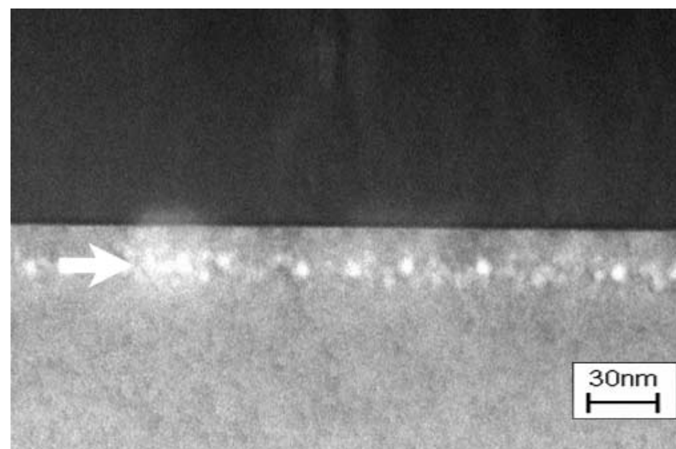

Fig. 5. – STEM-HAADF image of the same specimen presented in fig. 4. The arrow marks the position of the precipitates. Beside the smaller probe size provided by the 200 keV TEM, such an image does not greatly improve the useful information about the implanted profiles already achieved by low-energy STEM.

STEM was therefore carried out over the Sb-implanted silicon specimen. Figure 4 clearly shows that about 20 nm below the surface a Sb-rich region is formed. The bright contrast of the implanted area shows also that formation of Sb precipitates was promoted by the thermal treatment. Such information is highly interesting for the optimization of the implantation parameters. In this case, the dimension of the probe resulted small enough to resolve some important features of the specimen. For the sake of a comparison, the same specimen was investigated by high-resolution TEM. The dark-field image, achieved by scanning a 200 keV beam focused into a sub-nm probe and collecting the transmitted electrons that have been incoherently scattered at high angles, is presented in fig. 5. The two images are directly comparable. Despite the higher spatial resolution achievable by the high-voltage microscope, the information regarding the Sb distribution in silicon is essentially revealed by both techniques.

5. – Concluding remarks

The low-energy STEM has been demonstrated useful for the characterization of dopant profiles in semiconductors. Such technique requires a simple modification of a scanning electron microscope through implementation of a STEM detector. This technique takes advantage of an incoherent imaging procedure and is capable to detect very fine chemical non-homogeneities in crystalline materials.

Further development of the technique will address the optimization of detection geometry and collection efficiency. Such experimental effort should be accompanied by a thorough consideration of the beam-specimen interaction in order to achieve a quantitative description of the specimen features.

* * *

This research is partially supported by the PON SVISENARIA project.

REFERENCES

[1] Rau W. D., Schwander P., Baumann F. H., Hoppner W. and Ourmazd A., *Phys. Rev. Lett.*, **82** (1999) 2614.
[2] Formanek P. and Kittler M., *J. Phys. Condens. Matter*, **16** (2004) S193.
[3] Perovic D. D., Castell M. R., Howie A., Lavoie C., Tiedje T. and Cole J. S. W., *Ultramicroscopy*, **58** (1995) 104.
[4] Elliot S. L., Broom R. F. and Humphreys C. J., *J. App. Phys.*, **91** (2002) 9116.
[5] Lulli G., Bianconi M., Nipoti R., Albertazzi E., Cervera M., Carnera A. and Cellini C., *J. App. Phys.*, **82** (1997) 5958.
[6] Lulli G., personal communication.
[7] Voyles P. M., Grazul J. L. and Muller D. A., *Ultramicroscopy*, **96** (2003) 251.
[8] Merli P. G., Morandi V. and Corticelli F., *Appl. Phys. Lett.*, **81** (2002) 4535.
[9] Merli P. G., Morandi V. and Corticelli F., *Ultramicroscopy*, **94** (2003) 89.