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Experimental Determination and Parametric Study of Depth Resolution Function in Secondary Ion Mass Spectrometry Analysis

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ABSTRACT

The depth resolution function (DRF) of Secondary Ion Mass Spectrometry (SIMS) analysis has been measured by analyzing delta layers of boron in silicon in different experimental condition using Cameca Ims-6f apparatus. This function has been fitted with an analytical expression initially proposed by Dowsett et al. This is described by three parameters: the rising exponential decay, the standard deviation and the decreasing exponential decay. Physical phenomena related to these fitting parameters are well detailed, in particular, falling exponential decay characterizes the residual mixing effect. The latter is considered the main mechanism responsible for the degradation of the depth resolution in the range of the primary ions energy used in this work. It is shown that when the experimental conditions change, the parameters that govern the analytical expression vary in a satisfactory way. Our depth resolutions are compared with those of the literature. It is shown that our results are in good agreement with those of other authors.

Introduction

Secondary ion mass spectrometry, SIMS, is an effective and powerful technique for analyzing small volumes of almost any non-volatile material and for characterizing very thin structures [1-3]. The remaining problem, in SIMS analysis, is the experimental limit of the depth resolution; it is governed by several phenomena inherent to the penetration of the primary ions in the substrate and their incorporation in the matrix.

The depth resolution in SIMS analysis is perfectly well described by the Depth Resolution Function (DRF), which is the response to the analysis of an ideal delta-marker. The physical phenomena involved in the analysis impose that the DRF possess precise properties [2, 3], and lead to an analytical expression of the DRF [3]. The analytical description of the DRF has shown its relevance in the case of the analysis of boron in silicon with an oxygen primary beam. The parameters of the DRF which can be described by the convolution of a Gaussian with a double Exponential (rising and decreasing), reflect perfectly the characteristics of the depth resolution and must be analyzed in experimental conditions [6].

In signal processing field, particularly the deconvolution applied in SIMS framework for enhancement the depth resolution, the knowledge of response function is important [4, 5]. Indeed, SIMS response function (DRF) can be obtained by profiling a single ideal impurity, δ-layer, grown in a substrate material. However, such a δ-layer is an abstraction, and even if it were not, there could be no means to recognize it. A measured response function will have a shape that is determined by a mixture of sample dependent and SIMS related effects. Thin layers of an adequate quality for response function measurement at probe energies can be obtained. But sample related structure becomes progressively a more significant part of a profile as the energy is reduced. This structure can result from steps in the δ-layer across the analyzed area, the statistical distribution of atoms about the ideal depth, surface segregation during growth, and finite diffusion; the contribution of these phenomena provides the shape of the measured DRF [6, 7].

The aim of this paper is to experimentally determine the depth resolution function using different primary energies and to study the validity of its fitting parameters, and to relate them to the physical phenomena which take place during the SIMS analysis. To help the user of the deconvolution, the type of DRF filter is determined for both analytical and experimental response function in the frequential domain.

Depth resolution function

The depth resolution characterizes the precision of a profile. Roughly speaking, it is the range in units of sputtered depth which limits the knowledge of the variation in sample composition. The way to the valuation of the depth resolution is to measure the depth resolution function and to exploit its proprieties. Indeed, the depth resolution function is a specific function that has to be determined for each set of experimental conditions. In the case of a SIMS experiment, the DRF changes each time the primary ion beam energy, the impact angle, the matrix or the impurity under investigation changes.

In the case of the SIMS analysis of boron doped layers in silicon under O_2^+ primary beam and with an oblique incidence angle, the collisional mixing is the responsible for a degradation of the depth resolution (Fig. 1), that can be expressed in terms of a convolution of the original profile with a depth resolution function. This DRF, represented by the function *h*(*z*), is the normalized response of a delta-doped layer when the analysis process is linear and invariant, that is when the resolution function does not vary with depth, which is true for depths ranging from ≈ 15 nm to 800 nm [4]. A real concentration distribution *x*(*z*) will result in a measured profile *y*(*z*) defined by:

$$y(z) = \int_{-\infty}^{+\infty} x(z')h(z-z')dz' + n(z), \tag{1}$$

where *n*(*z*) is the noise which adds independently to the perfect measured profile

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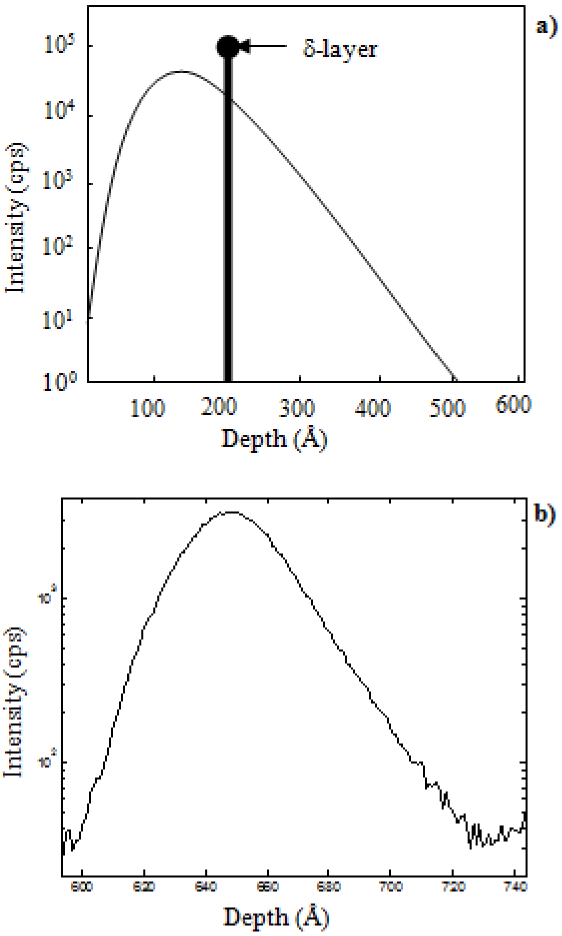

Figure 1. a) Representation of SIMS analysis of delta-layer. b) Colisional mixing effect on the delta-layer of boron in silicon (experimental profile, 3keV/O_2^+)

The linearity of the process is verified so long as the concentration, of the boron-doped layer, stays below the dilute limit. Thus, the assumption on which SIMS analysis can be described in terms of the convolution of an initial profile with a DRF that depends on the instrument and the analysis conditions seems to be valid, as has already been pointed out by Dupuy et al. [6].

From Eq. (1), it is easy to see that if the input signal is a delta function, then $y(z) = h(z)$, which represents the depth resolution function. The DRF can thus be found from the measurement of a delta function. This function has to be determined for each specific combination of matrix, impurity and experimental conditions.

From a practical point of a view, the elaboration of a delta-doped structures of uniformly doped layers with ultra sharp interfaces is better controlled, so that is possible to get the DRF from the measurement of a rapid thermal chemical vapor deposition (RTCVD) grown sample, which can be supposed to be very abrupt and very thin.

Beyond the capabilities of the deconvolution, which are well described elsewhere [3-6], it is possible to take advantage of the analytical properties of the DRF. For example, a partial analytical deconvolution is possible. The precise fitting of the Gaussian parameter of the DRF allows us to measure the broadening of the profiles due to e.g. the diffusion, with a nanometric precision, and using only a medium primary energy SIMS analysis [6]. Other advantages, to use an analytical function that perfectly fits the experimental data, are [8]:

1. If the DRF is experimentally measured for a lot of energies, it is possible to determine the variation of the fitting parameters, versus the energy: a better determination of these parameters for a given energy can be obtained by checking a continuity criterion.
2. The possibility of describing the depth resolution with an analytical expression, via its moments (see Eqs.5, 6), makes the performance of the analysis more comparable for different users working with different apparatus.
3. One is free to choose the extent of the analytical DRF so that the fitting covers only the experimental data (in that case, the dynamic range of the analytical DRF is the same as the experimental one) or to extrapolate the DRF to simulate a very large dynamic range.

In the first case, the use of an analytical function is just a smoothing of the analytical data. It is as well as the case where there is no reason that the analytical form should not be implemented in a convolution or in deconvolution scheme, as claimed by Dowsett et al. [3]. In this sense, it is assumed that the sample is a real data layer (MBE-grown sample are known to be very abrupt) and that the entire response is due to the measurement process.

When looking at a SIMS profile of a delta-doped layer of boron in silicon (Fig.1), we notice that it comprises an exponential trailing (decreasing) edge and a Gaussian-like rounded top. The rising part of a profile is often exponential too, although it is why we have chosen to implement an analytical DRF, initially proposed by Dowsett et al. [3] which is constituted by the convolution of a double exponential function, *D*(*z*), with a Gaussian function *G*(*z*).

$$D(z) = A.\begin{cases} \exp\left((z - z_0)/\lambda_u\right) & z < z_0 \\ \exp\left(-(z - z_0)/\lambda_d\right) & z > z_0, \end{cases} \tag{2}$$

$$G(z) = \left(B/\sqrt{2\pi}\sigma_g\right)\exp\left(-z^2/2\sigma_g^2\right). \tag{3}$$

The result of this convolution is given by the normalized expression [3-5, 7, 8]:

$$h(z) = \frac{1}{2(\lambda_u+\lambda_d)}\begin{pmatrix} \exp\left(\frac{z-z_0}{\lambda_u} + \frac{\sigma_g^2}{2\lambda_u^2}\right)\operatorname{erfc}\left(\frac{z-z_0}{\sqrt{2}\sigma} + \frac{\sigma_g}{\sqrt{2}\lambda_u}\right) + \\ \exp\left(-\frac{z_0-z}{\lambda_d} + \frac{\sigma_g^2}{2\lambda_d^2}\right)\operatorname{erfc}\left(-\frac{z_0-z}{\sqrt{2}\sigma_g^2} + \frac{\sigma_g}{\sqrt{2}\lambda_d}\right) \end{pmatrix} \tag{4}$$

This DRF can be described by three parameters λ_u, λ_d, σ_g and a fourth parameter z_0 that represents the position of the cusp of the double exponential. erfc (z) is the complementary error function defined by : $erfc(z) = \frac{2}{\sqrt{\pi}}\int_z^{\infty} e^{-z^2}dz$.

By using the analytical form of the resolution function, we can determine the mean value of *h*(*z*) by the first-order moment, μ_1, the second-order moment μ_2 represents the depth resolution [6, 8].

$$\begin{cases} \mu_1 = \bar{z} = \lambda_d - \lambda_u \\ \mu_2 = \sigma_{DRF}^2 = \sigma_g^2 + \lambda_d^2 + \lambda_u^2 \end{cases} \tag{5}$$

Knowing that the *k* order of the centered moment of a function *h*(*z*) is defined as:

$$\mu_k = \int_{-\infty}^{+\infty}(z - \bar{z})^k h(z)dz \tag{6}$$

By extrapolating the fitting parameters λ_d, λ_u and σ_g, it is possible to extrapolate the shape of the resolution function, whose its knowledge is essential for the implementation of a deconvolution procedure [5, 7, 8].

Results and discussion

The response function should contain only information on the broadening and the shift (and possibly the ion yield), but must not contain sample dependent features (unless one wishes deliberately to remove such features from the recovered data). Ideally, the response function is the transient measured from an infinitesimally thin layer (or at least a single atomic plane, *δ*-

layer). However, the intrinsic resolution of SIMS improves through the use of sub-keV probes [9]. A = 50.46 and n = 0.665

For this experimental study, we have chosen to measure the resolution function of boron in silicon under various experimental conditions of energy and angle of incidence (The energy varies from 3.5 keV to 10.5 keV and the angle from 45° to 36°); boron is presented in the form of deltas-layers. Each sample is characterized by the concentration of boron and the number of deltas and the distances between them. The advantage of determining the DRF in homogeneous structures with low concentrations of dopants that one overcomes the variations in the sputtering and the ionization yields with the sample composition (matrix effect). In these conditions, the distortions of the profiles due to the matrix effect are absent; it is therefore easier to study the alterations of concentration profiles, which are only due to the analysis process.

The experiments have been carried out with a Cameca Ims-6f apparatus, with O_2^+ primary ions beam, collecting the positive secondary ions on diameter area of 70 µm (diaphragm field 2 contrast diaphragm 1) and a scanning 450 µm x 450 µm, and 70 nA of the primary current, which ensuring a good focusing. With these operative conditions we consider that the collisional mixing as the only source of degradation of the depth resolution because the segregation mainly due to the formation of SiO_2 stoichiometric is absent. Furthermore, the depths of analysis are enough low to cause an induced roughness.

Figure 2 shows a sample of nine δ-layers. On this sample the depth resolution function is measured on the peaks that have the higher dynamic range (peak 9, peak 8 and peak 5) thus allowing to fit the exponential slopes with a good accuracy.

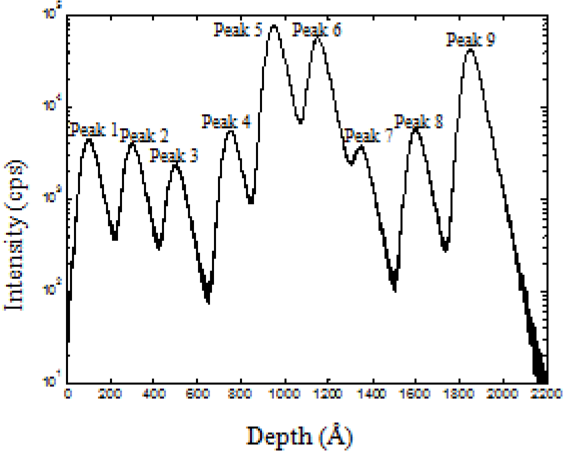

Figure 2. SIMS profile of boron in silicon (9.5 keV/O_2^+, 40.9°). A version of the sample in which the DRF is measured

Figure 3 shows the fitting of the depth resolution function for two experimental conditions 5.5 keV/O_2^+, 48.3° and 9.5 keV / O_2^+, 40.9°.

Results of the fitting parameters are not expressed with respect to the primary ion energy but versus the primary ion range, R_P, given by the formula derived from SRIM simulation using O^+ ions with the same impact angles [6]:

$$R_p\,(\text{Å}) = A\,E_p^{\,n}\cos\theta \tag{7}$$

Where θ is the implicit angle of the beam and E_P is the primary energy per incident oxygen ion. This representation has been chosen in order to have synthetic results taking into account both energy and the angle of impact, which are not independent in a magnetic sector instrument. The values of A and n have been calculated in the case of O^+ and Si which are: A = 49.26 and n = 0.740. In the same conditions, Gautier et al. [8] found A = 50.46 and n = 0.665.

So the Eq. (7) takes the proposed following form:

$$R_p\,(\text{Å}) = 49.26E_p^{0.740}\cos\theta \tag{8}$$

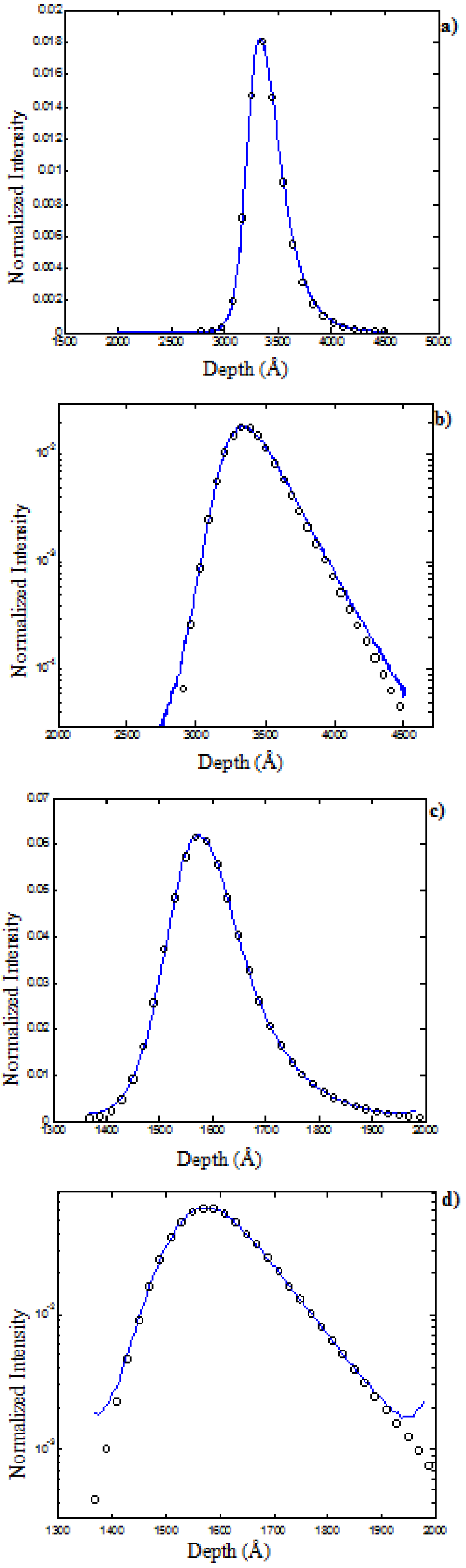

Figure 3. Result of the Fitting of the DRF by an analytical form. Full lines: experimental curve; circles: fitting. (a, b): 5.5 keV/O_2^+, 48.3°; (c, d): 9.5 keV / O_2^+, 40.9°

Figure 4 shows the variations of the DRF with the primary ions energy. When the primary ions energy increases, we clearly distinguish the evolution of the shape of the DRF. This is characterized by the flattening of the round top and the decrease of the exponential slope of the DRF, following hence a behavior increasingly longer. Moreover, we notice that when the energy of the primary ion decreases, the noise is more pronounced in the measured profile, this is due firstly to the size of the ion probe at low energies, secondly to the wrong focus of this probe (crater edge effect).

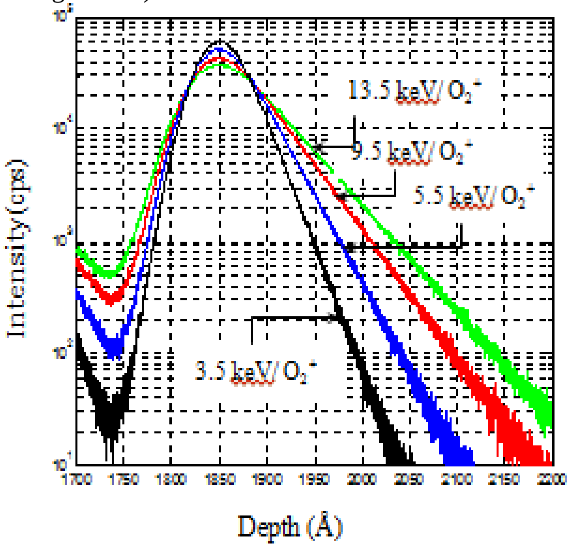

Figure 4. Evolution of the shape of the DRF with the primary ion energy

The variations of R_P with respect to the primary ion energy E_P and the angle of impact are illustrated in Fig. 5.

The variation of the fitting parameters when changing the experimental conditions conforms to the behavior of their physical homologues (Fig.6). We notice in particular the increase of λ_d and σ_g with R_P. However, λ_u presents a chaotic behavior with values almost constant varying from 3 to 8 Å around an average of about 5 Å. This parameter is not sensitive to the variations of R_p, its large values correspond to low energies, one suggests so that at these low energies other phenomena are involved for example the induced roughness. Gautier et al. [8] suggest that this parameter is linked to the instrumental conditions and may vary, among other parameters, with the shape of the beam. Our results confirm the results obtained from Dowsett et al. [3, 7] and Gautier et al. [4, 8].

In the range of energy from 1.5 to 13 keV Gautier et al. [8] studied the evolution of fitting parameters, he also obtained a linear behavior of the three parameters, except for energies below 3.5 keV (i.e. R_p <47.9 Å). He attributed the differences to parameters for these low energies to the difficulty to focus the primary ion beam and the effects of grazing impact angle which lead to asymmetric and inclined crater. In fact, the significant noise present in its experimental profiles makes the evaluation of exponential slopes much less accurate.

We notice that the ordinate value at the origin of each fitting parameter is not null. One could yet think that when the primary ion range vanishes (nonexistent collisional mixing) the resolution will be perfect canceling hence the value of all fitting parameters. Indeed, it is possible that some parameters that degrade the resolution are insensitive to primary ions energy, particularly those which are related to the roughness of the sample as well as phenomena related to the statistical nature of sputtering. These phenomena determine the ultimate depth resolution accessible by SIMS analysis.

The comparison of our depth resolutions with those of the literature is summarized in Fig.7. According to this compilation of results, we can see that our results are in good agreement with those of other authors, particularly those of Meuris et al. [10] and Smirnov et al. [11], the best results are obtained by Gautier et al.[8]. The difference in results is certainly due to the quality of the samples (manufacturing processes of delta-layers), the analysis conditions, type of the used apparatus, etc. In reality, it is not obvious to make such absolute comparison as there are several types of apparatus and each team can customize its own apparatus by changing its characteristics. Nevertheless, the comparison of our boron resolution functions in silicon with those obtained for other species in silicon clearly show that the depth resolution depends on the type of dopant, although the theory does not provide for variations in the case of diluted dopant [7-12].

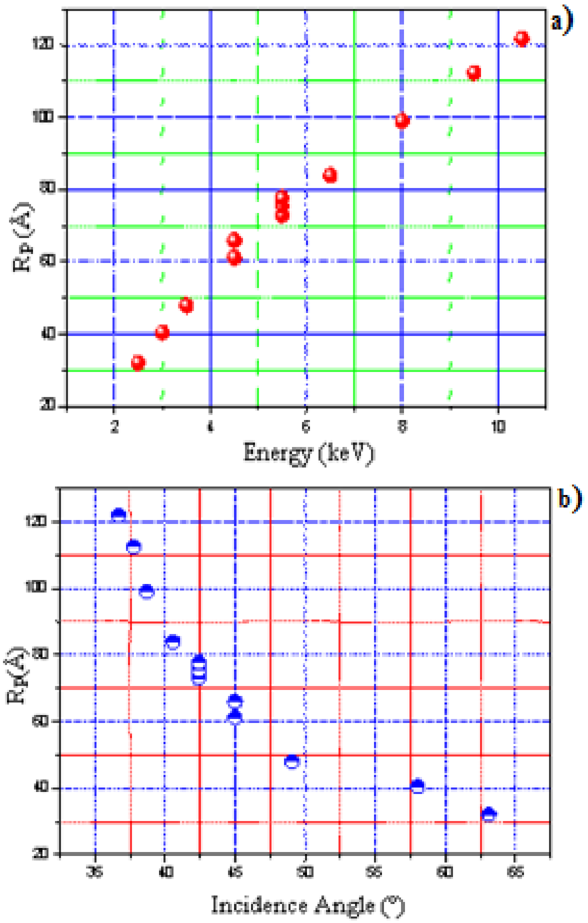

Figure 5. (Color online) a) Evolution of the primary ion range, R_p, with the ion beam energy. b) Evolution of the primary ion range, R_p, with the incidence angle

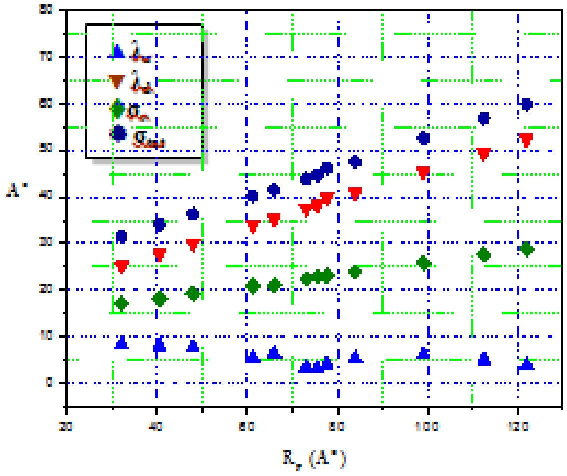

Figure 6. (Color online) Evolution of the fitting parameters of the DRF with the primary ion range R_p

With a full modeling, the ideal fitting of the DRF parameters results in an analytical form linking all physical phenomena since the primary ion beam until the detection of secondary ions. Unfortunately, this is not always the case because there is no way to link unambiguously the three fitting parameters. In addition, the sputtering phenomenon is so far to completely be understood; mechanisms are too dependent on the chemical species of the matrix species, incidental species and their relative concentrations, and the large number of experimental parameters managed by the operator. Sometimes the sensitivity of all physical and instrumental parameters makes difficult the reproducibility of measurements. Nevertheless, the energy dependence of the fitting parameters can allow us to suppose some assumptions about their physical nature.

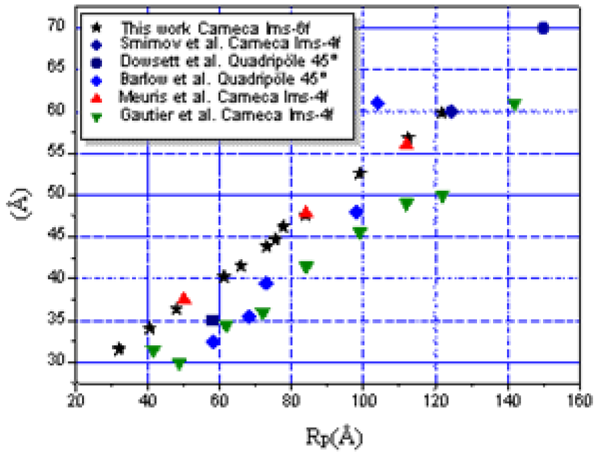

Figure 7. (Color online) Comparison of depth resolution of this work with those of the literature [7,8, 10-12]

As we have seen in the previous section the origin of λ_d is the collisional mixing. The evolution of this parameter with R_p corresponds well to the model of collisional mixing, except at low energy where other phenomena involve. The parameter σ_g is related to the cascades of isotropic collisions that take place within the material, and increases with the extent thereof. Littmarck et al. [13] shown that even under experimental conditions assumed ideal: homogeneous scanning, flat bottom of crater, no roughness, etc. the rounded top of the DRF exists and depends on R_p. However, the parameter λ_u remains ambiguous, because it is insensitive to the energy of the primary ions. Turner et al. [15] affected this parameter the exhaust depth of secondary ions. In all cases, this parameter seems not to be dependent on the collisional mixing. A particular study in low primary energies was addressed by Fares et al. [9] Badheka et al. [14] and Tomita et al. [16], in which they considered that this parameter strongly related to the microscopic roughness of the sputtered surface at the bottom of crater, in this case the DRF varies with the eroded depth (variant DRF).

Figure 8 shows the spectrum of the DRF experimentally determined at 9.5 keV / O_2^+ and the spectrum of its fitting by an analytical form. The two spectra are composed of a main peak (low frequency) which falls very rapidly to zero. Above a certain cut-off frequency υ_c, the other components vanish. Consequently, the function H(υ) (the spectrum of the DRF) is a low-pass filter [5]. This is an important characteristic that helps the user of deconvolution to resolve the ill-posed problem of the SIMS system, hence to retrieve the shape of the original profile broadened by the analysis and so to push the depth resolution to its ultimate limit.

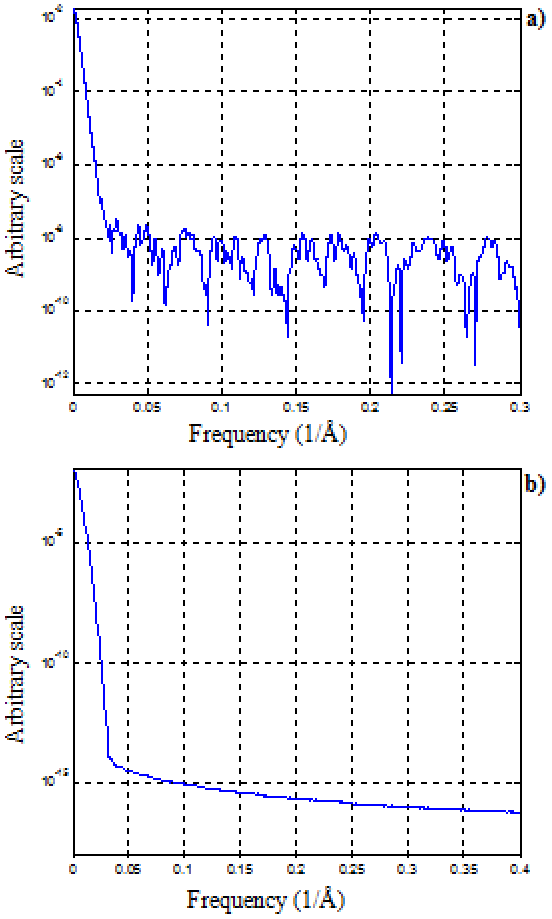

Figure 8. Spectrum of the depth resolution function. a) experimental; b) analytical

Conclusion

In this paper, a detailed study of the depth resolution function (DRF) has been conducted since its generation by the analysis of delta-layers until its fitting by an analytical expression. The SIMS profiles were obtained by analysis of delta-layers of boron in silicon with an oxygen primary beam using Cameca Ims-6f apparatus.

We have seen that in the range of high energies, the main source of degradation of the depth resolution is of ballistic nature, the collisional mixing. The behavior of the three fitting parameters of DRF was determined and expressed as a function of the primary ion range. Two of the three parameters (the decreasing exponential and the standard deviation of the Gaussian) showed a linear behavior with the primary ion range, while the third parameter (the rising exponential) showed a chaotic behavior. The comparison of our depth resolutions with those of the literature shows that our results are in good agreement with those of other authors. Finally the spectrum, in both cases analytical and experimental, of the DRF is determined which shows that the DRF is a low pass filter. This leads to take advantage of the analytical properties of the DRF in deconvolution framework to push the depth resolution beyond its instrumental and physical limits.

References

[1] P. A. Yunin, Y. N Drozdov, and M. N. Drozdov, "A new approach to express ToF SIMS depth profiling surface and interface analysis", Surf. Interf. Anal., vol. 47, pp. 771-776, 2015.

[2] S. Hofmann, "Sputter depth profiling: past, present, and future", Surf. Interf. Anal., vol. 46, pp. 654-664, 2014.

[3] M. G. Dowsett, G. Dowlands, P. N. Allen, and R. D. Barlow, "An analytic form for the SIMS response function measured from ultra thin impurity layers", Surf. Interf. Anal., vol. 21, pp. 310-315, 1994.

[4] B. Gautier, G. Prudon, and J. C.Dupuy, "Toward a better reliability in the deconvolution of SIMS depth profiles", Surf. Interf. Anal., vol. 26, pp. 974-983, 1998.

[5] M. Boulakroune, D. Benatia, and T. Kezai, "Improvement of depth resolution in secondary ion mass spectrometry analysis using the multiresolution deconvolution", Japanese Journal of applied physics, vol. 48, pp. 066503-1,15, 2009.

[6] J. C. Dupuy, G. Prudent, C. Dubois, P. Warren, and D. Dutartre, "Depth resolution in SIMS study of boron delta-doping in epitaxial silicon", Nuclear Instruments and method for physic Research. Vol. 85, pp. 379-382, 1994.

[7] M. G. Dowsett and D. P. Chu, "Quantification of secondary ion mass spectroscopy depth profiles using maximum entropy deconvolution with a sample independent response function", J. Vac. Sci. Technol B. vol. 16, pp. 377-381,1998.

[8] B. Gautier, J. C. Dupuy, R. Prost, and G. Prudon, "Effectiveness and limits of the deconvolution of SIMS depth profiles of boron in silicon", Surf. Interf. Anal. Vol. 25, pp. 464-477, 1997.

[9] B. Fares, B.Gautier, J. C. Dupuy, G. Prudon, and P. Holliger, " Deconvolution of very low energy SIMS depth profiles", Applied Surface Science. Vol. 252, pp. 6478-6481, 2006.

[10] M. Meuris and W. Vandervost, "On the effect of an oxygen beam in sputter depth profiling", J. Vac. Sci. Technol., vol. 9, pp. 1482-1488, 1991.

[11] V. K. Smirnov, S. G. Simakin, E. V. Potapov, and V. V. Makarov, "SIMS depth profiling of delta-doped layers in silicon", Surf. Interf. Anal., vol. 24, pp. 469-475, 1996.

[12] R. D. Barlow, "Redistribution of Ge in the SIMS altered layer during normal incidence O_2^+ bombardment of SiGe alloy material", In: SIMS VIII proceeding of the SIMS international conference, 1991, A. Benninghoven ed. New York: Wiley, pp. 359-362, 1992.

[13] U. Littmark and W. Hofer, "Recoil mixing in solids by energitic ion beams", J. Nucl. Instr. Meth. Phys. Res B. Vol. 168, pp. 329-342, 1980.

[14] R. Bedheka, M. Wadsworth, J. A. Van Den Berg, and D. G. Armour, "Theoritical calculations of dilute B profiles in Si using IMPETUS", In International Conference on Secondary Ion Mass Spectrometry 1993: Yokohama, Japan (SIMS IX), A. Benninghoven ed. New York: Wiley, pp. 219-222, 1994.

[15] J. E. Turner, H. Keller, and D. E. Mars, "High depth resolution SIMS by deconvolution of the instrumental function", In: International Conference on Secondary Ion Mass Spectrometry 1989: California, USA (SIMS VII), A. Benninghoven ed. New York: Wiley, pp. 95-98, 1990.

[16] M. Tomita, H. Tanaka, M.Koike, M. Yoshiki, S. Takeno, N. Sugita, and M. Obuchi, "MEM-deconvolution analysis of ultra-shallow junction depth profiles", In SIMS XV proceeding of International Conference on Secondary Ion Mass Spectrometry 2005, Manchester, UK, Elsevier, 235-240, 2005.