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CRITICAL EVALUATION OF CALIBRATION PROCEDURES FOR DISTRIBUTION ANALYSIS OF DOPANT ELEMENTS IN SILICON AND GALLIUM ARSENIDES†

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Critical evaluation of calibration procedures for distribution analysis of dopant elements in silicon and gallium arsenides

Abstract – Strategies for highly accurate quantitative distribution analysis of the most important dopant elements in silicon (B, As) and gallium arsenide (Si) by combination of various methods with different systematic errors are discussed. Analytical figures of merit are given for the methods applicable to a specific problem evaluating Secondary Ion Mass Spectrometry (SIMS), Rutherford Backscattering Spectrometry (RBS), Neutron Activation Analysis (NAA), Charged Particle Activation Analysis (CPAA), electrical and magnetic measurements.

1. INTRODUCTION

The major techniques for distribution analysis of dopant elements in silicon and gallium arsenide are SIMS, NAA, RBS, electrical and Hall effect measurements. The information content, depth resolution, dynamic range, detection power and accuracy of these methods is described in the report of IUPAC commission V.2 "Evaluation of Analytical Methods for the Determination of Trace Elements in Various Matrices VII. Trace Analysis of Semiconductor Materials: Part B: Distribution Analysis" [1].

This report therefore centers on the topic of combination of these different techniques for obtaining the distribution (depth profile) of a trace element with maximum accuracy. The combination of various techniques with different physical principles, consequently different systematic errors, allows to determine the accuracy of a single technique used routinely for distribution analysis (e.g. SIMS) and to characterize defined materials sufficiently that these can be used as reference materials. Such approaches have been applied successfully for bulk characterization of a large number of materials.

For distribution analysis of semiconductors only limited efforts concerning the determination of systematic errors and consequently accuracy have been undertaken (e.g. by the American Society for Testing of Materials [ASTM], the National Bureau of Standards [NBS], the Community Bureau of Reference [BCR] and the National Physics Laboratory [NPL], Teddington, UK). One of the reasons for this is certainly the small number of methods available, their often narrow range of applicability and rather large systematic errors. One of the major tasks at the present state of experience is therefore the development of a suitable strategy for the combination of various techniques for a specific analytical goal (e.g. the distribution analysis of B in silicon) to achieve a most accurate characterization of a material.

2. STRATEGIES FOR DISTRIBUTION ANALYSIS OF SEMICONDUCTOR MATERIALS BY COMBINATION OF VARIOUS METHODS

2.1 General requirements

The general requirements for distribution analysis are [2]:

- large dynamic range of analysis and high detection power: 10^{14} – 5.10^{21} cm^{-3} (1 ng/g - %)
- high spatial (depth) resolution : $\Delta z = 1 - 10$ nm
- high accuracy of distribution information (concentration vs. depth)

In order to be able to combine methods of elemental analysis with electrical measurements for reference purposes the trace elements have to be in an electrically active state which demands annealing if doping has been performed by ion implantation. For non-annealed implant samples, the calculation of the distribution of the dopant by mathematical-physical models is another feasible way for checking experimental results. The distribution can be described in terms of statistical distribution functions, such as the Gaussian (2 moments) or the Pearson IV (4 moments) [3,4] or by Monte Carlo simulation [5,6]. Since, however, the accuracy of calculated distributions has to be determined experimentally these methods cannot be seen as reference methods, but rather as supplementary techniques to assess the accuracy of a measurement.

2.2 Boron in silicon

Boron is the most important p–dopant element for silicon [7]. The most widely used method is SIMS: Detection limits of 10^{14} cm^{-3}, a dynamic range of 10^7 and an accuracy of 5 – 10 % rel. in the concentration scale and a few nm in the depth scale when stylus techniques or interference microscopy are applied for crater depth measurements are typical. Both isotopes of B can be analyzed [8–10].

NAA using the $^{10}B(n,\alpha)^7Li$ reaction and measuring the energy distribution of the α–particles to obtain depth information is the most prominent reference technique with excellent figures of merit : accuracy ca. 1 % rel., detection limit 10^{14} cm^{-3}. The depth resolution (10 – 50 nm) and dynamic range (10^3) are inferior to SIMS which limits the use of NAA as a reference method either at high or low concentrations. NAA is also very useful for the analysis of high concentrations since it does not exhibit any matrix effects [11–14].

Generally excellent agreement is obtained between the two methods for B in silicon (see Fig. 1 [15]). A disadvantage of NAA is its limitation to ^{10}B since normally ^{11}B is used for devices.

Both methods need external standards for calibration or knowledge of the dose of implanted particles. For SIMS only silicon substrates with B doping certified by another technique can be used.

NAA is able use thin film standards (e.g. evaporated boron films [16]) or homogeneous materials like a boro–silicate glass as offered by NBS. These standards enable also to control the fluence of implanted particles. This fluence is usually obtained from measurement of the time integral of the ion current during implantation, but has to be corrected for backscattering effects, especially for light elements like B [17].

Another technique for determining the dose is CPAA using the $^{10}B(p,\alpha\gamma)^7Li$ or $^{11}B(p,\gamma)^{12}C$ reaction [18].

As electrical techniques, spreading resistance (SR) measurements of angle lapped specimens and surface resistance measurements in combination with electrochemical or chemical removal of thin surface layers (4–point technique, also called differential sheet resistance measurements) are suitable for distribution analysis of B in silicon [7,19–21]. SR offers in general some excellent features like a detection limit of 10^{14} cm^{-3}, a depth resolution of ca. 10 nm (for very small angles of lapping – e.g. 10 minutes), a dynamic range of 10^6 and an accuracy of about 10 % rel. Differences of elemental profiles are encountered at concentrations below 10^{18} cm^{-3} [20] and at high concentrations due to the presence of inactive dopant elements if the electrical solubility limit ($2 \cdot 10^{20}$ cm^{-3} for B in silicon annealed at 1000 ° C [15]) is exceeded. The major reasons for the differences encountered between electrical and elemental profiles at concentrations below 10^{18} cm^{-3} are: sensitivity of spreading resistance to lattice damage and degradation of carrier mobilities; dependence of the relation between carrier density and resistivity on the carrier distribution and interactions of mobile charge carriers and the immobile charged dopant atoms [20]. These differences depend on type and concentration of the dopant atoms

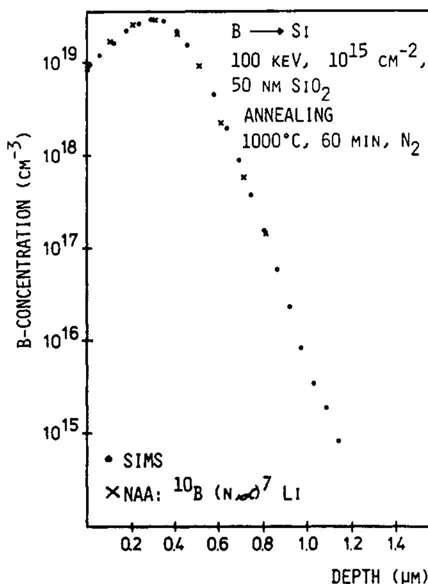

Fig. 1 : Comparison of quantitative depth profiles of B in silicon obtained with SIMS (•) and NAA (x). Implantation through 50 nm SiO_2, E = 100 keV, $Q = 10^{15}$ cm^{-2}, annealing at 1000 ° C, 60 min, N_2 [15].

[21–24]. In comparison to SIMS steeper SR profiles have usually been found which can be explained by the electrical effects described above and profile broadening in SIMS due to recoil phenomena [25–29].

SR–measurements have to be calibrated by external standards. Material from NBS (e.g. (111)Si surface resistance reference material) is available. Conversion of the spreading resistance data has to be performed with suitable algorithms [30] following, e.g. ASTM standard practice [31].

The 4–point method used in combination with the removal of discrete surface layers exhibits the disadvantage that it is very time consuming (ca. 2 days per sample), that it can only be used with n–type substrates and surface damage of the substrate can occur due to the large weight of the probe (ca. 40 g). Also its detection limit is only $10^{17} - 10^{18}$ cm^{-3}. Furthermore anodic oxidation can cause migration of the dopant atoms causing a severe systematic error for the distribution representation. Chemical etching (H_2F_2/HNO_3) has to be prefered for this reason. The strength of the 4–point method lies in the possibility of the determination of the lateral homogeneity of dopants in a wafer by measuring the sheet resistivity as a function of localization. A high accuracy (1 - 2 %) for such homogeneity measurements is achievable.

Conclusion:
For distribution analysis of B in silicon the best combination of methods is SIMS, NAA, SR.

2.3 Arsenic in silicon

Arsenic is the most important n–dopant element for silicon [7]. SIMS is the standard technique. Due to the availability of Cs^+–ion sources the interference problem encountered in the use of oxygen primary ions as a consequence of the formation of Si_2O–ions with the same mass as As (M = 75) has been greatly reduced [32]. Furthermore Cs^+ bombardment causes a very high yield of negative secondary ions. In the case of As in silicon the secondary ions As^- and $AsSi^-$ deliver the highest sensitivity [32]. A detection limit of $3 \cdot 10^{15}$ can be achieved for distribution analysis [33,34]. A dynamic range of 10^6 can be obtained with SIMS.

Quantification is performed using the same principles as for B. For the integration method [35,36] the dose measurements obtained by integrating the implantation current are of higher accuracy due to smaller backscattering effects.

The accuracy of distribution analysis with SIMS seems to be of the same order as for B, but this has to be subjected to further evaluation.

A potential reference technique is NAA [11]. The dose of the dopant element can be determined with high accuracy (2 – 5 %) by measurement of the γ–radiation emitted from the daughter nucleus ^{76}As of the process $^{75}As(n,\gamma)^{76}As$. The detection limit for the determination of the dose is $10^{16} - 10^{17}$ cm^{-3}. Distribution information can be obtained by combination with anodic oxidation and chemical removal (H_2F_2) or etching (H_2F_2/HNO_3) and subsequent measurement of the γ–radiation in the solutions containing the material removed. For achievement of high accuracy a simultaneous determination of Si in the solution is preferable. The detection limit is about 10^{17} cm^{-3} (depending on depth resolution), the depth resolution in the order of 50 nm for distribution analysis in the low concentration range [11,16].

Major disadvantages of distribution analysis of As in silicon with NAA are a severe migration of As during anodic oxidation (Fig. 2) and large time consumption for this tedious procedure. As a consequence NAA is used only for reference purposes and if absolutely required.

Also differences between SIMS and NAA profiles at larger and lower concentrations are encountered which need further investigations (Fig. 3 [15]).

RBS [37] exhibits a low dynamic range, precision and accuracy for distribution analysis of As in silicon [38] but provides possibilities for an accurate determination of the dose. For dose calibration, the backscattering yield from the dopant atoms is either compared with an external standard or with the height of the backscattering continuum from the matrix atoms in the same measurement, making use of tabulated stopping power values. For both methods an accuracy of about 2 % in dose measurements can be expected if channeling effects are carefully avoided [39]. For distribution analysis by measurement of the energy distribution of the backscattered helium ions a rather poor detection power is encountered: detection limit $10^{18} - 10^{19}$ cm^{-3} [16,38]. Also depth resolution is significantly lower than that of SIMS which leads to profile broadening. Channeling effects have to be avoided by

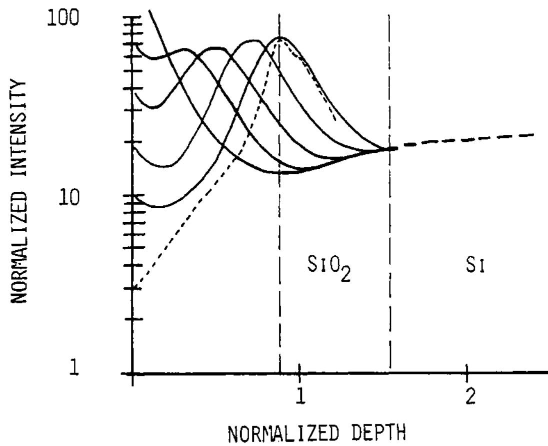

Fig. 2 : Migration of As in silicon during anodic oxidation : - - - original profile, — profiles obtained after repeated anodic oxidation. The depth scale has been normalized such that the surface is always at zero depth [15].

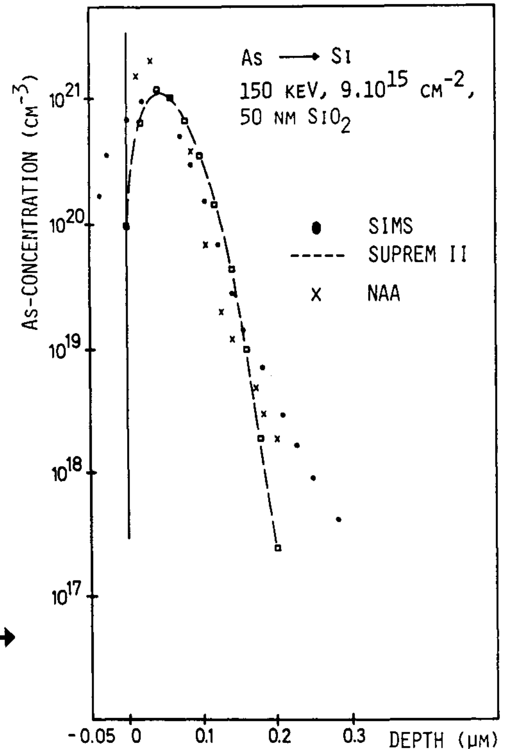

Fig. 3 : Comparison of quantitative depth profiles of As in silicon obtained with SIMS (•) and NAA (x) (supplemented by calculated distribution with SUPREM II–05 ( )). Implantation through 50 nm SiO_2, E = 150 keV, $Q = 3 \cdot 10^{15}$ cm^{-2} [15]. ⟶

producing a random orientation between substrate and helium ion beam. As electrical measurements SR and the 4–point technique can be used with characteristics similar to boron; the upper limit is determined now by the formation of As clusters. The electrical solubility of As is then $2 \cdot 10^{20}$ cm^{-3} for annealing at 1000 ° C [15]. Differences between spreading resistance and SIMS profiles occur to a similar extent as for B [21,22,38,40,41].

Conclusion :
The major combination for quantitative distribution analysis of As in silicon is SIMS and SR. NAA can be used as a reference method if absolutely necessary. RBS is useful for high dopant concentrations but suffers from poor depth resolution.

2.4 Silicon in gallium arsenide

Silicon is an important n–dopant element for gallium arsenide [7]. For distribution analysis with SIMS the major problem is that there may be interferences between ^{28}Si (which usually used as a dopant) and molecular ions, mainly originating from the ion source and the vacuum (^{28}CO,$^{28}N_2$...). For analysis of Si with primary oxygen ions, mass separation of the ion beam is necessary. Furthermore ultra high vacuum conditions are required to reduce the background. Residual interferences are eliminated by energy filtering or measurement in high mass resolution [29, 32, 42]. With signal excitation by oxygen ions Si^+ is measured. Using a Cs^+ beam Si^- or $AsSi^-$ are registered. Interferences are then negligible. Due to the high yield of these Si–ions a detection limit of $4 \cdot 10^{14}$ [29] resp. $7 \cdot 10^{13}$ [43] for Si^- and $3 \cdot 10^{15}$ cm^{-3} [44] for $AsSi^-$ can be achieved. The accuracy of SIMS distribution analysis of Si in gallium arsenide is about 20 % rel. [42].

NAA is very limited as a reference method since only ^{30}Si can be analysed via ^{31}Si following the reaction ^{30}Si (n,γ)^{31}Si. Depth information has to be obtained by chemical etching and analysis of the solutions. The detection power is rather poor.

CPAA enables distribution analysis of ^{28}Si, ^{29}Si and ^{30}Si using proton resonance reactions at 3,1 MeV, 1,37 MeV and 0,62 MeV respectively leading to the emission of γ–radiation in the range between 1,779 and 7,8 MeV. Depth profiling is performed by variation of the energy of the incident protons. A depth resolution between 60 and 200 nm at the surface is achieved. The major disadvantage of the method is its poor detection limit, about 10^{19} cm^{-3} for ^{28}Si [18]. However, in principle, this method should be suitable as a reference method for SIMS at high concentrations. RBS is not suitable for precise dose determination or distribution analysis of Si in gallium arsenide because the backscatter

signal originating from a light element in a heavier matrix is superimposed on a large background from the matrix. For calibration of implant doses with RBS a lighter substrate – like carbon – can be used [16].

For an accurate dose determination of Si in gallium arsenide quantitative trace analysis after chemical dissolution of the gallium arsenide layer which contains the implanted silicon seems to offer a good potential – but only for rather high doses ($> 10^{15}$ cm^{-2}). An accuracy of a few percent is achievable for high silicon concentrations in gallium arsenide [45]. There is still significant potential for improvement in regards to the detection power which is presently limited by high blank values. This technique – if fully developed – offers at least a basic potential for distribution information if only thin layers are removed by chemical etching.

The general problem of all techniques for elemental analysis with the exception of SIMS is the lack of detection power. This is a particularly severe disadvantage since the Si concentrations in gallium arsenide devices are rather low ($< 10^{17}$ cm^{-3}).

Electrical measurements of dopant profiles of silicon in gallium arsenide exhibit the problem of high contact resistances and the often partial inactivation of the dopant element [46]. Therefore accurate agreement between electrical measurements and elemental analysis cannot be expected. As an alternative capacity potential (C/V) and Hall effect measurements can be applied [47].

The C/V method can only be used for the concentration range $10^{14} - 10^{17}$ cm^{-3}. Depth resolution is rather poor, thus it is not possible to characterize steep profiles.

Hall effect measurements are applicable in combination with chemical etching, for depth profiling within a concentration range of $10^{15} - 10^{19}$ cm^{-3}. A depth resolution of 10 nm is possible for favourable cases [48]. The total uncertainty for the concentration scale in the profile is estimated to be in the order of 20 % (due to uncertainties in the scattering factors).

Conclusion :
SIMS is the technique with by far the greatest potential. For the assessment of accuracy of distribution analysis combination with NAA, CPAA and Hall effect measurements (if dopants are electrically active) for ^{30}Si at high concentrations and with C/V–resp. Hall effect measurements at low concentrations is possible. In any case comparatively large systematic errors of these methods have been taken into account. Systematic further studies are necessary to increase the accuracy of quantitative distribution analysis of silicon in gallium arsenide.

3. GENERAL CONCLUSION

Further intensive intercomparison of the most important methods for distribution analysis of dopants [22,26,34,38,40,41,49] is necessary. Round robin studies [50,51] must be performed to assess the accuracy of these methods more precisely and to enable the individual analyst to discover systematic errors in his laboratory. Certified reference materials have to be produced.

All of these investigations and production of material then has to be extended to heterogeneous structures – e.g. SiO_2/Si, $Si_3N_4/GaAs$, $GaAl_xAs_{1-x}$. There the problems become even much more difficult.

The potential of Secondary Neutrals Mass Spectrometry (SNMS) using plasma [52] or resonant/non-resonant laser ionization [53–55] has to be studied thoroughly in this respect.

Generally international cooperation in the measurement science of surfaces has to be increased. Strong efforts are presently being undertaken, e.g. within the Versailles Project on Advanced Materials and Standards (VAMAS) [56,57].

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