Auger analysis of high-dose ion-implantation of arsenic in silicon

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Abstract. The paper deals with Auger electron spectroscopy (AES) analysis of high-dose ion-implantation of arsenic in silicon. Six profile distributions are obtained with doses covering uniformly the range 2×10^{16} - 2×10^{17} atoms/cm². The peak from the matrix (Si) is only monitored; it is assumed that the alteration in its intensity is due to the other element of the binary composition (As). This method of quantification is applicable to ion implantation. The profile depth is calibrated by Rutherford backscattering spectroscopy (RBS). The Ar ion (3 keV) sputtering rate depends weakly on the dose.

1. Introduction
Ion implantation (IIm) is a basic method for modifying surfaces of materials. However, studies by AES of the depth distribution of the introduced implants is limited to high-dose implantation because of the comparatively low detection threshold of the method. But it is precisely in such cases where AES is very useful, as the accompanying processes (ion sputtering, amorphization, recrystallization) make the experimental data critical in modeling the implantation.

About 145 scientific articles have been devoted to the research on IIm and related problems by means of AES. The first ones (for P in Si and F in GaAs) date from 1972; by 1980, their number has become 11, and has monotonically increased afterwards. However, for As implanted in Si, literature is scarce. In [1-3], the distributions of the implant are presented, but the doses do not surpass 1×10^{16} atoms/cm². The quantification has always been performed using the elemental sensitivity factors method (ESFM).

This work presents the experimental distributions of As in Si for the dose range 2×10^{16} - 2×10^{17} atoms/cm² obtained through AES profiling by argon ion sputtering. Additional Rutherford backscattering spectrometry (RBS) data are used to calibrate the profile depth. The way we apply AES can be of interest in other cases of high-dose IIm.

2. Experimental

2.1. Samples
Arsenic is implanted in silicon at energy 40 keV with six doses (2×10^{16}, 4×10^{16}, 8×10^{16}, 1.2×10^{17}, 1.6×10^{17}, 2×10^{17} atoms/cm²), well covering the range (2×10^{16} - 2×10^{17}). More details for the samples preparation can be found in [4, 5], which works are also the source of the RBS analysis data.
2.2. Auger analysis and experiment
The AES analysis is performed by a microprobe (including a cylindrical mirror analyzer with a coaxial electron gun) mounted in a stainless-steel bell at a working pressure of 1.3×10⁻⁸ Pa. The analyzer’s energy resolution is about 0.3%; a 0.5-μA 3-keV electron beam with a diameter 10 μm directed normally to the surface is used. The spectra are differential; the modulation voltage is 4 V p.t.p. The profiling is performed by a 3 keV Ar⁺-ion sputtering. The ion beam is incident at an angle of 79° to the surface normal. Only the silicon peak is monitored during the continuous sputtering. To guarantee a constant etching rate, the SiO₂ standard is etched before and after the profiling of every implanted sample and the data obtained is used to correct the profile time axis.

2.3. Quantification
The arsenic content (in at. %) is obtained from the Auger data by the formula:

\[ c_{As} = 100 \left( \frac{I_{Si0} - I_S}{I_{Si0}} \right) \]  

(1)

3. Results
3.1. Derivation of equation (1) for AES quantification
The experimentally determined relation of proportionality between the content, \( c_A \), of the element A in the sample and the intensity of its Auger peak, \( I_A \), is a main postulate of Auger quantification. However the relation, obtained as quotient, after applying the aforesaid for two samples,

\[ \frac{c_A}{c_A'} = \frac{I_A}{I_A'} \]  

(2)

is true only if all the conditions concerning the Auger process are equal for the two samples. Let us assume this is correct and the second sample is a standard of the element A (for a “pure sample” \( c_0 \) is 1):

\[ c_A = \frac{I_A}{I_A}. \]  

(3)

Let the sample examined be binary. The concentration of the second element, \( c_B \), is obviously \((1 - c_A)\). This relation and (3) yield (1), in atomic parts. The main requirement for the conclusion’s correctness is equality of the Auger conditions for the two samples.

3.2. Calibration of the x-axis
We equalized the time axes for all profiles, but the absolute profiling depth is not known. The influence of the high implant doses over the etching rate is not entirely clarified. A priori, different – even opposite – tendencies are possible (for example, the included arsenic, being a heavier element, should slow the etching down, while silicon amorphization should speed it up [6]). Therefore, we resort to the available RBS profiles. Taking the middle of the dose range profile (1.2×10¹⁷ atoms/cm²) we fit it over the corresponding RBS. For that purpose, first we approximate it with splines. After that, we change the x-axis scale of this analytic curve, while monitoring (by the least squares method) its fit over the RBS profile. The optimal fitting gives the wanted depth calibration of the AES profile. The same stretching of the x-axis is done for the remaining AES profiles. The superposition of the corresponding AES and RBS profile is shown in figure 1. The fitting is acceptable for all couples, i.e. it does not depend strongly on the dose. The same holds for the ion etching rate of the layers. The profiles for all doses studied are presented together on figure 2.

4. Discussion
4.1. Reasons proving the correctness of using formula (1)
It is known that Auger quantification requires the use of relative intensities (This compensates the intensity difference between the samples, which do not have concentrational origin.). Although only one peak (Si) is monitored (at each etching step) during our analysis, the profiling should be considered as a unified experiment, for which all the Auger conditions are equal (the sample, the
apparatus and the analytical mode remain strictly the same). Actually, the quantification is performed using relative intensity (the normalization is carried out by the intensity $I_{Si0}$, which can be considered as obtained in the same experiment). From the analytical conditions, only two points of the profile can be attributed to the matrix effects or changes caused by the etching; however, accounting for such differences is not an important problem of the Auger practice.

4.2. About the matrix effects and their corrections

In the quantification variant suggested by (1), the matrix effects are not taken into account. This is preferable to introducing corrections using unclear criteria (for the ion-implanted materials). Here we suggest an evaluation of their order of magnitude. The silicon intensity in (1) is corrected relative to the intensity of the silicon standard. The correction of the backscattering effect (for the plateau in the beginning of the profile), performed according to [7], is shown in table 1. The correction is in the right direction, but exceeds the RBS data. A combined rough correction for a change in the atomic density, $N$, and the attenuation depth can be expressed as $(N/N_0)^{1/2}$ [8]. The atomic density of As is higher by 10 % than that of crystalline Si. On the contrary, the silicon atomic density should decrease in high-dose Im; one tentative value is that of amorphous Si (5% less than the value of the crystalline Si [9]). In the presence of voids, however, there exist data showing a decrease by 18 % (or even 25 %). For the calculations, we use the value of 10 % and assume that silicon is fully amorphized and As is built in as a substitute atom. The result from the total correction is also presented in table 1. Despite the close match with the RBS results, we should emphasize that real corrections necessitate a crystallographic characterization of the layer.

Figure 1. Depth scale calibration, showing the fitting between AES and RBS data.

Figure 2. As concentration profiles for all doses.
4.3. Detection limit of the implanted element and accuracy in its determination

The minimal implant concentration, \( c_{\text{min}} \), if the signal exceeds the noise by a factor of 2.5 (with double-amplitude \( N \)), is:

\[
c_{\text{min}} \, (\%) = 250(I_{T0}/N)^{-1} = 250(S/N)c^{-1}(S_{C0}/S_{T0}).
\]  

(4)

### Table 1. Matrix correcting for the averaged maximum of the As distribution.

| Dose, cm\(^{-2}\) | Without correcting | With a backscattering correction | With a total correction | RBS data |
|---------------------|---------------------|---------------------------------|-------------------------|----------|
| \(2 \times 10^{17}\) | 28.0                | 33.6                            | 30.0                    | 31.3     |
| \(1.6 \times 10^{17}\) | 31.4                | 37.3                            | 33.9                    | –        |
| \(1.2 \times 10^{17}\) | 29.9                | 36.6                            | 32.2                    | 31.5     |
| \(8 \times 10^{16}\)  | 27.9                | 33.5                            | 29.9                    | –        |
| \(4 \times 10^{16}\)  | 18.9                | 23.6                            | 19.4                    | 19.4     |
| \(2 \times 10^{16}\)  | 11.3                | 14.5                            | 9.8                     | 10.9     |

There, the signal from a pure target element, \( I_{T0} \), determines the ratio “signal-noise” for the apparatus, \( I_{T0}/N \). If the latter is known as an apparatus parameter, \( (S/N)c \) (for a standard element C), the second record can be used; \( S_{C0} \) and \( S_{T0} \) are the standard and the target sensitivities. The implant detection limit is determined by the element of the target (improved by a factor of \( S_{T0}/S_{C0} \) compared to the ESFM calculated).

The relative error of the implant concentration (from the intensities), \( \Delta c/c \), is determined from (1). The expression, written as an explicit function of \( c \), decreases with the decrease of \( c \):

\[
\Delta c/c = (\Delta I_{S}/I_{S0})(2 - c)/(1 - c).
\]  

(5)

For example, for a dose \(2 \times 10^{17}\) atoms/cm\(^2\), its value in the beginning of the profile \((c \approx 0.3)\) is around \(2.4N/I_{S0}\) and decreases to \(2N/I_{S0}\) in the profile “tail”. This is opposite to the error behavior in the ESFM quantification, in which case the error increases when \( c \) decreases, reaching 40 % at the detection limit (20 times as high as in our method). This feature leads to “smoother” profile „tails“.

### Conclusions

I. The profile distributions of As implanted in silicon at energy 40 keV in the dose range \(2 \times 10^{16}-2 \times 10^{17}\) atoms/cm\(^2\) are obtained using AES.

IIa. In what concerns quantification, the silicon peak is only monitored during the profiling and the arsenic content is calculated by the decrease in Si intensity relative to the Si elemental standard.

IIb. The profile depth scale calibration is carried out using additional (RBS) data.

III. For ion implantation with dose over \(2 \times 10^{16}\) atoms/cm\(^2\) in the system “As in Si”, the sputtering rate by Ar (3 keV) ions does not depend strongly on the dose.

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