Journal of ELECTRICAL ENGINEERING, VOL. 56, NO. 9-10, 2005, 270–273

COMMUNICATIONS

AN AUTOMATIC MEASUREMENT SYSTEM WITH HALL PROFILING AND A FOUR POINT PROBE FOR CHARACTERIZATION OF SEMICONDUCTORS

Rudolf Kinder * — **Ladislav Hudec** ** — **Marian Blažíček** **

This article deals with the design, construction and a control program of an automatic measuring system (AMS). The AMS allows to obtain the carrier concentration profile $n(x)$ and mobility profile $\mu(x)$ from the sheet resistance and sheet Hall mobility by van der Pauw measurements, while gradually removing layers of the semiconductor by anodization/etch procedures. AMS can also be used for sheet resistance measurements by the four-point-probe method and for calculating the specific resistance. Further, the software of the AMS contains graphical subroutines for conversion of resistivity to concentration and mobility. The experimental $n(x)$ profile has been compared with that calculated by program SUPREM.

K e y w o r d s: Hall system, van der Pauw, Si, four point probe, SUPREM

1 INTRODUCTION

The research and development of semiconductor devices require reliable determination of the doping profiles in device layer structures. Several methods are used for doping profile measurement. The various measurement techniques can be divided into two groups: analytical and electrical. The first group (doping profile measurement) includes Secondary Ion Mass Spectrometry, Auger Electron Spectroscopy, Rutherford Back Scattering and others, while the second comprises carrier concentration $n(x)$ measurements, capacitance-voltage, four-point-probe, spreading resistance, electrochemical capacitance-voltage, differential Hall and others. The most widely used method for electrical profile measurement is the spreading resistance method. A disadvantage of the method is that for calculating the $n(x)$ profile it needs corrections assuming variations of the mobility with concentration. For mobility measurements the differential Hall technique is employed. The professional equipment is from fy Bio-Rad, HL5900 Striping Hall System. This system can measure $n(x)$ and mobility $\mu(x)$ profiles [1–3].

This paper describes an automatic striping Hall system with the four-point- probe method. Optimum measurement conditions for determination of $n(x)$ and mobility $\mu(x)$ profiles by the four-point-probe method are established. The $n(x)$ profile is compared with that calculated by program SUPREM.

2 THEORY

From the measured sheet conductivity and Hall coefficient at various depths, the carrier concentration and mobility may be found from the differential equations

$$n(x) = \frac{r}{q}\Big(\frac{\mathrm{d}\sigma_s}{\mathrm{d}x}\Big)^2 \Big(\frac{\mathrm{d}\big(R_s\sigma_s^2\big)}{\mathrm{d}x}\Big)^{-1}, \qquad (1)$$

$$\mu(x) = \frac{\mathrm{d}\big(R_s\,\sigma_s^2\big)}{\mathrm{d}x}\Big(\frac{\mathrm{d}\sigma_s}{\mathrm{d}x}\Big)^{-1}. \qquad (2)$$

Here, r is the Hall scattering factor (μ_H/μ), μ_H is the Hall mobility, R_s is the sheet Hall coefficient, σ_s is the sheet conductance.

The values for local carrier concentration and mobility are found from the measurement made at discrete depths using an approximation to the differential equation (1) and (2). Then from the data for the i-th and $(i+1)$-th points [1]

$$n\Big(x + \frac{\Delta X_i}{2}\Big) = \frac{1}{q\Delta x_I}\frac{\big(\Delta(\sigma_s)_i\big)^2}{\Delta(R_s\sigma_s^2)_i} \qquad (3)$$

$$\mu\Big(\mu + \frac{\Delta x_I}{2}\Big) = \frac{\Delta\big(R_s\,\sigma_s^2\big)_i}{\Delta\big(\sigma_s\big)_i} \qquad (4)$$

where $\Delta x_i = x_{(i-1)} - x_i$, $\Delta\big(\sigma_s\big)_i = \sigma_{s(i+1)-si}$. The Hall scattering factor has been taken as being equal to unity.

As with any measurement, it is important to be aware of the errors inherent to the profiling method so that no possible misinterpretation of the results can occur. At least some errors need to be considered [1]. The first error arises from the fact that the value chosen for the Hall scattering factor, r, is not always unity. The value for the Hall scattering factor varies with the carrier concentration, the precise relationship depending on the semiconductor material being measured. Second, the errors in the sheet Hall coefficient and sheet conductivity give rise to a scatter in the profiles. The depth resolution is directly limited by the accuracy to which the sheet values can be measured. Third, there is a depletion of carriers at the surface of the sample. This effect will clearly depend on the material under consideration and the doping level. This is generally not a problem for Si but can be a problem for GaAs where surface charge induced space charge regions are very common. Other errors are described in detail in [1, 3].

In the technique of the four-point-probes placed along a straight line, the resistivity ρ of a homogeneously doped

* Faculty of Electrical Engineering and Information Technology, Department of Microelectronics e-mail: rudolf.kinder@stuba.sk,
** Faculty of Informatics and Information Technologies, Institute of Computer Systems and Networks, e-mail: lhudec@fiit.stuba.sk, Slovak Technical University, Ilkovičova 3, 812 19 Bratislava, Slovakia,

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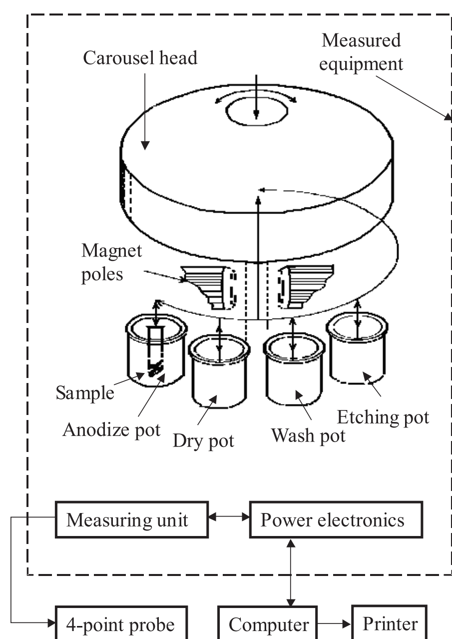

Fig. 1. Automatic measuring system.

semiconductor can be calculated from

$$\rho = R_s H = 4.53 \frac{V}{I} H k \,. \tag{5}$$

Here, H is the thickness of the semiconductor sample, I is the current flowing through the outer tips of the probe, V is the voltage drop measured on the inner tips, k is the correction factor. The accuracy of calculating the parameters by the four-point-probe is described in [4].

To reveal the $n(x)$ profile it is necessary to calculate the parameters of anodic oxidation (AO), the thickness Δh (Δx) of Si to be removed, the number of removed layers Δh_n needed to find the whole profile, the voltage needed to create an oxide layer with thickness Δh which is subsequently removed by chemical etching. The thickness Δh of Si to be removed follows from the assumption that at least ten values are needed to define the $n(x)$ profile. Parameter Δh can be calculated from the depth x_j or from the following empirical relation [4]

$$\Delta h = C \sqrt{\Delta R_{PSi}^2 + 2Dt} \,. \tag{6}$$

Here $C = 0.5$ is a constant factor which was found experimentally, ΔR_{PSi} is the mean quadratic deviation of the projected range of the implanted particles in Si, D is the diffusion coefficient, and t is the time of annealing. The voltage V_k needed to create an oxide layer by anodic oxidation with a thickness of Δh which is subsequently removed by chemical etching is given by the following relation

$$V_k = V_p + \Delta V \tag{7}$$

where V_p is the voltage determined experimentally (17 V). Its value depends on the resistance of the electrolyte between the anode and cathode. ΔV is the voltage drop across the anodic oxide. For thickness Δh, the magnitude of ΔV can be calculated. For p- or n-type semiconductors the following relations have been derived experimentally.

For p-type semiconductor (boron-implanted silicon)

$$\Delta h = 12.8662 \exp(6.8022 \times 10^{-3} \Delta V) \tag{8}$$

and for n-type (phosphorus-implanted silicon):

$$\Delta h = 11.5162 \exp(7.91435 \times 10^{-3} \Delta V) \,. \tag{9}$$

The values of the concentration profile $n(x)$ from the measured values $\rho(x)$ can be obtained from conversion graphics (Irvin curves, for n- and p-type). The curves express the mutual dependence between resistivity ρ, mobility μ and concentration n, according to formula [4, 5]

$$n(x) = \frac{1}{\rho(x)\, \mu(x)\, q} \,. \tag{10}$$

For automatic calculation of ρ or n, the value of μ is used given by empirical formula

$$\mu = \mu_{\min} + \frac{\mu_{\max} - \mu_{\min}}{\big(1 + (n/n_{ref})\big)^{\alpha}} \,. \tag{11}$$

Here $\mu_{\min}$ and $\mu_{\max}$ are the values of minimal and maximal mobility, n_{ref} is the reference concentration and α is an exponential factor [4].

3 DESIGN OF EQUIPMENT AND MEASUREMENT ROUTINE

The measurements and calculations were carried out on an automated set-up designed and constructed at the Department of Microelectronics, Slovak Technical University in Bratislava [6]. The automatic measuring system is seen in Fig. 1. The automatic measuring system consists of a PC, special measuring card, mechanical part, electronics of the measuring unit, power electronics, electromagnet and 4-point probe equipment. The menu driven software AMS insures communication with the measuring equipment, commands its single parts, measurement of the required parameters and communication with the operating personnel. The operating personnel, by means of the driven software, enters the data needed for the measurement process and at the same time the results of measurement form a listing on the display or a file. The AMS provides also cooperation with the external program SUPREM. The $n(x)$ profile obtained experimentally can be compared with theoretical ones based on Gauss and Pearson distribution (SUPREM) using parameters gained from LSS theory. As development tools, the operating systems MS Windows and Borland C Builder 6 were used. Hardware requirements on the development tools are as follows: PII 400, 128,MB RAM, 600 MB HDD.

In the measurement routine a standard series of Hall voltage and resistivity measurements a low specimen current in the range 10 nA to 100 mA (depending on the resistivity of the sample) is used and a magnetic field of 0.36 T at current 1.8 A. Anodic oxidation is performed at a constant current in the range from 0 to 10 mA and voltage from 0 to 300 V. The sample (mainly n-type material) was illuminated by 100 W halogen lamps. Details about the AMS are described in [6]. The AMS contains a program for testing single parts of equipment (step motors, block current source, switching field *etc*).

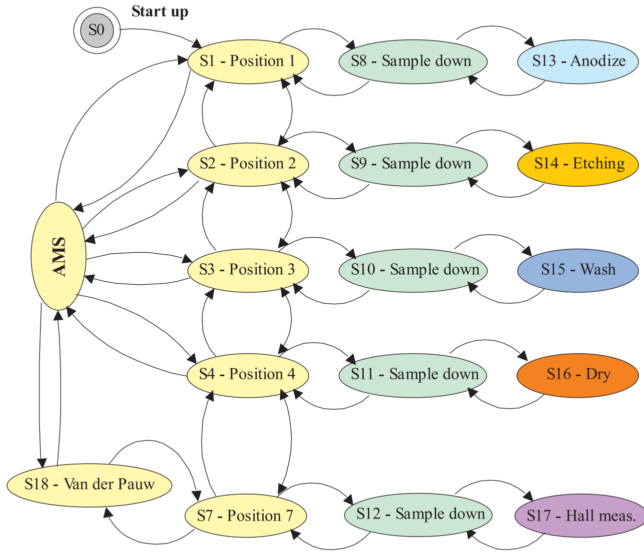

Fig. 2. Diagram of the AMS.

The sequence of single operations of one cycle is as follows: anodic oxidation - washing - drying - etching - washing - drying - measurement out of the magnetic field (van der Pauw) – measurement in the magnetic field (Hall). The optimum condition for anodic oxidation and measurement is agitation, circulation of the electrolyte for AO, deionized water for washing and 8 % HF electrolyte for etching from reservoirs by pumps.

A simplified diagram of the AMS used to obtain electrical profiles is shown in Fig. 2.

In picture Fig. 2 one can see single positions which can be assumed by AMS. S0 is the initial condition, S1 to S7 are positions with sample up, S8 to S12 are positions with sample down, S13 is position for anodizing, S14 for etching, S15 for drying, S17 for Hall measurement, and S18 for van der Pauw measurement. The possible control of the sample holder by AMS is marked by arrows.

Preparation of the sample for measurement is as follows: Before measurement, the sample is etched in 8 % HF, rinsed in deionized water and dried in N_2. On the implanted Si sample with dimensions $2 \times 2\,\mathrm{cm}^2$ a pattern in the form of a quatrefoil was created by photolithography. Around the quatrefoil, the implanted region was isolated by mesa etching into a depth of 5 um and rinsed in deionized water and dried in N_2. Then the sample was mounted into the sample holder. In the corners of the quatrefoil, ohmic contacts were created with an InGa alloy and connected with wire contacts of the sample holder. In the middle of the samples, a circular area with diameter 1 cm was defined for anodic oxidation by a rubber sealing. The sample with contacts (apart from circular areas) and part of sample holder was coated with black wax.

Now, the sample holder is connected into the socket set in the carousel head, Fig. 1. Measuring the I-V characteristics by AMS can be affected by the quality of ohmic contacts between the sample and sample holder. If the I-V curves are linear, then the ohmic contacts are very good.

For repeated and well controlled thin layers from a doped semiconductor anodic oxidation at a constant current was used [4, 6]. The anodization/etch procedures were as follows: 10 sec washing in deionized (DI) water, 10 sec drying in the flow of N_2, 60 sec etching of anodic SiO_2 in 8 % HF, 60 sec rinsing in DI water, 120 sec drying in N_2. The electrolyte for AO consisted of ethylenglycol, 0.4 % of KNO_3, 10 % H_2O, 1g/$Al(NO_3)_3.9H_2O$. The anodic oxide was grown at a constant current density $I = 5\,\mathrm{mA/cm}^2$.

To acquire $n(x)$ and $\mu(x)$ profiles it is necessary to measure the sheet conductance σ_S (or $1/R_S$) and sheet Hall constant R_{HS} in successive steps from the surface to the depth x of semiconductor by anodization/etch procedures. From the technological parameters of the measured implanted sample and Eqs. (6), (7) and (9) the thickness $\Delta h = 20$ nm and $V_k = 98$ V were calculated. The number of removed layers is $\Delta h_n = 8$. The value was determined from the calculated depth of the pn junction and knowledge that the sensitivity for measuring the sheet Hall constant R_{HS} is minimal at the concentration $10^{17}\,\mathrm{cm}^3$. The sheet resistivity and sheet Hall coefficient are calculated from the measured voltages using equations (3) and (4). The longest operation is the formation of the oxide layer by anodic oxidation, it takes a few tens of minutes.

The switching field of AMS was used for linear 4-point probe (200 g and a reduced tip pressure loading of 20 g) method measurement. For calculating the values of R_s and ρ, Eq. (5) can be used. The conversion of ρ to n for p- and n-type Si according to Eqs. (10) and (11) can be performed by a conversion code. For calculating R_s, ρ and n it is needed to know these parameters: type of 4-point probe, type of semiconductor, thickness of the sample or layer, value of the correction factor, current (max. 100 mA) and number of measurements. Then from the number of measurement the average values of R_s, ρ and n are calculated (Eqs. (5), (10), (11)).

4 EXPERIMENTAL RESULTS

To verify the results achieved by applying the automatic measuring system, wafers with an n-type implanted layer and a p-type, (100)-oriented substrate with concentration $N_B = 1.5 \times 10^{15}\mathrm{cm}^3$ were used. The wafer was phosphorus implanted through a SiO_2 layer (thickness 75 nm) at energy 60 keV and dose $10^{15}\mathrm{cm}^2$. The implanted wafer was annealed at 900 °C for 10 minutes in N_2 ambient. The samples for measurement of the sheet resistance by the 4-point probe were, first, with a p-type, (100)-oriented substrate with surface concentration $N_B = 7 \times 10^{19}\mathrm{cm}^3$ and sample thickness about 630 μm and, second, a homogeneous sample with p-type, (100)-oriented substrate with concentration $N_B = 1.5 \times 10^{15}\mathrm{cm}^3$ with sample thickness about 300 μm. For both samples, the value of k was 0.9818 (Eq. 5).

The measured and calculated profiles $n(x)$, $\mu(x)$ and $N_T(x)$ are in Fig. 3.

The $n(x)$ profile was compared with $N_T(x)$ profile. A Fourier series was used to approximate the experimental profile. In this case, $n(x)$ is in good agreement with the

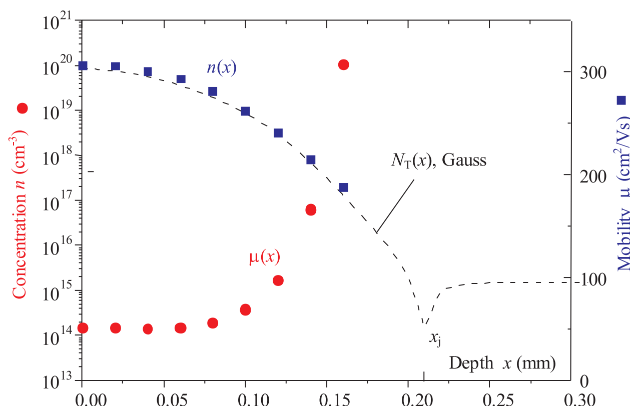

Fig. 3. Profiles $n(x)$ and $\mu(x)$. The depth x_j of the pn junction of phosphorus implanted into silicon. $N_T(x)$ is the theoretical profile.

theoretical profiles $N_T(x)$ shown in Fig. 3. At the calculation of $n(x)$ and $\mu(x)$ profiles for implanted phosphorus in Si the Hall scattering factor was 1. The accuracy of determining the $n(x)$ profile depends on the gradient of the implanted layer. The accuracy of determining of the depth of $n(x)$ profile is $\pm\Delta h/2$ and the profile is evaluated with an accuracy of 10 %. The depth of the $n(x)$ profile can be controlled by Talystep measuring the depth of the crater created by anodization/etch procedures. In the case of difference the depth of the $n(x)$ profile can be corrected by the code in AMS.

Further, with the help of AMS and 4-point-probe method we have measured and calculated, according to Eq. (5), the values of ρ and n. The measurements of R_s were made using a linear 4PP at a reduced tip pressure loading of 20 g. For the first sample we measured a value $\rho = 0.017\,\Omega$cm and calculated $n = 6.5\times10^{19}$cm^{-3}. For the second sample, $\rho = 11.4\,\Omega$cm and $n = 1.7\times10^{15}$cm^{-3}. The above-mentioned results ρ and n satisfy the technological parameters of samples.

The accuracy of conversion of ρ to n and counter calculated by Eqs. (10) and (11) is 0.01 %. The calculated results are identical with the results obtained from Irvin curves.

5 CONCLUSION

AMS provides carrier concentration and mobility profiles of semiconductor materials as a function of depth. Further, the classical Hall measurement system has been completed by a four-point-probe method which employs a relay network for measuring the sheet resistance and then calculates the resistivity of semiconductor layers. The software of the equipment contains graphics for converting the resistivity into concentration and mobility. The experimental results have been compared with theoretical ones based on Gauss $N_T(x)$ distributions. The AMS can be used for profiling other semiconductors material. For other materials it is necessary to insert appropriate parameters and for anodization/etch procedures another electrolyte. Potential problems inherent to the AMS have been described and minimized as far as possible.

Acknowledgement

This work was supported by the grant of the Slovak Grant Agency Vega No. 1/0154/03.

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Received 22 March 2005

Rudolf Kinder (doc, Ing, CSc), was born in Pezinok in 1940. In the sixties, he worked in electrotechnical industry in Bratislava. Then he worked at VURUP (Research Center for Petrol Chemistry) in the field of automation of chemical services. He graduated in technical cybernetics from the Faculty of Electrical Engineering, Slovak Technical University, Bratislava. He received a CSc (PhD) degree in electronics and vacuum technology in 1984. He has worked as a senior scientist since 1989. Since 1996 he has been Associate Professor in microelectronics. His research activities are oriented towards semiconductor technology, particularly to analysis and simulation.

Ladislav Hudec (doc, Ing, CSc), currently Associate Professor of Computer Science and Engineering and Deputy Director of the Institute of Computer Systems and Networks, Faculty of Informatics and Information Technology, Slovak Technical University. In 1974 he received the Ing diploma with summa cum laude in electronics from Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University, Prague, in 1985 he was received CSc degree (PhD) in Computer Machinery from the Faculty of Electrical Engineering, Slovak Technical University, Bratislava, in 1989 he was appointed Associate Professor at the Slovak Technical University. He is author or co-author over 30 scientific papers and over 50 technical papers in the field of fault tolerant computing, embedded systems, parallel computing and computer security. Dr. Hudec was a founding member of the Slovak Centre of the IEE, he is member and President of the Slovak Association for Information Security (SASIB), member of the Information System Audit and Control Association (ISACA), holder of the CISA license. Since 1993 he serves as National Coordinator at the European Cooperation in Science and Technology (COST).

Marian Blažíček (Ing), was born in Trenčín in 1978. He graduated in computer systems and networks from the Faculty of Informatics and Information Technologies, Slovak Technical University, Bratislava. He worked as programmer-analyst for financial institutions. Since 2000 he has worked as network administrator.