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THE DISTRIBUTION OF PHOSPHORUS AND GOLD IN NEUTRON IRRADIATED SILICON

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Résumé. — L'homogénéité de la concentration de phosphore créé par transmutation nucléaire ainsi que la distribution d'autres éléments tels que l'or ont été étudiées dans le but d'examiner l'influence à l'échelle microscopique du flux de neutrons dans du silicium irradié aux neutrons. Deux techniques ont été utilisées : i) sectionnement électrochimique et ii) sectionnement mécanique. Les deux méthodes montrent que la concentration de phosphore est uniforme. L'or, au contraire, est concentré à la surface.

Abstract. — The uniformity of phosphorus concentration produced by nuclear transmutation and the distribution of other elements such as gold have been studied to observe the possible influence of the neutron flux at a microscopic level in neutron irradiated silicon. Two techniques were used : i) electrochemical sectioning and ii) mechanical sectioning. Both methods show that the phosphorus concentration is uniform throughout. Gold, on the other hand, is concentrated at the surface.

1. Introduction. — The doping of silicon by phosphorus by nuclear transmutation using the following reaction :

is utilized increasingly [1-5]. The two chief advantages are the ultimate control on the desired donor doping level and the uniformity of doping [1, 3]. In the present paper, the uniformity of distribution of the phosphorus atoms created by nuclear transmutation has been studied on a microscopic scale (50 nm-1 000 nm) not investigated before. Further, the distribution of the other elements present in silicon has been studied after irradiation, on the same microscopic scale. In particular, gold added as a lifetime killer in order to decrease the switching time of the device [6] has been carefully investigated.

2. Experimental. — The irradiation of highly pure silicon by neutrons has two roles both of which are used in this paper. The first one is doping by nuclear transmutation. The second one is neutron activation

of silicon and background impurities. The measurement of beta-activity of Si-31 gives the number of phosphorus atoms, while the gamma-activity of the activated impurities identifies them and gives their concentration.

2.1 SAMPLES USED. — We have used *hyper pure silicon* crystals supplied by *Wacker*. They are float-zone, dislocation free, grown in the 1-0-0 orientation. All the samples considered here are p-type, boron doped, the concentration of boron ranging from 10^{13} upto $10^{16}/\text{cm}^3$. Our activation analysis confirms the high purity if one excepts the gold as will be discussed later.

2.2 IRRADIATION. — The silicon wafers are normally irradiated in a thermal neutron flux $J = 3.5 \times 10^{13}$ neutrons/cm²/s for the time $t_1 = 24$ hours.

From the specific activity due to the emitted beta-rays which corresponds to the half-life time of Si-31 (2.62 h) the neutron cross-section is found to be $\sigma = 0.12$ barn from the equation [7] :

$$A = 36 \times \frac{\sigma \times J}{M} \times \frac{1 - \exp(-\lambda t_1)}{\exp(\lambda t_2)} \times RC \times AI \times PM \quad (2)$$

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where A is the specific activity in counts per minute and per gram of silicon ($M = 28.1$), at the time t_2 after removal from the pile. The efficiency of our low background (1 cpm) Geiger counter is $RC = 0.30$ for this electron energy, the isotopic abundance of Si-30 is $AI = 0.030$, the fraction of mode of decay is $PM = 1$ and λ is the radioactive constant of Si-31.

For irradiation times of one day or more, the activity of Si-31 attains its saturation value. The concentration C of P-31 atoms expressed as the number of atoms per cm^3 of silicon is then derived from the following equation :

$$C = A \times \exp(\lambda t_2) \times \rho \times t_1 \quad (3)$$

where ρ is the specific mass of silicon.

2.3 ELECTROCHEMICAL SECTIONING. — Samples of dimensions $12 \text{ mm} \times 12 \text{ mm}$ are cut from the wafers and mounted in an electrochemical cell. Sectioning of the crystals is done by successively oxidizing and removing layers of silicon parallel to the flat surface of the wafer using the anodic oxidation technique. Thickness of each section is determined by weighing the crystal with a microbalance immediately before and after oxidation. The average thickness can be varied up to 100 nm with the present apparatus. The beta rays emitted by Si-31 are counted for each section removed with an anti-coincidence beta counter. Equation (2) is again used to derive the concentration of the phosphorus atoms (per cm^3 of Si) from the specific activity.

2.4 MECHANICAL SECTIONING. — Strips of width 5 mm are cut from the wafers and then sectioned with a microtome into $3 \mu\text{m}$ slices perpendicular to the original flat surface of the wafer according to the technique developed earlier [7].

The radioactivity is measured in each slice using a beta counter for determining the concentration of phosphorus as well as a gamma counter for determining the concentration of the elements activated in the process, especially gold.

3. Results. — **3.1 DISTRIBUTION OF PHOSPHORUS.** — The concentration of the phosphorus atoms calculated from the activity of Si-31 in an anodic oxidation experiment is shown on figure 1. We accumulate the countings, for typically 10 minutes, to get a total number of 2 000 counts per slice. The background being only 1 cpm, the error of counting is the statistical error only, namely $1/\sqrt{2\,000} \approx 0.02$. The thickness removed in each anodic oxidation as measured from the difference in weight of the sample with a microbalance is equal to $(60 \pm 10) \text{ nm}$. The error on the thickness is thus 16 %, this gives a total error of 18 %. The standard mean deviation (Fig. 1) is slightly less than 10^{14} atoms/ cm^3 , so that the mean fluctuation of the data points is equal to

FIG. 1. — Concentration of phosphorus in successive layers of silicon sectioned by anodic oxidation parallel to the main surface.

15 %. We can then conclude the uniformity of phosphorus distribution within the limits of error. The point to be emphasized is that the uniformity already demonstrated at the macroscopic scale is found in the present work at the microscopic scale of 60 nm .

However, as the reproducibility of the thickness of the anodic sections is not excellent and as the time for anodic oxidation is too long compared with the limited time available with Si-31, microtome sectioning is found to provide a better way for investigating the uniformity of P in Si. As a matter of fact, it is possible with a single irradiation to measure the concentration of P in 400 sections of preset thickness ranging from 1 to $10 \mu\text{m}$ with a very good reproducibility of thickness. The results plotted on figure 2 again show the uniformity of phosphorus distribution within the experimental errors of measurement which is much less for this mechanical sectioning (5 %).

In all the samples which have been studied, about 50 % of the data points lie within $\pm 5 \%$ of the average concentration.

In preliminary experiments some sections exhibited more beta activity. This was resolved as due to impurities present at the surface of the crystal which were activated during irradiation. All the impurities were easily removed by washing the crystal in 5 % HF + 5 % HNO_3 solution except for gold which is present in the whole bulk.

FIG. 2. — Concentration of phosphorus in successive layers of silicon sectioned by microtoming perpendicular to the main surface. The activity of Si-31 has been separated from Au-198 activity and corrected for the radioactive decay.

3.2 DISTRIBUTION OF GOLD. — About 4 days after removal from the pile, when Si-31 has completely decayed (Fig. 3), p-type (boron doped) silicon samples were sectioned using anodic oxidation in order to determine the concentration of gold. The gamma

FIG. 3. — Decay curve of a radioactive section as a function of time.

energy spectrum taken at that time (Fig. 4) of a removed section shows the 0.4 MeV peak of Au-198. The gamma energy spectrometer was adjusted on this peak, to determine the concentration of gold in each section of 90 nm thick. Figure 5 shows the concentration of gold atoms per cm³ as a function

FIG. 4. — Gamma energy spectrum of a section showing all the peaks of Au-198.

FIG. 5. — Content of gold atoms in successive layers of silicon obtained by anodic oxidation (thickness of each section : 90 nm).

of depth ; it drastically decreases from 10¹⁷ cm⁻³ in the first section down to 10¹⁵ cm⁻³ at 1 μm to finally drop to the value of 3 × 10¹³ cm⁻³ in the bulk of the crystal. This profile is typical of gold [8]. This distribution may already be present during the preparation of the wafers, or due to an outward diffusion during the irradiation or the subsequent annealing (one hour at 500 °C) necessary for obtaining conductive samples for the anodic oxidation.

Lastly, it was noticed that two sections were more active than their neighbours. It was often found that one section out of about ten contains an anomalously high gold content. The origin of this is not as yet clear.

4. Conclusions. — a) The phosphorus produced by nuclear transmutation is distributed uniformly in the crystal. Such a conclusion had already been drawn from macroscopic electrical measurements [1]. This conclusion is confirmed on a microscopic scale down to 50 nm. In particular, no aggregation of the created atoms has been observed on this scale.

b) In those samples in which gold atoms have deliberately been added a rapid decrease of three orders of magnitude is found between the concentration at the very surface and in the bulk. This decrease occurs over a few microns.

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