UBPub-EPPG-PHYS-22

BUCHAREST UNIVERSITY

EXPERIMENTAL PARTICLE PHYSICS GROUP

FERMILAB
APR 24 1995
LIBRARY

Fermilab Library
0 1140 0041481 2

UBPub.EPPG/Phys.22
February 1995

EFFECTS ON THE RESISTIVITY AND HALL COEFFICIENT OF NEUTRON IRRADIATED SILICON

U.Biggeri, E.Borchi, and M.Bruzzi
University of Florence & INFN Florence, Italy

S.Lazanu+
Institute for Atomic Physics, Bucharest, Romania

presented by Sorina Lazanu at the 4-th International Conference "Advanced Technology and Particle Physics", Como, Italy, October 1994

Abstract

High temperatura annealing treatment has been carried out on fast neutron irradiated silicon samples with temperatures up to 300 C. Fluences of irradiation up to 10^{14} n/cm^2 were used. Before annealing, samples irradiated with fluences higher than 10^{13} n/cm^2 suffered the type conductivity inversion from n-type to p-type. The changes in resistivity and Hall coefficient during high annealing step have been measured by Hall effect analysis. Results indicate the possible creation of acceptors for low temperature annealing up to 150 C, and the Phosphorus release by E centres at annealing temperatures among 150 and 200 C. Heating samples up to 300 C allows recovering the resistivity to its value before inversion, with the peculiarity that bulk inverted to p-type after irradiation does not come back aftes annealing.

+now at JINR-Dubna

Annealing Effects on Resistivity and Hall Coefficient of Neutron Irradiated Silicon

U. Biggeri, E. Borchi, and M. Bruzzi

Dipartimento di Energetica, Universita' di Firenze, Via S.Marta 50139 Firenze, Italy

S.Lazanu

Institute for Atomic Physics, POBox MG-6, Bucharest, Romania now at JINR-Dubna

High Temperature Annealing (HTA) treatment has been carried out on fast-neutron irradiated silicon samples with temperatures up to 300C. Fluences of irradiation up to $1x10^{14}$n/cm^2 were used. Before annealing, samples irradiated with fluences higher than $1x10^{13}$n/cm^2 suffered the type conductivity inversion from n-type to p-type. The changes in the resisitivity and Hall coefficient during each annealing step have been measured by Hall effect analysis. Results indicate the possible creation of acceptors for low temperature annealing up to 150C and the phosphorous release by E centres at annealing temperatures among 150C and 200C. Heating samples up to 300C allows the recovering of the sample resistivity to its value before irradiation, with the peculiarity that bulks inverted to p-type after irradiation does not come back to n-type after annealing.

1. Introduction

The damage induced in silicon detectors by incident radiation may be favourably investigated by means of the Hall effect analysis [1]. This technique allows the direct evaluation of the concentration of free carriers inside the bulk of the irradiated device. Previous Hall effect studies [2,3,4] have shown a general increase in the resistivity with increasing the fluence of irradiation up to a value f_o (of the order of 10^{13} n/cm^2 for 1MeV neutron irradiations). For further irradiations the resistivity was seen to decrease. This effect is accompained by a change in the Hall coefficient value R_h. Increasing the fluence, the absolute value of the negative Hall coefficient R_h increases and, for fluences higher than f_o , it changes sign to positive. This effect has been explained considering a change of the conduction type of the irradiated bulk, initially n-type, to p-type [2,3,4]. Increasing the irradiation fluence, a decrease of the electron concentration seems to occur which is correlated to an increase of the hole concentration, to satisfy the mass action law. The maximum of the resistivity corresponds to the nearly intrinsic material. High Temperature Annealing (HTA) has been carried out in past by several experimental groups [5,6,7,8] to study the correspondence among the macroscopic damage of the irradiated device and the radiation-induced lattice disorder. In particular, efforts have been spent to observe the electric properties recovery by leakage current and capacitance versus reverse voltage characteristics after HTA processes [5,6]. The release of deep levels after HTA has also been studied [5,8,9] by means of DLTS [10] and TSC techniques [11]. Conversely, almost no informations have been collected up to now on the possible recovery of both the resistivity and the Hall coefficient with HTA by Hall effect analysis on high resistivity material. In this paper, the changes in resistivity and Hall coefficient occurred in neutron irradiated silicon samples after an high temperature annealing process are presented. Results are discussed in terms of a possible recovery after annealing of the main radiation-induced lattice defects.

2. Experimental Setup

In the present study square-shaped samples, 10x10mm^2 area and 1mm thickness, of n-type silicon of initial resistivity 4-6KΩcm were used. The starting material was phosphorous doped silicon, with low initial compensation. The ohmic contacts in the corners were realised by aluminium vacuum deposition on the skratched silicon surface. The samples have been produced at the Brookhaven National Laboratory, USA.

Neutron irradiation was performed at the University of Lowell, USA, with fast neutrons of average energy 1 MeV, obtained from 4 MeV accelerated protons by means of the reaction 7Li(p, n) [12]. Used neutron fluences are reported in table 1. Resistivity and Hall coefficient measurements were performed at room temperature (RT), at the Dipartimento di Energetica di Firenze (DEF), Italy. The experimental setup [2] performs the analysis by a d.c. four point probe Van der Pauw method [13].

The high temperature annealing (HTA) process was performed one year and an half after the neutron irradiation. During this time the samples were mantained at room temperature. The changes observed in the resistivity for self annealing during this period are reported in [3]. During the HTA process the samples were kept inside the oven at annealing temperatures of 155, 207, 255 and 307 C, with steps of two hours each. All samples were measured before and after each annealing step after half an hour the annealing was finished .

3. Experimental Results

Figures 1 (a) and (b) show the resistivity ρ and Hall coefficient R_h dependence on the neutron fluence after irradiation and after one year and half selfannealing.

The increase in the resistivity below the fluence f_o~$1x10^{13}$ n/cm^2 is followed for higher fluences by a decrease. The Hall coefficient increases its absolute value until f_o is reached and then changes sign switching to positive for higher fluences. We therefore conclude that an inversion from n-type to p-type of the bulk conductivity occurred in the irradiated samples at fluences higher than $1x10^{13}$ n/cm^2. To analyse the results obtained after the annealing process we divide the range of fluences in the following three regions:

(a) $f << f_o$
(b) $f >> f_o$
(c) $f \sim f_o$

Neutron Fluence 10^{13} n/cm^2						
f_1	f_2	f_3	f_4	f_5	f_6	f_7
0.3	0.4	0.6	1.0	2.1	5.7	15.6

table 1. Fluence values of neutron irradiation

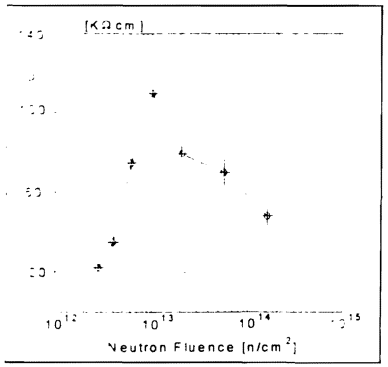

Figure 1 (a). resistivity after irradiation and before annealing

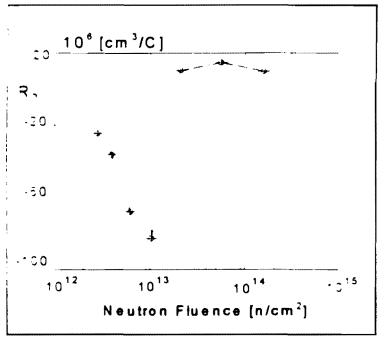

Figure 1 (b). Hall Coefficient after irradiation and before annealing.

The results of the resistivity and Hall coefficient analysis during the annealing process are summarized in the following sections.

Fluence region (a)

For fluences before the inversion of the conductivity type (f_1, f_2 and f_3), the dependence of the resistivity on the annealing temperature is shown in figure 2 (a). A small increase from room temperature (RT) to 150 C is observable, followed by a considerable decrease between 150 and 200 C. The resistivity is approximately constant at higher temperatures. In the plot, the resistivity value before the annealing process increases with the irradiation fluence: this corresponds to the evolution of the bulk towards the intrinsic. After the 150 C annealing, the higher resistivity has been measured for the higher irradiation fluence before inversion. The rate of the resistivity increase with temperature between RT and 150C is the same for fluences f_2 and f_3.

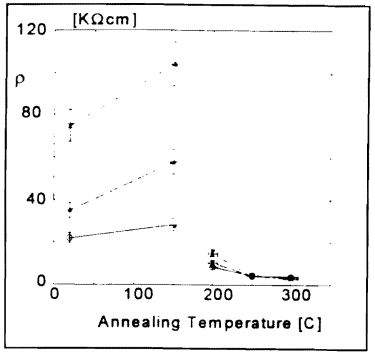

Figure 2 (a). Resistivity change after annealing for samples irradiated with fluences: (-) f_1; (--) f_2; (- · -) f_3.

Figure 2 (b) shows the dependence of the Hall coefficient by the annealing temperature in this fluence region. For fluences f_1 and f_2, the Hall coefficient is approximately constant in the annealing temperature interval, starting from higher absolute values for higher irradiation fluences. A pronounced decrease in absolute value is measured between 150

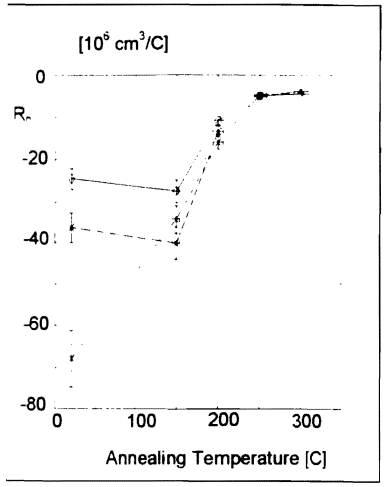

Figure 2 (b). Hall Coefficient changes after annealing for samples irradiated with fluences: (-) f_1; (--) f_2; (- · -) f_3.

and 200C, followed by a slower further increase. For the highest fluence of this group, f_3, a monotonic increase of the R_h absolute value with increasing annealing temperature was measured.

Fluence region (b)

For fluences much higher than f_6, f_6 and f_7, the behaviour of the resistivity, shown in figure 3 (a), can be summarised as a first decrease between RT and 150C, followed by increase between 150C and 200C and by slow decrease for higher temperatures.

The Hall coefficient as a function of the annealing temperature for the same fluence group is shown in Figure 3 (b). The sample irradiated with the fluence f_6 shows a pronounced decrease between 150C and 200C. Both the samples are characterized by an overall positive R_h during the annealing.

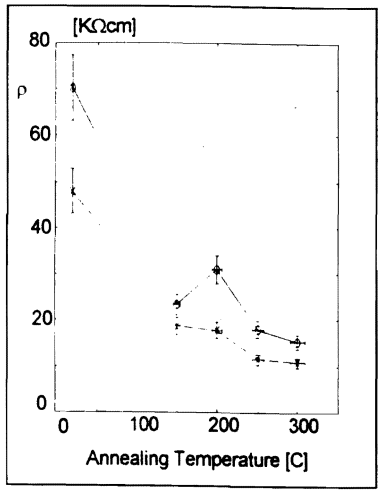

Figure 3 (a). Resistivity changes after annealing for fluences: (-) f_6; (--) f_7.

Fluence region (c)

In the fluence interval close to the inversion point, for fluences f_4 and f_5, the resistivity and Hall coefficient behaviour are reported as a function of the annealing temperature in Figures 4 (a) and (b) respectively. Between RT and 150C the resistivity decreases, as in the region (b) (see figure 3 (a)). Between 150C and 200C, the resistivity decreases in the sample irradiated with the fluence f_4 as for low fluences of region (a), while it is approximately constant in the sample irradiated with the fluence f_5. The Hall coefficient before the high temperature annealing is high and negative for f_4, and positive for f_5. After annealing at 150C, nearly the same values for the R_h and the same dependence on the annealing temperature were measured for the two fluences.

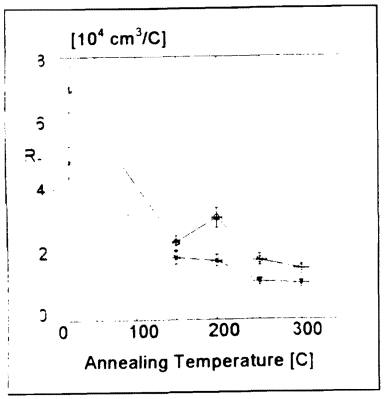

Figure 3 (b). Hall Coefficient changes after annealing for samples irradiated with fluences: (-) f_4, (--) f_5.

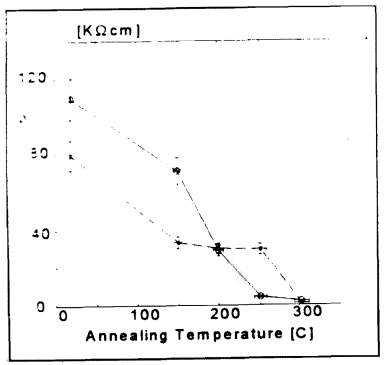

Figure 4 (a). Resistivity changes after annealing for samples irradiated with fluences: (-) f_4 ; (--) f_5.

Of note is the fact that in this region an high inhomogeneity of the samples was observed. The succession of sign in the Hall voltages used to determine the hall coefficient changed in fact during the annealing steps.

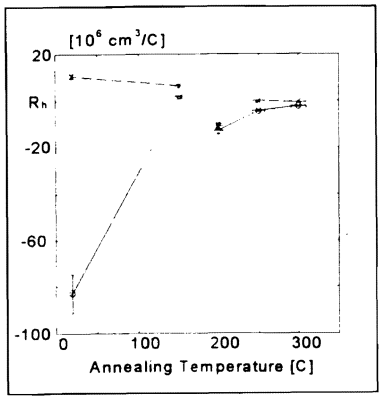

Figure 4(b). Hall Coefficient changes after annealing for samples irradiated with fluences: (-) f_4 ; (--) f_5.

4. Discussion

In the forthcoming discussion the resistivity measurements have been used to determine the behaviour of the free carrier concentrations, while the Hall coefficient ones are used as a confirmation. Hall coefficient depends, in fact, on both the carrier concentration and the Hall factor [1], whose both could vary with the irradiation fluence. In particular, the Hall factor depends on the scattering mechanism of charged carriers: as the high fluence irradiation produces different types of defects, the number of which is increasing with the irradiation fluence, it is possible that the Hall factor changes with the fluence. Let us discuss now the results presented in the previous section. In the fluence region (a), the material is n-type, and the resistivity before annealing increases with the irradiation fluence, corresponding to the evolution of the material bulk towards the intrinsic condition, by acceptor creation. This effect is enhanced by the heating process from RT to 150C. The pronounced decrease of the resistivity between 150 and 200C annealing temperatures, observed in Figure 2 (a), is related with the annealing-out of the E centre or vacancy-phosphorous complex [6]. This is equivalent to phosphorus liberation, and produces a recovery of the n-type conductivity in the material bulk. At higher temperatures, the resistivity remains approximately constant. The annealing-out of the divacancy V_2 is in fact not observed. In this range of resistivities, the Fermi level is situated near the intrinsic level, so that the V_2 defect must be the neutral charge state. The results of Hall coefficient measurements support this interpretation. The Hall coefficient corresponding to the n-type material is negative and is higher for lower electron concentrations. The phosphorous liberation by the E centres produces an increase in the electron concentration and consequently a decrease in the absolute value of the Hall coefficient, as is clearly visible in figure 2 (b) in the 150C to 200C temperature region. In the high fluence region (b), the resistivity decreases from RT to 150C, and increases between 150C and 200C. As for the region (a), the decrease of the resistivity for annealing temperatures between RT and 150C may be related to acceptor creation, while the decrease from 150 to 200 C may be caused by the release of phosphorous from the E centre. In this region in fact the samples, before the annealing process, have already suffered the bulk inversion from n-type to p-type. The acceptor creation so increases the hole concentration decreasing the resistivity, while the release of the phosphorous, increasing the concentration of electrons, which are minority carriers, is responsible of the increase the resistivity in the 150C-200C range. For both the neutron fluences of this region, the Hall coefficient before the high temperature annealing is positive, corresponding to p-type material, the net increase in the hole concentration after annealing corresponds in the plot to a lowering of R_h with increasing the annealing temperature. The acceptor creation between RT and 150C is responsible of a sharp lowering in R_h, while between 150C and 200C the E centre release causes a slow increase of the Hall coefficient. To discuss the intermediate fluence region (c), placed close to the inversion point, we observe that the resistivity and Hall coefficient dependences on the annealing temperature present similarities with both the extreme fluence regions (a) and (b). In particular, between RT and 150C the resistivity decreases as in the high fluence region (b). This means that the two samples are definitely p-type after irradiation. Conversely, from 150C to 200C the resistivity decreases for the fluence f_4 as in the low fluence region (a), while it remains constant for f_5. Adding the fact that a strong inhomogeneity has been observed in samples, we may conclude that both the phosphorous release by the E centre and the acceptor creation, mechanisms occurring during the temperature annealing, bring to the production inside the sample bulk of microscopic regions of different type (n or p).

By an overall view of the analysis, we observe that the dominant mechanisms during high temperature annealing are: the acceptor creation, dominant for temperatures up to approximately 150C, and the E centre release, observed for temperatures at 150-200C. Measurements discussed above evidentiate also two important facts: the first is that heating samples up to 300C allows the recovering of the sample resistivity value, and the second is that bulk inverted to p-type after irradiation does not come back to n-type after annealing. This suggests that created acceptors anneal out for higher temperatures than 300C. Lugakov and Lukashevich [4], analysing silicon samples type inverted after irradiation with ^{60}Co, observed in fact a

complete recovery of the sample type for temperatures over 600C. By concluding, we note that the acceptor creation mechanism measured during the low heating stage up to 150C is probably the same responsible of the reverse annealing effect observed during the room temperature storage of the irradiated samples. Heating the samples up to 150C simply accelerates the reverse annealing effect, keeping it clearly observable in the irradiated samples after short times. A detailed work on the reverse annealing effect is presented in [14].

5. Conclusions

Annealing with temperatures up to 300C has been performed on silicon samples irradiated with fast neutron fluences up to 10^{14}n/cm^2. Before annealing, samples irradiated over $1x10^{13}$n/cm^2 suffered the type conductivity inversion from n-type to p-type. The changes in the resistivity and Hall Coefficient have been measured after each annealing step. Results have been discussed keeping into account both acceptor creation during the low temperature stage of the annealing (from room temperature to 150C) and phosphorous release from the E centre (phosphorous-vacancy complex) at temperatures from 150 to 200C. The acceptor creation may be the same responsible of the reverse annealing effect occurring after long times of storage at room temperature in irradiated samples. Heating samples up to 300C allows the recovering of the sample resistivity to its value before irradiation, with the peculiarity that bulk inverted to p-type after irradiation does not come back to n-type after annealing.

Acknowledgements

Authors wish to thank Dr.Z.Li of the Brookhaven National Laboratory, Upton, NY, for having supplied and irradiated the silicon samples.

References

1. J. P. McKelvey, "Solid State and Semiconductor Physics" , Harper International, New York 1966, p.290.

2. U. Biggeri, E. Borchi, M. Bruzzi, S. Lazanu, Z. Li, and R. Bettenmuller, Conf Proc. of the Italian Physical Society, vol. 46 (1994) 115.

3. U. Biggeri, E. Borchi, M. Bruzzi, and S. Lazanu, presented at the 6-th Pisa Meeting on Advanced Detectors, Isola d'Elba, May 22-28 1994, to be published in Nuclear Instr.& Meth in Phys. Res. A.

4. P.F.Lugakov and T.A.Lukashevich, Phys.Stat.Sol. (a), 110, (1988), 403.

5. G. Lindstrom, E. Fretwurst, H. Herdan, M. Rollwagen, P. Thomsen, and R. Wunstorf, in "Proc. Workshop on Calorimetry at the Supercollider, Tuscaloosa, 1989", ed. R. Donaldson and R. Gilchriese, World Scientific, 1990, p.659.

6. SICAPO Collaboration. "Proc. Workshop on Calorimetry at the Supercollider, Tuscaloosa, 1989", ed. R. Donaldson and R. Gilchriese, World Scientific, 1990, p.489.

7. G.D.Watkins and J.W.Corbett , Phys. Rev. 121, 1001, (1961)

8. Y.Tokuda, and A.Usami, IEEE Trans. Nucl. Sci. NS-28, 3564 (1981)

9. E.Borchi, C.Bertrand, C.Leroy, M.Bruzzi, C.Furetta, R.Paludetto, P.G.Rancoita, L.Vismara and P.Giubellino, Nucl. Instr & Methods, A279, 277 (1989)

10. D.V.Lang, J.App.Phys. 45, 3023 (1974)

11. M.G.Bueheler, Solid State Electron. 15, 69 (1972)

12. G. H. R. Kegel, P. F. Bertone, D. L. Case, D. J. Desimone, C. K. Jen, and C. Narayan Conf. Proc. of the Italian Physical Society, vol. 46 (1994) 87.

13. L. J. Van der Pauw, Philips Res. Rep. 13 (1958) 1

14. U.Biggeri, E.Borchi, M.Bruzzi, J.Bates, M.Glaser, F.Lemeilleur, and C.Furetta, "Self Annealing Effect on Neutron Irradiated Silicon Detectors Investigated by TSC Analysis", these Proceedings.