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Hall Effect measurements on proton-irradiated ROSE samples

U.Biggeri, M.Bruzzi, E.Borchi
I.N.F.N. of Florence and Dipartimento di Energetica,
Via S.Marta 3, 50125 Firenze, Italy

S.Lazanu
University of Bucharest, Bucharest, Romania

Z.Li
Brookhaven National Laboratory, Upton, N.Y.

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1. Introduction

Strong efforts have been spent in past to study the radiation-induced damage in silicon detectors to be used in high-level radiation environment as LHC [1]. Up to now, works are focused in finding possible solutions to limit this damage: the ROSE (RD48) collaboration in particular is intended to develop alternative process technologies to increase the radiation hardness of the silicon devices.

The degradation of the silicon detector performance due to irradiation is mainly caused by the creation of a multiplicity of vacancy-related lattice defects and clusters in the silicon bulk. The introduction of interstitial impurity atoms during the growth process, as carbon, oxygen, nitrogen, tin, is believed to act as a vacancy sink during and after irradiation, so limiting the detector bulk damage. For this reason, the RD48 collaboration decided to analyse the radiation damage of exotic silicon produced with large concentrations of such impurities in the starting material.

In this study, two sets of bulk samples obtained from wafers with a different oxygen content have been analysed by Hall and Van der Pauw techniques. Measurements have been carried out in the temperature range from 18K to 370K. The samples have been irradiated with 24 Gev/c protons up to $2\text{x}10^{14}$ p/cm^2; a thermal treatment (30 minutes at 100C) was performed to accelerate the reverse annealing process. Bulk resistivity and Hall coefficient versus temperature have been measured after irradiation and after the annealing treatment in order to

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investigate the sample bulk properties, to obtain informations both on the free carrier concentrations and on the carrier mobility.

2. Experimental Details

In this study two wafers (P40 and P126) of a silicon monocrystal material produced by Float-Zone refinement in Polovodice (Prague) have been analysed. One of the two wafers (P126) has been cut from an oxygenated ingot: during the refinement a pure oxygen gas jet was injected in the region of the RF heating coil. Infra Red absorption measurements made in Prague showed an oxygen content around 10^{16} atoms/cm^3. The two wafers have been processed at BNL, Upton, NY, to produce Van der Pauw samples. Four aluminum dots have been evaporated on the silicon skratched surface of each 1cm^2 square sample. Almost thirty Van der Pauw samples have been obtained from each wafer.

Bulk resistivity and Hall coefficient have been measured by means of an Hall effect Keithley facility at Dipartimento di Energetica di Firenze (DEF). A Varian magnet supplied a transverse magnetic field of 5000 Gauss. During the measurement the sample under test wass placed inside a Galileo K1 helium closed-cycle cryogenerator to perform thermal scans in the range from 18K to 370K. The temperature was measured using a carbon glass resistor driven by a Lake Shore DRC91 temperature controller. Details on the experimental setup are given elsewhere [1].

Six couples of samples from P40 (p) and P126 (o) wafers have been irradiated at the CERN PS facility with a 24 Gev/c protons fluence from 10^{13} p/cm^2 to 2x10^{14} p/cm^2: irradiation fluences are listed in table 1. In the table, the same sample number refers to the same position in the wafer. After the irradiation, a thermal treatment (30 minutes at 100C) was carried out to accelerate the reverse annealing process in the silicon bulk.

Fluence 10^{13} p/cm^2	Pure sample (P40)	Oxygenated sample (P126)
0.98	p53	o52
1.98	p54	o53
4.71	p55	o54
7.30	p57	o56
10.12	p58	o57
17.44	p59	o58

table 1. Irradiation fluences.

3. Experimental Results

Bulk samples obtained from wafers P40 and P126 have been measured by means of the Hall effect and of the Van der Pauw techniques before irradiation. Mainly the same behaviour was found for the pure P40 (p) and oxygenated P126 (o) samples. The average resistivity at room temperature is 2.45KΩcm through the two sets, with a relative error close to 15% .

Figure 1 shows the resistivity of a not-irradiated samples versus the temperature measured from 18K to 370K (sample o66) and fitted using [2]. Three regions are clearly distinguishable, in agreement with literature [2]. At high temperatures the bulk behaves as intrinsic, due to the fact that the electron - hole pair generation dominates. At intermediate temperatures (approximately from 50K to 350K) the bulk is extrinsic: the e-h generation is negligibile and the free carrier concentration is constant. The almost quadratic resistivity dependence on the temperature is due to the carrier mobility. In the low temperature region (below 50K) the carrier frozen-out effect produces an increase in the bulk resistivity. The resistivity peak observed going from the extrinsic to the intrinsic regions is shown in a logarithmic plot in figure 2a (p66). Two exponential trends are clearly visible, corresponding to energy levels close to 0.56eV in the high temperature region and around 0.06eV, close to the the shallow dopant energy level, in the low temperature one.

The Hall coefficient and Hall mobility have been also measured through the two wafers. Figure 2b shows the Hall coefficient dependence on temperature in the range from 10C to 80C (o66). The two different exponential slopes, corresponding to the extrinsic and intrinsic regions, are clearly visible. Table 2 shows the ρ, R_h and μ_h values for two reference samples, p66 and o66 at 27C, together with the shallow donor concentration N_D. The Hall mobility $\mu_h \approx \mu_n{*}r_h$ evidences the n-type conductivity and the Hall factor r_h is found to be around 1.18, corresponding to acoustic-phonon scattering.

sample	ρ [KΩ cm]	R_h [10^6cm^3/C]	μ_h [cm^2/Vs]	N_D [10^{12} cm^{-3}]
p66	2.39	-4.48	1778	1.65
o66	2.52	-3.74	1565	1.97

Table 2. Resistivity, Hall coefficient, hall mobility and shallow donor concentration for the pure (p66) and oxygenated (o66) samples used as a reference.

In figure 3 the bulk resistivity of an irradiated sample (4.7×10^{13} p/cm^2) is compared with that of a not-irradiated one: the shape of the two curves is quite different. The irradiated sample is no more characterised by an extrinsic region at intermediate temperatures: the exponential behaviour of the intrinsic range completely dominates the investigated temperature range down to 100K. This is probably due both to the shallow donor removal occurring during the irradiation and to the radiation-induced creation of deep energy levels acting as generation-recombination centers in the silicon band gap. The resistivity of the irradiated sample reachs values around 10^3 MΩcm, and, below 100K, it saturates: this is probably due to a background limit in the current read out of the Hall Effect Keithley facility. Currents as low as few pA are in fact flowing inside the sample under test in this temperature region.

The exponential behaviour with temperature corresponding to the presence of deep energy levels in the silicon gap has been also observed in the irradiated samples by measuring the Hall coefficient R_h: an example is given in figure 4 (7.30×10^{13} p/cm^2). R_h decreases its absolute value increasing the temperature, due to an increase of the free carrier concentration. The $\rho(T)$ and $R_h(T)$ exponential shape observed measuring the irradiated samples has been fitted to determine the corresponding deep energy level: values around 0.6-0.7eV have been determined for all the investigated (pure and oxygenated) samples.

Table 3 shows the resistivity, Hall coefficient and Hall mobility measured at 27C for the six couples of irradiated pure and oxygenated samples before the annealing treatment. Table 4 lists the same parameters measured at 27C after the heating treatment, accelerating the reverse annealing process.

	pure			oxygen.		
Fluence [p/cm^2]	ρ [KΩ cm]	R_h [cm^3/C]	μ_h [cm^2/Vs]	ρ [KΩ cm]	R_h [cm^3/C]	μ_h [cm^2/Vs]
0.98×10^{13}	35.23	-2.22×10^7	631.41	32.77	-2.24×10^7	683.97
1.98×10^{13}	73.3	-6.12×10^7	834.68	72.64	-5.61×10^7	773.04
4.71×10^{13}	95.38	-6.66×10^7	698.49	75.11	-4.24×10^7	565.30
7.30×10^{13}	124.94	-3.92×10^7	313.81	127.82	-3.81×10^7	297.83
10.12×10^{13}	92.97	-1.45×10^7	155.96	112.80	-2.32×10^7	205.57
17.44×10^{13}	110.63	-9.82×10^6	88.66	113.36	-1.98×10^7	174.36

Table 3. Resistivity, Hall coefficient and Hall mobility at 27C after irradiation.

	pure			oxygen.		
Fluence [p/cm^2]	ρ [KΩ cm]	R_h [cm^3/C]	μ_h [cm^2/Vs]	ρ [KΩ cm]	R_h [cm^3/C]	μ_h [cm^2/Vs]
0.98x10^{13}	*	*	*	29.06	-1.90x10^7	653.82
1.98x10^{13}	69.53	-4.75x10^7	682.87	64.33	-4.05x10^7	634.50
4.71x10^{13}	106.89	-2.42x10^7	320.44	110.61	-2.30x10^7	209.10
7.30x10^{13}	102.65	-7.58x10^6	73.88	105.30	-8.17x10^7	70.83
10.12x10^{13}	79.40	+0.61x10^6	7.57	85.53	+0.14x10^5	1.59
17.44x10^{13}	78.72	+4.10x10^6	38.40	85.36	+2.24x10^6	26.19

Table 4. Resistivity, Hall coefficient and Hall mobility at 27C after the annealing treatment. Sample p53 was incidentally broken during the thermal cycle.

Before the thermal treatment, the resistivity increases with increasing the fluence reaching a plateau around 100 KΩcm for fluences over 4.71x10^{13} cm^{-2} for both the pure and the oxygenated samples. After the annealing, the three more irradiated couples show approximately a 20% decrease in resistivity. The Hall coefficient, before the thermal cycle, is negative in all the samples, but it switches to positive in the two more irradiated couples after the heating treatment. As a consequence of the R_h switch in sign an Hall mobility minimum is found at 1x10^{14} p/cm^2.

4. Discussion

To analyse the resistivity and the Hall coefficient dependence on the fluence, a simple model taking into account of the radiation-induced creation of an equivalent deep acceptor in the silicon bulk is considered [3]. Recent studies have in fact pointed out that, if the main consequence of radiation is to create a multiplicity of levels inside the silicon energy gap, the change in the fundamental electrical properties can be favourably described considering that a unique deep acceptor level appears in the band gap, with a concentration linearly increasing with the fluence by an introduction rate β. The model takes into account of an exponential removal of the initial shallow donors. The $\rho(T)$ and $R_h(T)$ exponential fits obtained for the irradiated samples strongly support the hypotesis of an equivalent acceptor level around 0.6-0.7eV, already suggested in [3]. Figures 5a and b show the resistivity, Hall coefficient measured before and after the annealing treatment in the pure and oxygenated samples. The fits have been obtained considering Et = 0.64eV and $\beta = 0.07$ 1/cm and one of them accounts for the reverse annealing effect simulated by the thermal treatment.

The fundamental equations relating the resistivity and the Hall coefficient to the free carrier concentrations and mobilities are the following:

$$\rho = (q \cdot \mu_p \cdot (p + z \cdot n))^{-1} \qquad Rh = 1/q \cdot (p - z^2 \cdot n)/(p + z \cdot n)^2$$

where $z = \mu_n/\mu_p$, $\mu_n(27C) = 1400 cm^2/Vs$, $\mu_p(27C) = 450 cm^2/Vs$. Due to the difference in the electron and hole mobility, R_h switches from negative to positive when $p = z^2 \cdot n$:, this means that the hole concentration is almost an order of magnitude higher than the electron one. To determine the inversion point, the expression of n can be derived:

$$n = 1/((z+z^2) \cdot \rho \cdot q \cdot \mu_p) \cdot (1 - R_h/(\rho \cdot \mu_p))$$

To determine the hole concentration, p, the well known action-mass law can be used. Results are shown in figures 6 a, b, c, d: the free carrier concentrations n and p are reported respectively for the pure and oxygenated samples before and after irradiation. In the plots, it is clearly visible the increase in the hole concentration with increasing the fluence. The inversion fluence (n = p) is found to be around $7x10^{13}$ p/cm^2 and $4x10^{13}$ p/cm^2 respectively before and after the annealing treatment. The free carrier concentration dependence on the fluence is quite similar for the pure and oxygenated sets. However, some differences are present, before the heating cycle, in the more irradiated samples. The oxygenated ones are in fact still quite intrinsic both at $1x10^{14}cm^{-2}$ and $1.7x10^{14}cm^{-2}$, while the corresponding pure samples present a hole concentration definitely higher than the electron one. This difference seems to disappear after the annealing treatment. The overall samples irradiated with fluences over $2x10^{13}$ p/cm^2 , before and after the annealing treatment, should nevertheless considered as quasi-intrinsic because of the low values found both for n and p. The poor radiation hardening effect due to the oxygen content can be explained considering that the oxygen concentration is still rather low inside the silicon lattice to produce evident effects. The Polovodice silicon monocrystal material was produced by Float-Zone refinement: if too much oxygen is introduced during this process, the oxygen precipitates and the ingot is no more a monocrystal. By this method no more than 10^{16} oxygen atoms / cm^3 can be introduced in comparison of $5x10^{17}$ cm^{-3} which has been requested by the ROSE collaboration. This can explain why the radiation hardness of the oxygenated Polovodice material is not much better than the standard material one.

5. Conclusions

Van der Pauw samples cut from two wafers of a silicon monocrystal material produced by Float-Zone refinement have been tested by the Hall effect technique. One of the two wafers have been enriched with oxygen during the growth process.. Two sets of six pure and oxygenated samples have been irradiated with 24 GeV/c protons in the fluence range from 10^{13} p/cm^2 to 2x10^{14} p/cm^2. A thermal treatment (30 minutes at 100C) has been performed to accelerate the reverse annealing effect in the irradiated silicon. Resistivity, Hall coefficient and Hall mobility have been measured before and after irradiation in a temperature range from 18K to 370K. The extrinsic region is present from 100K to 350K only in the not-irradiated samples. For higher temperatures the not-irradiated semiconductor shows an intrinsic behaviour. In the low temperature region (below, 50K) the carrier frozen-out is visible, due to a partial excitation of the shallow dopants. The irradiated samples show a quasi-intrinsic exponential dependence on the temperature from 370K to 100K: the extrinsic and frozen-out regions are no more present. This can be explained considering that radiation produces both a progressive removal of the shallow dopants in the silicon bulk and a continuous creation of deep energy levels in the silicon gap. The slope of the exponential $\rho(T)$ and $R_h(T)$ curves leads to energy levels around 0.6-0.7eV in the irradiated samples.

The free carrier concentrations (n, p) have been evaluated at 27C in the whole investigated fluence range. The intrinsic condition ($n = p$) was found around 7x10^{13} p/cm^2 and 4x10^{13} p/cm^2 respectively before and after the annealing treatment. Only slight differences have been detected between the pure and oxygenated sets. Before the heating cycle and for $f \geq 1x10^{14}$ p/cm^2, the oxygenated samples are still quite intrinsic, while the corresponding pure twins present a hole concentration definitely higher than the electron one. The slight radiation hardening effect due to the oxygen content can be explained considering that the oxygen concentration , limited to 10^{16} cm^{-3} during the growth process to prevent the oxygen precipitation inside the monocrystal, is too low to produce evident effects in terms of the radiation hardness of the devices.

Acknowledgements

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[2] M.Sze, Semiconductor Physics,Wiley, NY, 1982.

[3] U.Biggeri, E.Borchi, M.Bruzzi, S.Lazanu, Z.Li: CV and Hall effect analysis in neutron irradiated samples, to be published on Nuclear Instruments & Methods A, 1997.

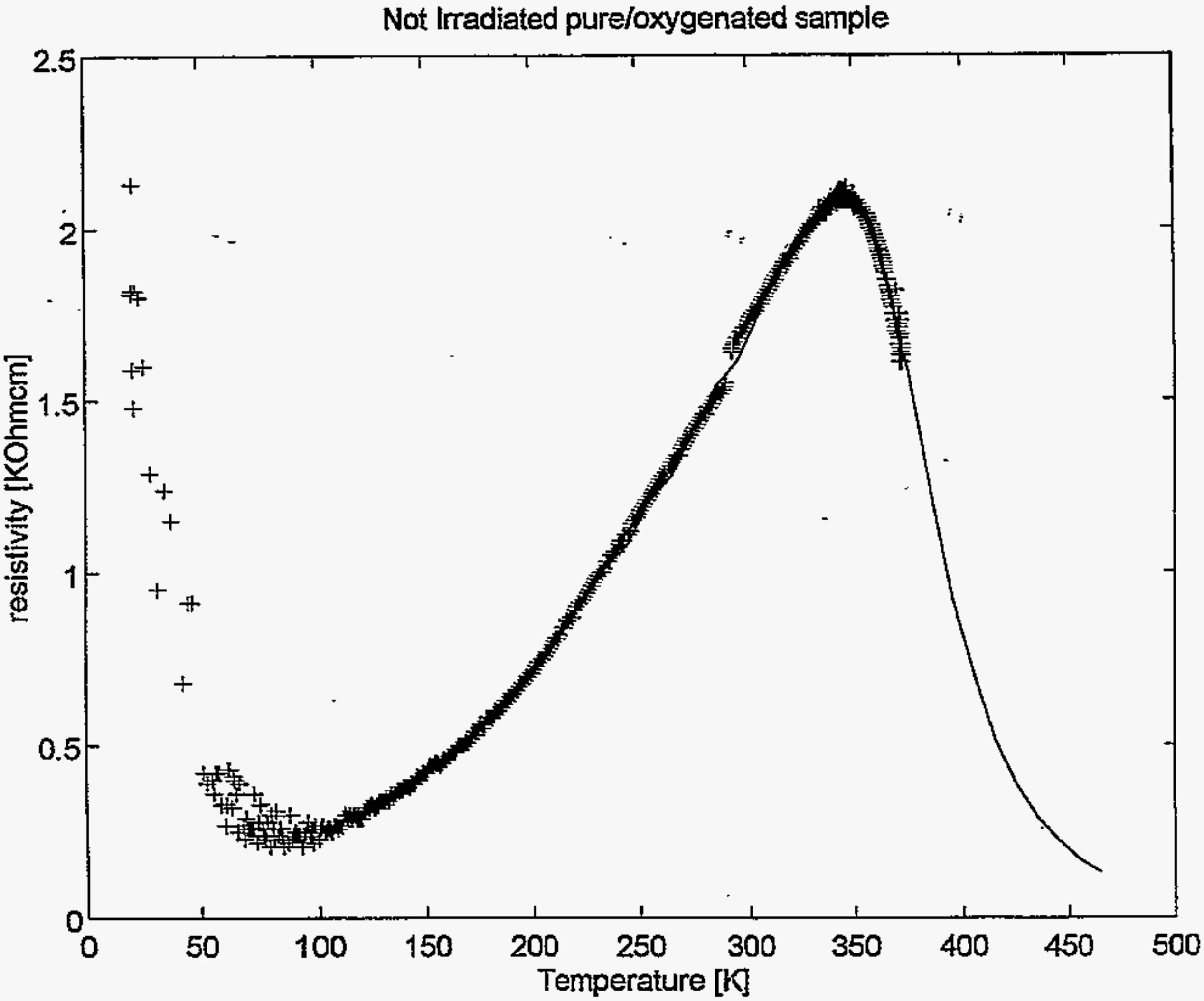

Figure 1 . Resistivity versus temperature for a ROSE sample before irradiation

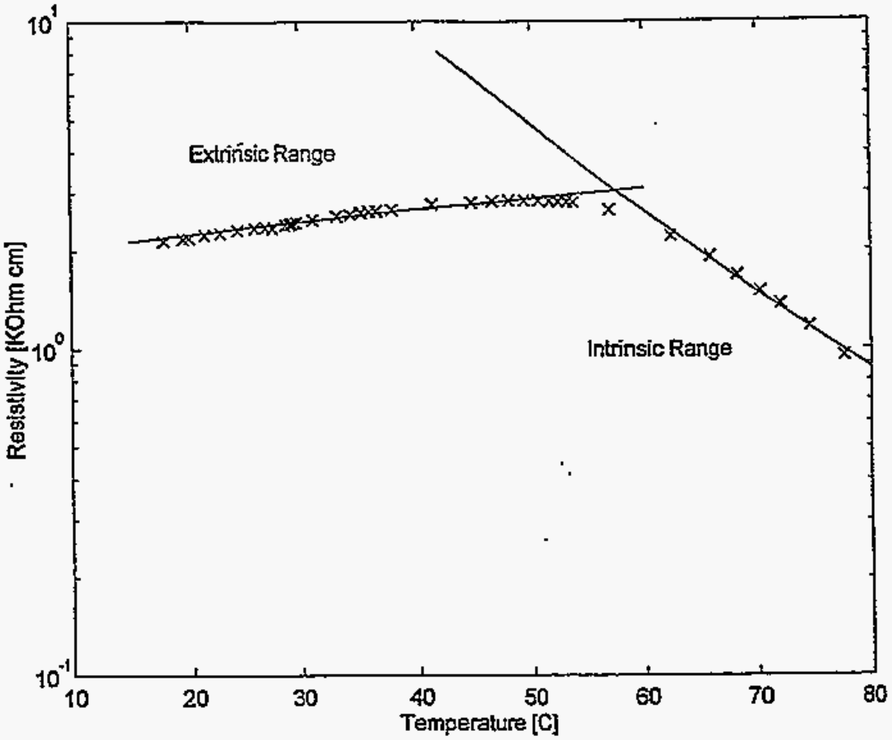

Figure 2a. Resistivity versus temperature for a ROSE sample before irradiation: Exponential fits evidencing the Extrinsic and Intrinsic ranges are added.

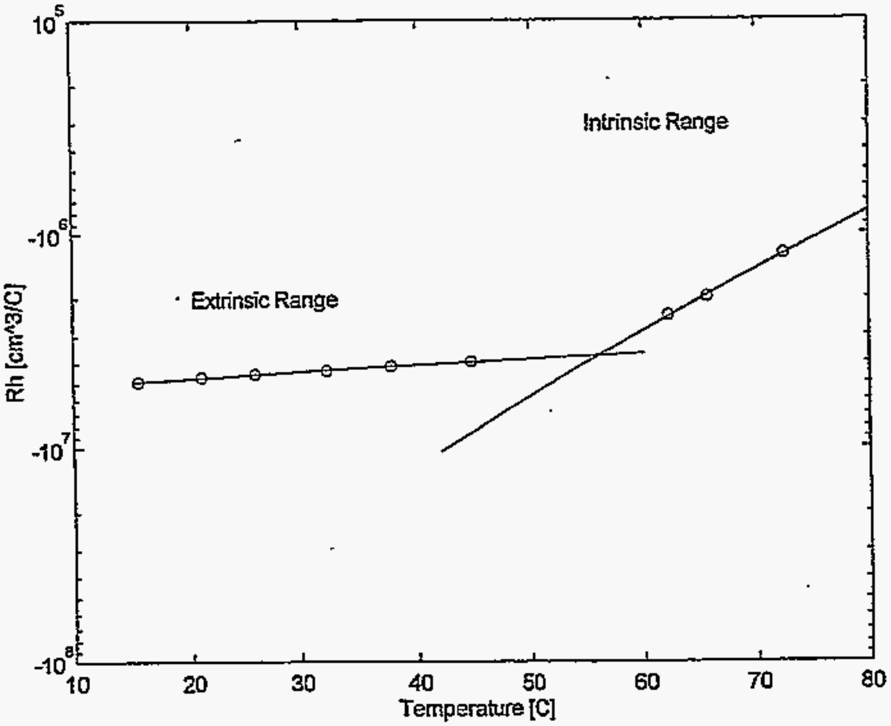

Figure 2b. Hall coefficient versus temperature for a ROSE sample before irradiation: Exponential fits evidencing the Extrinsic and Intrinsic ranges are added.

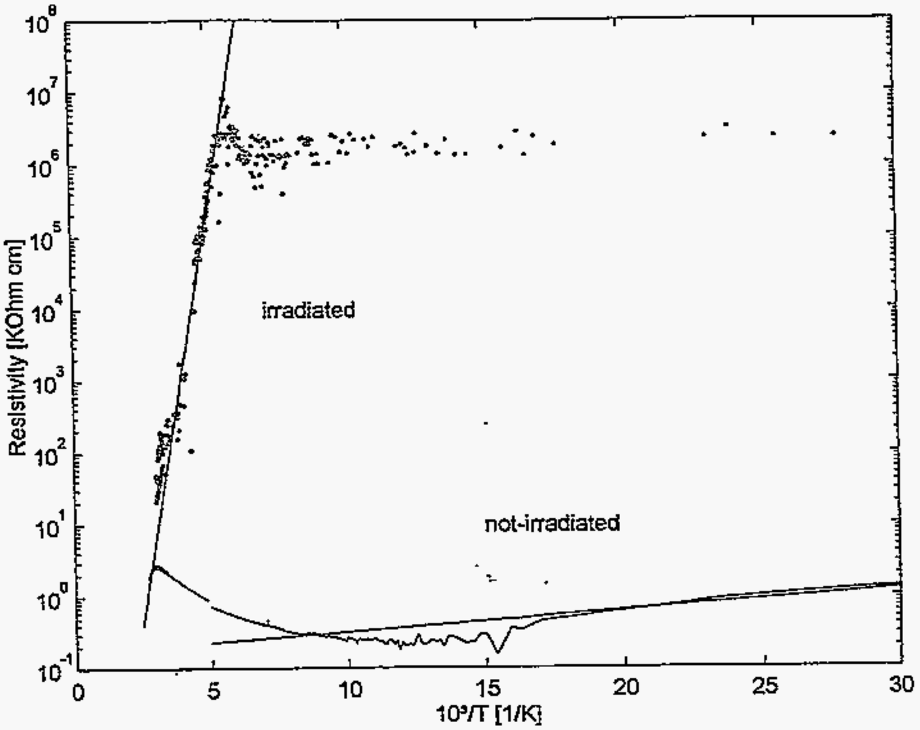

Figure 3 . Resistivity dependence on temperature for an irradiated ROSE sample and a not-irradiated one. Exponential fits evidencing the Extrinsic and Intrinsic ranges are added.

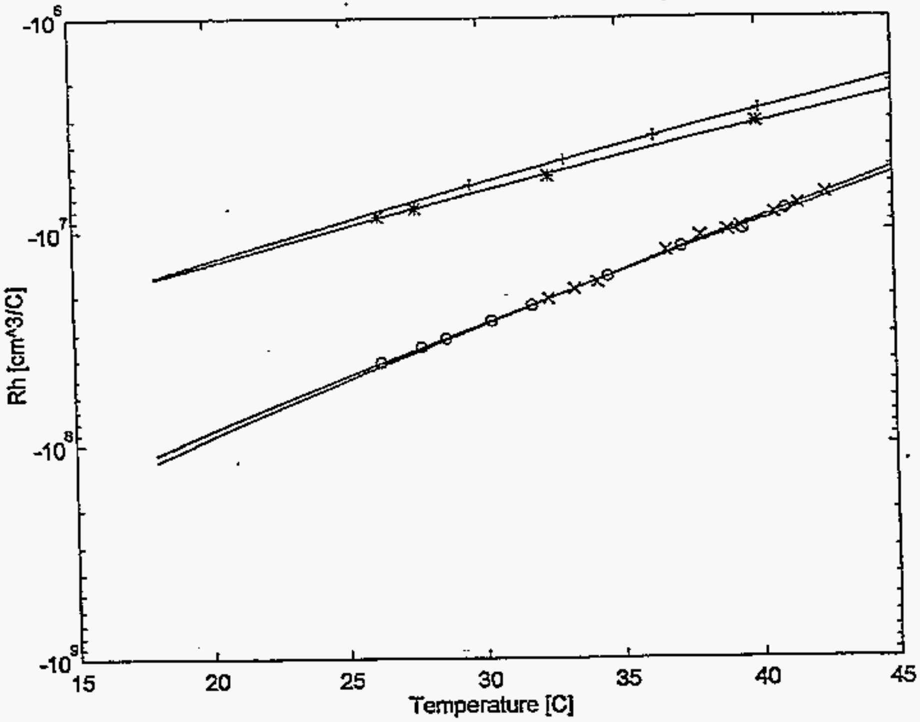

Figure 4 . Hall coefficient dependence on temperature for two ROSE sample irradiated with $7.3\text{x}10^{13}$ p/cm^2 : (+) p56 before annealing (*) o56 before annealing (x) p56 after annealing (o) o56 after annealing. Exponential fits are also given.

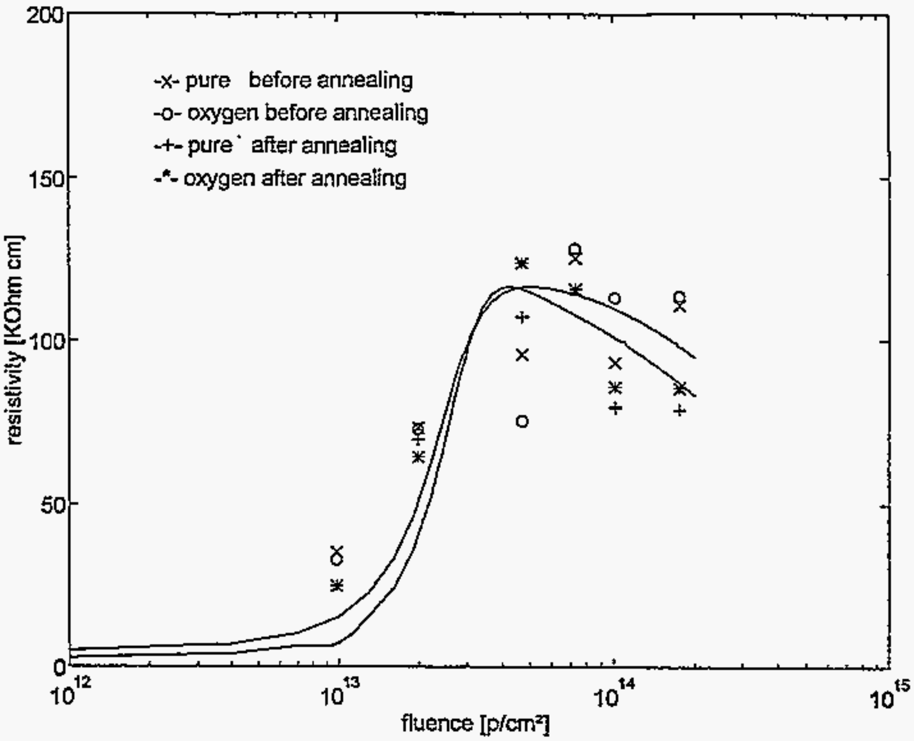

Figure 5a. Resistivity (measured at 27C) versus fluence for pure and oxygenated samples before and after the annealing treatment.

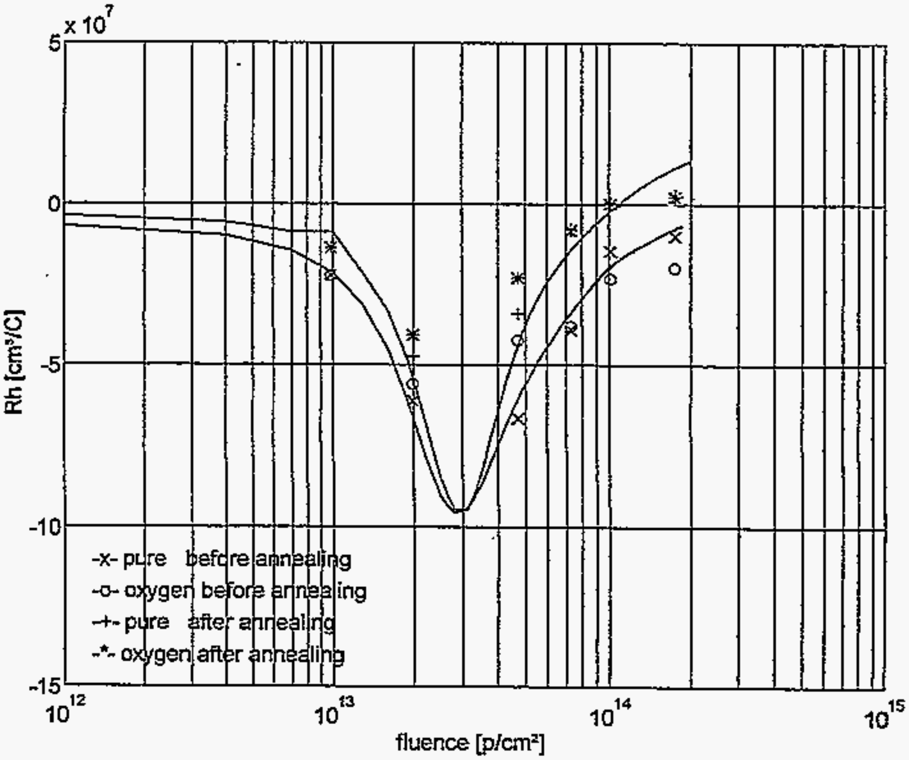

Figure 5b. Hall coefficient (measured at 27C) versus fluence for pure and oxygenated samples before and after the annealing treatment.

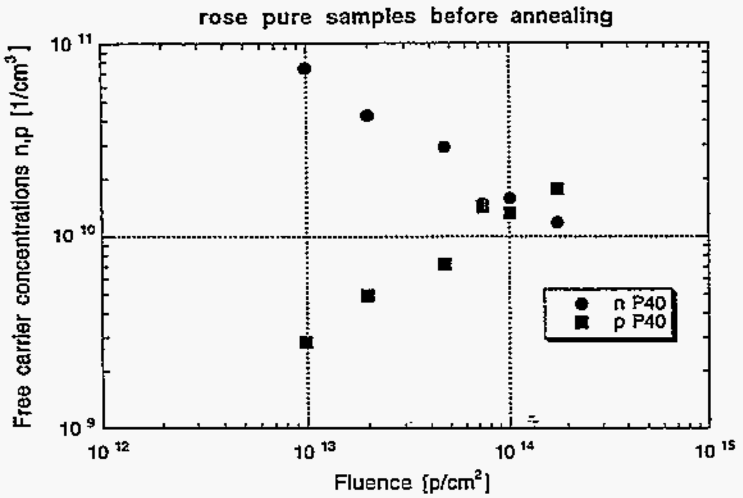

Figure 6a. Free carrier concentrations n and p calculated for the pure samples (P40) after irradiation and before the annealing treatment.

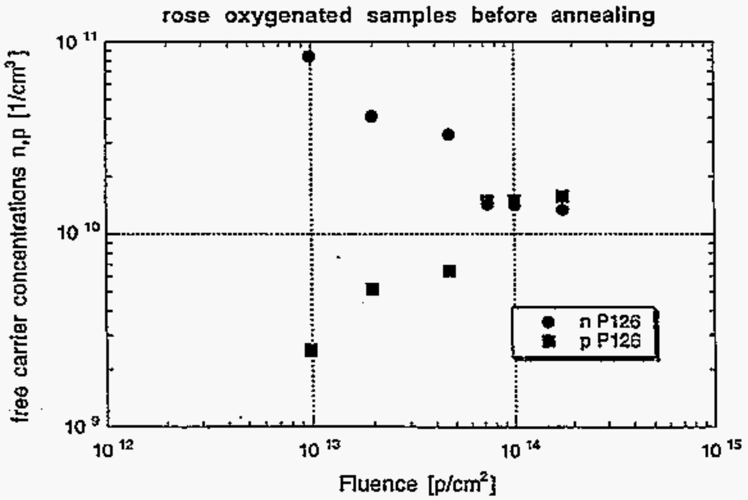

Figure 6b. Free carrier concentrations n and p calculated for the oxygenated samples (P126) after irradiation and before the annealing treatment.

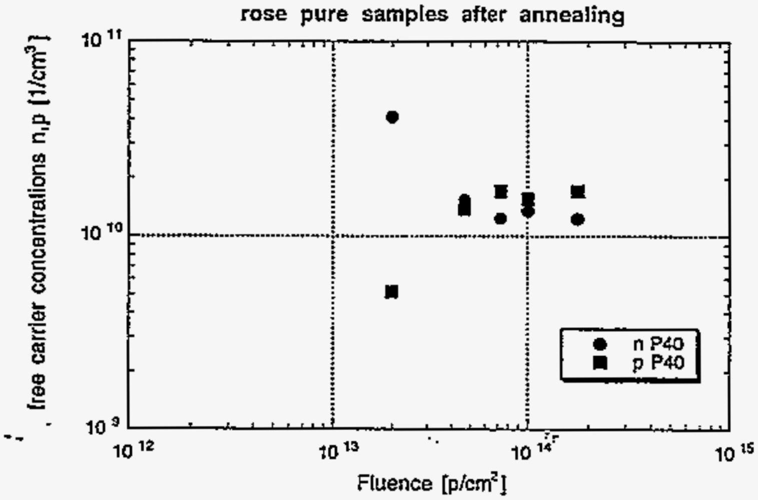

Figure 6c. Free carrier concentrations n, p for pure samples after annealing

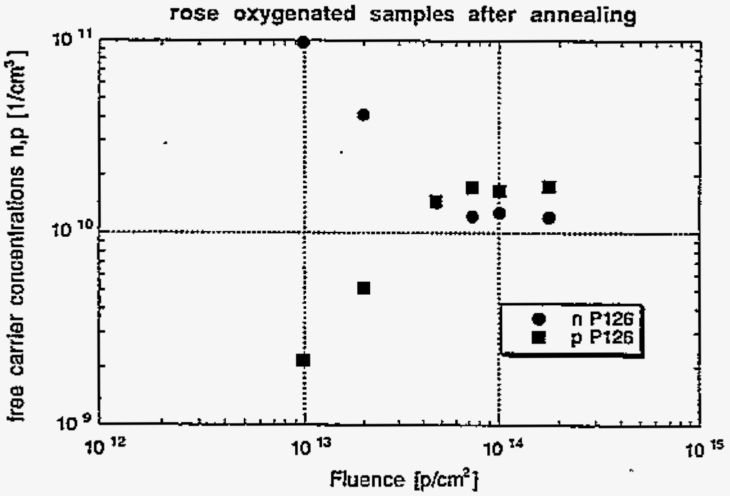

Figure 6d. Free carrier concentrations n,p for the oxygen. samples after annealing.