BUCHAREST UNIVERSITY

EXPERIMENTAL PARTICLE PHYSICS GROUP

UBPub.EPPG/Phys.21
February 1995

NEW RESULTS IN EXPERIMENTAL STUDIES OF RADIATION DAMAGE OF SILICON DETECTORS

(Progress Report)

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Abstract

The results obtained in the characterization of defects induced by neutron irradiation in the silicon lattice are reviewed: their dependence on the annealing time, and their spatial distribution. A general characterization of the neutron irradiated silicon by resistivity, Hall coefficient, C-V, I-V and spectroscopic measurements is given too.

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The results obtained in the characterization of defects induced by neutron irradiation in the silicon lattice are reviewed: their dependence on the annealing time, and their spatial distribution. A general characterization of the neutron irradiated silicon by resistivity, Hall coefficient, C-V, I-V and spectroscopic measurements is given too.

INTRODUCTION

The radiation damage in silicon detectors is extensively studied (see, e.g. [1] and [2]) in connection with their utilization at the Large Hadron Collider that will be constructed at CERN. It is believed that silicon detectors will survive the intense radiation environment only in limited regions of the detectors planned for LHC and elsewhere. Research studies are developing in parallel for other materials, e.g. diamond.

The phenomena that take place in the silicon lattice in radiation fields are not well understood, and this is the reason that the behavior of silicon detectors, in specific situations (radiation fields: particles, energy spectra, temperatures during irradiation and annealing, annealing time) is not known and is a matter of study. A lot of pieces are missing in this puzzle of silicon radiation damage, starting from the identification of microscopical defects in the forbidden energy band of silicon, from their interaction processes in the material, and arriving to the troublesome problem of reverse annealing of the effective impurity concentration in reverse biased p-n junctions.

There are now some limits established for the use of silicon detectors, regarding the optimal temperature at which the detectors should run and should be kept during the "unworking" periods [4]-[5]. The role of radiation damage studies is to understand and, if possible, to enlarge these limits.

The present paper is a review of the 1994 common research of the groups from the Bucharest University and the Institute for Atomic Physics, the Joint Institute for Nuclear Research - Dubna, the INFN and the University of Florence and the Brookhaven National Laboratory. It is a continuation of the 1993 work, reported in [3], where the field of interest of each part in this collaboration is also shown.

The studies which results we report here, and that are a continuation of the investigations made the previous year, were focused on the characterization of the damage induced in high resistivity silicon by fast neutrons, both at the microscopic level (specifically by the Thermostimullated Currents method), and at the macroscopic level, by resistivity, Hall coefficient, I-V and C-V measurements. As a more general situation in physics, the passage from microscopic to macroscopic characteristics is not done, and in fact cannot be done for the moment, because some important information and steps are missing. So, for the moment, the studies are developing separately at the macroscopic and microscopic level. In the following, the results obtained in the characterization of defects induced by neutron irradiation in the silicon lattice will be presented: their dependence on the annealing time, and their spatial distribution.

A general characterization of the irradiated silicon by resistivity and Hall coefficient measurements will be given too, as well as the effects of high temperature annealing. Some of the results obtained from measurements made on silicon detectors will be summarized too. Special attention will be paid to the neutron facilities properties necessary for the characterization of the neutron damage.

DAMAGE CHARACTERIZATION BY RESISTIVITY AND HALL COEFFICIENT MEASUREMENTS

The defect centers produced by irradiation are leading to free carrier concentration modifications, due to the interactions with the doping atoms. In the same time, these centers are supplementary scattering centers, that are acting towards the decrease of the mobility. These mechanisms are leading to modifications of the resistivity and Hall coefficient, as measurable physical quantities.

Irradiation induced changes of electron and hole concentrations are important for the clarification of the controverted problem of the inversion of the conductivity type. Up to now, the inversion was more studied in p-n junction structures, by C-V [6] and charge collection measurements [7].

The material investigated in this study was high resistivity, n-type, <111>, float zone silicon, with initial resistivity 4-6 KOhm*cm, from Wacker. Square shaped samples of 10x10 mm¬2 area and 1mm thickness, with four aluminium ohmic contacts in the corners, have been utilized [8]. The samples have been made at the Brookhaven National Laboratory, USA. Then the samples have been irradiated with fast neutrons [9], at the University of Lowell, the neutron fluence varied from 10¬12 to 10¬14 n/cm¬2. The changes in the resistivity and Hall coefficient induced by the neutron irradiation have been studied using d.c. four point probe measurements, at room temperature, at the Department of Energetic laboratories, Florence [10].

The dependencies of the resistivity and Hall coefficient on the neutron fluence are presented in fig 1. The resistivity first increases with the irradiation fluence, near the value corresponding to intrinsic resistivity of silicon, where reaches a maximum, in agreement with the results reported by other groups [11-12]; then a decrease of the resistivity at a further increase of the fluence is obtained. The Hall coefficient starts from low negative values, at low fluences, decreases with the increase of the fluence up to a minimum, and after that increases sharply and changes sign. This behavior corresponds to the decrease of the electron concentration in the sample, with the corresponding increase of hole concentration, to satisfy the mass action law.

MODELING OF THE RESISTIVITY

Bulk silicon degradation is understood today in terms of interaction of fast neutrons with the silicon atoms, with the formation of (Si sub(i)-V) pairs, unstable, and which, in their turn, recombine and form stable defect complexes with some impurities or other defects they found in their way in the lattice. If the impurities that are participating to the formation of defects are doping centers, as in the case of phosphorus (P) and boron (B), with the formation of P-V and B-V defects, a decrease of the doping concentration results. The defects generated by irradiation correspond to different energy levels in the band gap, which charge states depend on the relative position of the Fermi level. The defect structures produced by moderate irradiation fluences, in the order of 10¬12 n/cm¬2, had been studied by different physical methods, and the correspondence between the lattice defect and the energy level done [13]. The increase of the irradiation fluence conducts to the increase of concentrations of generated defects, and, in the same time, at the observation of more defects [14]. An interpretation of the resistivity of the irradiated silicon in terms of P-V and B-V center formation could be found in [15]. The most important contributions to the change of resistivity with the irradiation fluence in homogeneous samples could come from the modifications of doping concentrations. The presence of levels induced by irradiation is not important in the measurement of resistivity, as they are neutral and are not changing the charge state. The P and B concentrations are modifying at irradiation due to the their interactions with the silicon interstitials and vacancies, according to the following reactions:

n + Si sub(s) -> Si sub(i) + V (1)
P sub(s) + V -> P-V (2)
Si sub(i) + B -> Si sub(s) +B sub(i) (3)
B sub(i) + V -> B-V (4)

The up-mentioned reactions are neither the only ones where Si interstitials and vacancies are participating, nor the most important: the main mechanism of their disappearance is their mutual annihilation, and by the most important impurities with which they are forming stable defects is oxygen. Other sinks for them could be other substitutional impurities, defect clusters, dislocations and crystal surfaces. In the present model it will be assumed that the reactions (1)-(4) are important only for the decrease of P and B doping concentrations, and that other reactions will not modify the carrier concentrations. While the P-V defect is stable at room temperature, the B-V complex self anneals before room temperature, and restores this way the boron. The difference in the reactions of formation of P-V and B-V reflects in the dependence on fluence of P and B doping concentrations:

$$N_{P} = N_{0P}\exp(-b_{P}F) \qquad (5)$$
$$N_{B} = N_{0B}(b_{1} + b_{2}\exp(-bF)/b \qquad (6)$$

where N_{0P} and N_{0B} represent the P and B concentrations before irradiation, and the b parameters (b_P, b_1 and b_2, with $b = b_1+b_2$) are related to the probabilities of formation of the defects. Starting from the neutrality equation in the silicon bulk, from the mass action law and from the expression of resistivity in a semiconductor with ambipolar conductivity, the parameters N_{0P}, N_{0B}, b_P, b_1 and b_2 where determined by fit. It was assumed that the conduction mobility does not vary with the neutron fluence. Taking into account a decrease of the conduction mobility with the irradiation fluence, in agreement with the results reported in [16], the quality and the parameters of the fit do not change significantly. One year of selfannealing at room temperature does not change the shape of the resistivity versus fluence curve [15].

RESULTS AND INTERPRETATION OF HALL MEASUREMENTS

Room temperature Hall measurements could provide information about the Hall mobilities as a function of the neutron fluence. The Hall coefficient was calculated, from the electron and hole concentrations obtained from resistivity measurements, supposing that the predominant scattering mechanism of charge carriers is on ionized impurities, and taking into account the correction for non spherical energy surfaces of silicon [17]. A good agreement with the measurements has been obtained, up to fluences in the order of 10^{13} n/cm^{2}. To keep the same concordance, special assumptions are to be made on the Hall mobility, both for electrons and holes. Again, the introduction of a variation of the conduction mobility as a function of the neutron fluence has no so much influence on the Hall coefficient. The degradation of both hole and electron Hall mobilities, pronounced after fluences in the order of 10^{13} n/cm^{2} is the determining factor which could explain this discordance. The degradation of electron and hole Hall mobilities has to be correlated with the high concentration of radiation induced point defects and clusters, which are introducing supplementary scattering mechanisms and are changing their relative importance.

HIGH TEMPERATURE ANNEALING EFFECTS

The same samples were subjected to high temperature annealing. This was performed in a furnace, at the temperatures 155, 207, 255 and 307 C, each for two hours. All samples annealed at the temperature T were subjected to all previous temperature annealing. The measurements were performed each time half an hour after the annealing finished. The samples subjected to the high temperature annealing were irradiated one and a half year before, and all this time kept at room temperature.

The results of resistivity and Hall coefficient measurements after annealing were published in [18], where a detailed analysis of both resistivity and Hall coefficient behavior after annealing are discussed. In order to analyze the consequences of the annealing on the resistivity and Hall coefficient, the range of fluences was divided in three regions: (a) fluences up to inversion (2.78×10^{12}, 3.98×10^{12} and 6.09×10^{12} n/cm^{2}) (b) fluences after inversion (5.69×10^{13} and 1.04×10^{14} n/cm^{2}) (c) the inversion region (1.04×10^{13} and 2.1×10^{13} n/cm^{2}). The resistivities and Hall coefficients after irradiation, measured for each group of fluences, as a function of the annealing temperature, is represented in fig.2 - 4.

In all the forthcoming discussion the results of resistivity measurements will decide on the behavior of carrier concentrations, while the Hall coefficient will be utilized more to confirm the conclusions. This happens so, because in the expression of the Hall coefficient enter, besides carrier concentrations, electron and hole Hall mobilities or the Hall factor for electrons and holes [19]. All the up-mentioned quantities could vary with the irradiation fluence. The Hall factor depends on the scattering mechanism of charge carriers [20]; 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.

In the fluence interval (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. We correlated the pronounced decrease of the resistivity between 150 and 200 C annealing temperatures, observed in Fig. 2a, with the annealing-out of the E center [13]. This is equivalent to the phosphorus release, 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 is in fact not observed. In this range of resistivities, the Fermi level is situated near the intrinsic level, so that the divacancy must be in the neutral charge state. The results of Hall coefficient measurements support this interpretation. So, in a sample where the hole concentration modifies (and, correlated, the electron one), without any other modification (of, example given, scattering mechanism), the dependence of the Hall coefficient on the hole concentration is represented in Fig. 5 (from [17]). The Hall coefficient corresponding to the n-type material is negative, at the increase of the hole concentration it decreases, reaches a minimum and then increases, changes sign, has a maximum and then decreases. For the fluences corresponding to group (a), the increase of hole concentration between room temperature and 150 C annealing could be seen in the Hall coefficient and understood as going to right in Fig. 5, to higher holes concentrations. The annealing-out of the V-P center determines the Hall coefficient values to go then towards left, to higher electron concentrations, what is really seen in the temperature interval 150-200 C.

In the high fluence case (b), the resistivity decreases from RT to 150 C, and increases between 150 and 200 C annealing temperatures, as is seen in Fig. 3a. After neutron irradiation in this fluence interval, the material becomes p-type. The decrease of the resistivity for annealing temperatures between RT and 150 C might correlated to acceptor creation, as in case (a) in the same temperature interval, and from 150 to 200 C - to the release of P from P-V center, and so to the increase of electron concentration. As in this case the electrons are minority carriers, the increase of their concentration increases the resistivity. For both neutron fluences of this interval, the Hall coefficient before the high temperature annealing is positive, corresponding to p-type material. For the 5x10¬13 n/cm¬2 fluence, a step decrease of the Hall coefficient between 150 and 200 C is observed, followed by a slow increase at high annealing temperatures. In this case, the material remains p-type (as was calculated, p>n), but the electron concentration increases, as was interpreted from resistivity measurements.

In the intermediate fluence domain, placed in the neighborhood of inversion, the behavior of the resistivity present similarities with both extreme fluence regions - see Fig. 4a. So, between RT and 150 C, the resistivity decreases, like in the case of high fluence irradiation. From 150 to 200 C, the resistivity decreases for 10¬13 n/cm¬2 irradiation, as for low fluences, but remains constant for 2x10¬13 n/cm¬2.

The annealing studies showed that the inversion of the conductivity type produced by irradiation remains even after the highest temperature annealing, of 300 C, that between RT and 150C annealing temperature donor removal and/or acceptor creation is the most important phenomenon, while in the interval 150-200C an increase of electron concentration is recorded, corresponding to phosphorus release from the E center.

DAMAGE CHARACTERIZATION BY TSC MEASUREMENTS

The TSC method could give information about the microscopical effects of the neutron irradiation in the silicon lattice, even at high fluences, in the order 10¬14 n/cm¬2, where the DLTS is no more useful [21]. As a continuation of the studies of last year [3], where this method was applied in the characterization of the disorder produced in the lattice by radiations at increasing fluences [14] and as a technological tool in the radiation damage research [22], the same technique was applied this time to characterize the spatial distribution of the defects, and in the study of the selfannealing at room temperature [23].

The samples used were p+n junction detectors, on high resistivity (2-6 KOhm*cm) silicon, made at the Brookhaven Laboratory, and irradiated with fast neutrons of average energy 1 MeV, and 5.67x10¬13 n/cm¬2 at the University of Lowell [14]. The TSC measurements have been carried out at the University of Florence [24]. The thermal scans were obtained by the variation of the height of the sample holder relative to the surface of the boiling helium. The temperature during the measurements was monitored with a Lake Shore temperature controller, connected to a silicon sensor. The current was measured with a Keithley 617 electrometer, all computer controlled.

SPATIAL DISTRIBUTION OF THE DEFECT CENTERS

The spatial distribution of the radiation induced defects was determined doing TSC analysis at different reverse voltages applied to the sample. As the time between irradiation and measurement was quite long (around 14 months), the strong room temperature reverse annealing [25] brought the occurrence of the full depletion at very high bias (far above 100 V), so, for the reverse voltages of 5, 20, 35, 50 and 70 V, the values of the depletion depths obtained from C-V measurements are: 22, 37, 50, 63 and 81 um respectively.

In Fig. 6, the TSC signals normalized to the depletion depth (I /w) are plotted as a function of temperature, in the temperature range 10 - 210K. An increase of the TSC signal is visible for high temperatures, for high w values, indicating an increase of the concentration of radiation induced traps related to the deepest energy levels.

The application of the deconvolution method [26] gave both the energy levels and their concentrations. The results are summarized in tables 1 and 2. Up to 17 energy levels were detected in the energy range 0.03-0.50 eV. In table 1, the values of the energy levels at different depletion depth are shown, while in table 2 the concentrations of known defects are represented. It could be observed that there are not significant changes of the detected energy levels for different biasing conditions of the sample. In what regards their concentrations, an important increase of the deepest levels, in the interval 0.4 - 0.5 eV was found for with the increase of the depletion depth from 22 to 81 um, while for shallower defects the concentration changes slightly with the depletion depth. The fact that the concentration of the deeper centers increases with the distance from the surface is in accordance with the theoretical predictions of the Lutz model [27].

ANNEALING EFFECTS ON THE TSC SPECTRUM

An important open problem (may be the most important) for the radiation hardness of silicon detectors is the reverse annealing of the effective impurity concentration, that could limit their use. In order to understand the nature of the centers producing the phenomenon, and their mechanism of formation and development, the most valuable tool could be the TSC study of the levels at after different time periods between the irradiation and the measurement, as well as by the modification of the annealing temperature.

In the paper [23], the results of the TSC analysis of the same sample, a silicon detector made from high resistivity silicon (2-6 KOhm*cm), irradiated with fast neutrons of fluence 5.6x10^{13} n/cm^{2}, after 2 and 14 months from the irradiation, were reported. The sample were kept at room temperature in the period between irradiation and measurement.

In Fig. 7, the two TSC spectra are presented on the same graph. It is evident that the major changes are observing for the peaks appearing at high temperature, and corresponding to an increase of the concentrations of the deepest levels with the selfannealing time.

ELECTRICAL MEASUREMENTS ON SILICON DETECTORS

Radiation damage studies on pad silicon detectors [28] with fast neutrons have been performed at two facilities: Sigma-Sigma-ITN at the IAP-Bucharest Magurele [29] and IBR-2 at the JINR-Laboratory for Neutron Physics in Dubna.

The neutron fluence varied from 5*10^{10} to 10^{15} n/cm^{2}. The silicon pad detectors used were diode structures on n-type Wacker Chemitronics FZ <111> silicon manufactured in standard planar technology by ELMA-Zelenograd-Russia and ST (SGS-Thomson), obtained by INFN Milano and Florence-Italy.

Electrical measurements C-V, I-V characteristics have been performed in the Laboratory of Particle Physics at JINR-Dubna and at Microelectronica SA-Bucharest.

Depletion voltage values as a function on the irradiation fluence confirm the type inversion at about 8*10^{12} n/cm^{2}.

Reverse current density at full depletion voltage has a linear dependence on fluence. The value of the damage constant, alpha, has been corrected for selfannealing with a two exponential law (with the parameters fitted on the data), and the extrapolated value at zero cooling time was found:

alpha(0) = (8.9+/-1.0)*10^{-17} A/cm for the detectors irradiated at the Sigma-Sigma facility

and

alpha(0) = (7.1+/-1.4)*10^{-17} A/cm for the detectors irradiated at the IBR-2 facility.

Figure 8 shows the energy spectrum dependence on alpha(0). Though the errors in all experiments are quite large, a linear dependence of the damage constant in respect with the hardness parameter is seen. The spread of the data is an indication of the systematic errors which affect such experiments.

We tried to explain the rather large value of the damage constant at the Sigma-Sigma facility by supplementary irradiation effects due to neutrons of low and intermediate energy nuclear reactions in aluminium and boron and from gamma prompt background. All these contributions have been found to contribute with at most 1% in the 0.4 eV - 20 KeV energy range. Then, the only explanation of this high high value can be the raise of the temperature during the irradiation time for high fluences (the temperature varies from 20 C to 35 C in the range of fluences used in the experiment).

CHARGE COLLECTION IN NUCLEAR RADIATION ENVIRONMENT

Pulse height amplitude (PHA) for alpha particle irradiation of detectors on both sides (n+,p+) have been measured using an ORTEC standard spectrometric chain. Measurements have been performed at the Laboratory of Nuclear Spectrometry of the Physics Department at Bucharest University.

Detectors have been irradiated with alpha particles of 5.5 MeV (241-Am) and 4.8 MeV (233-U).

The plot of the PHA ratio Q(p+)/Q(n+) versus bias voltage is given in figure 9, in comparison with the C-V characteristic for fluence values before inversion. The depletion voltage value is in agreement in both methods. Up to 10^{13} n/cm^{2} no defect in the charge collection measurements could be seen.

PHA measurements at higher fluences have not been performed, due to the high reverse current value.

In conclusion, further measurements are necessary in order to check the effect of the temperature raising during irradiation and the interesting effect of the "reverse annealing", which has not been put into evidence in our experiment.

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Figure captions

Fig.1: The resistivity (a) and the Hall coefficient (b) of silicon as a function of the neutron fluence.

Fig.2: The resistivity (a) and the Hall coefficient (b) of silicon as a function of the annealing temperature for fluences before inversion.

Fig.3: The resistivity (a) and the Hall coefficient (b) of silicon as a function of the annealing temperature for fluences after the inversion of the conductivity type.

Fig.4: The resistivity (a) and the Hall coefficient (b) of silicon as a function of the annealing temperature for fluences near inversion.

Fig.5: The dependence of the Hall coefficient on the carrier concentrations (from [17]).

Fig.6: The TSC signal normalized to the depletion depth, recorded in a detector irradiated with fast neutrons, 5.67×10^{13} n/cm^{2}.

Fig.7: The effect of room temperature annealing on the TSC spectrum, for the same detector as in Fig.5.

Fig.8: Energy spectrum dependence of alpha(0).

Fig.9: PHA ratio versus bias voltage and C-V characteristics for fluences before inversion.

Table captions

Table 1: Energy levels of the defects found by the TSC method, at different depths from the junction.

Table 2: Concentrations of the irradiation induced defects at different depths from the junction.

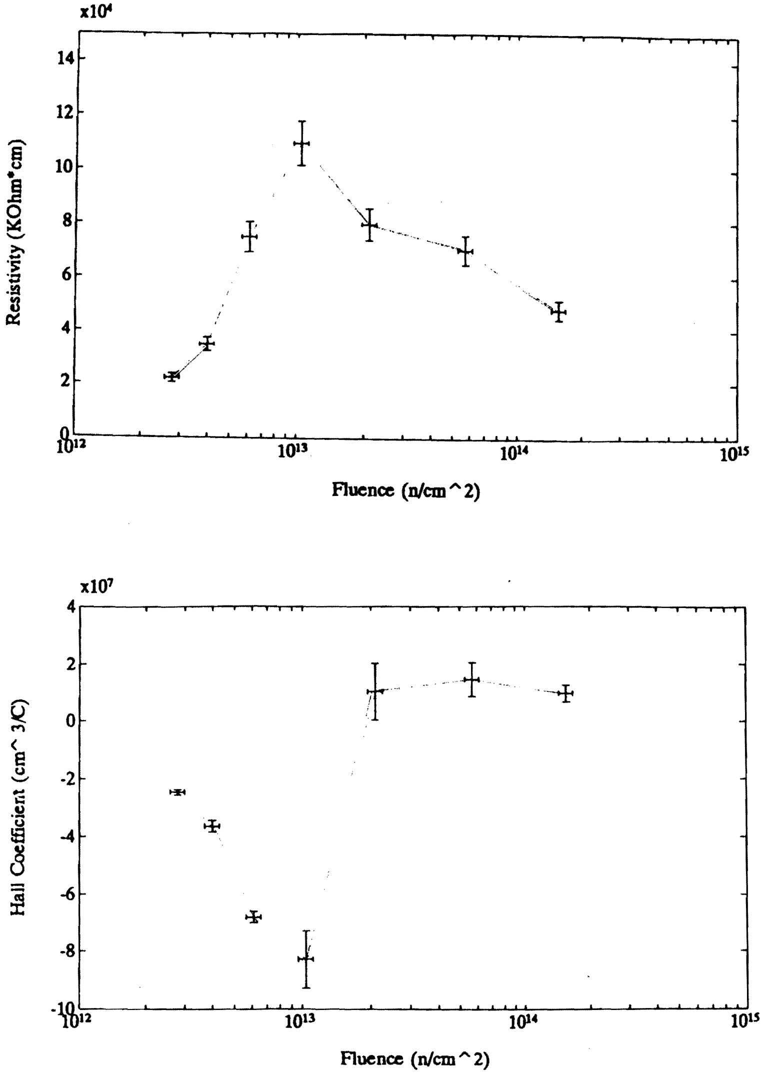


Figure 1

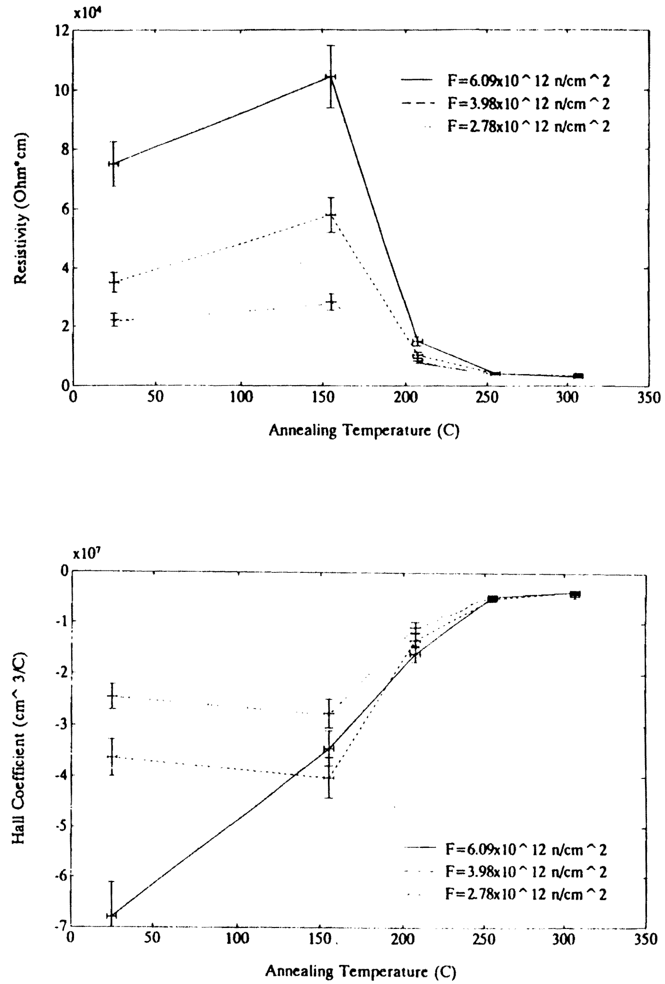


Figure 2

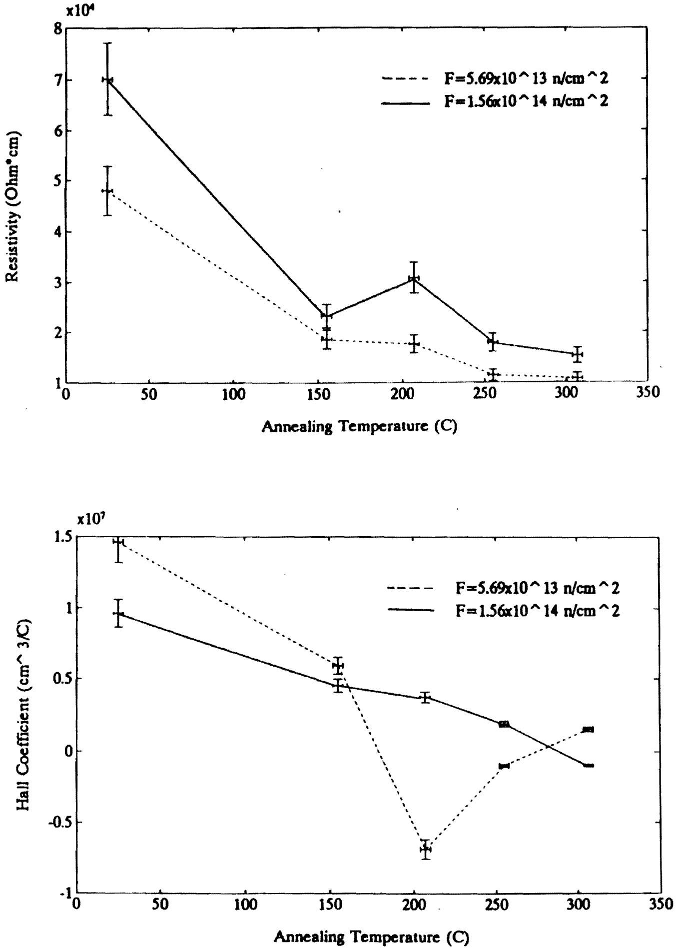


Figure 3

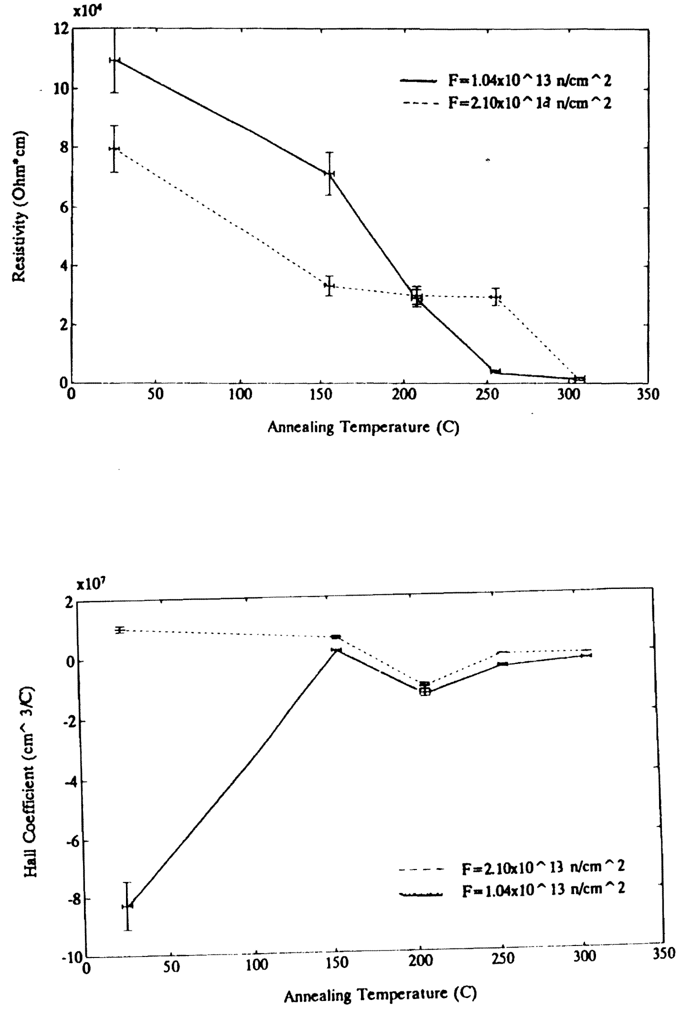


Figure 4

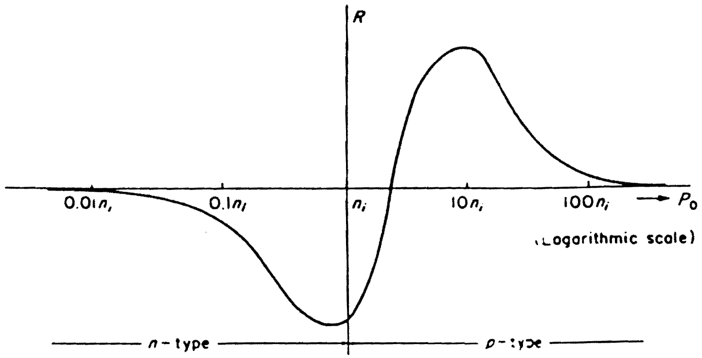


Figure 5

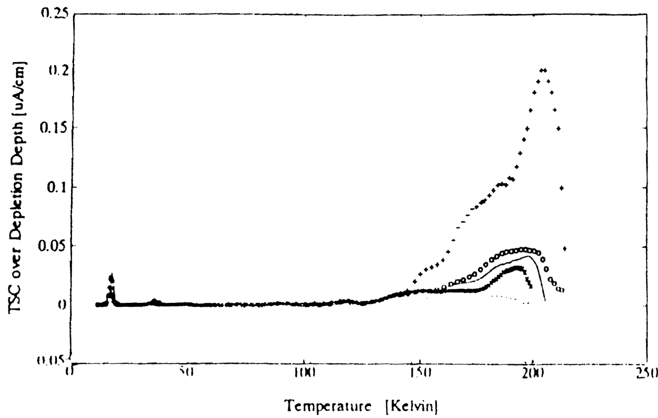


Figure 6

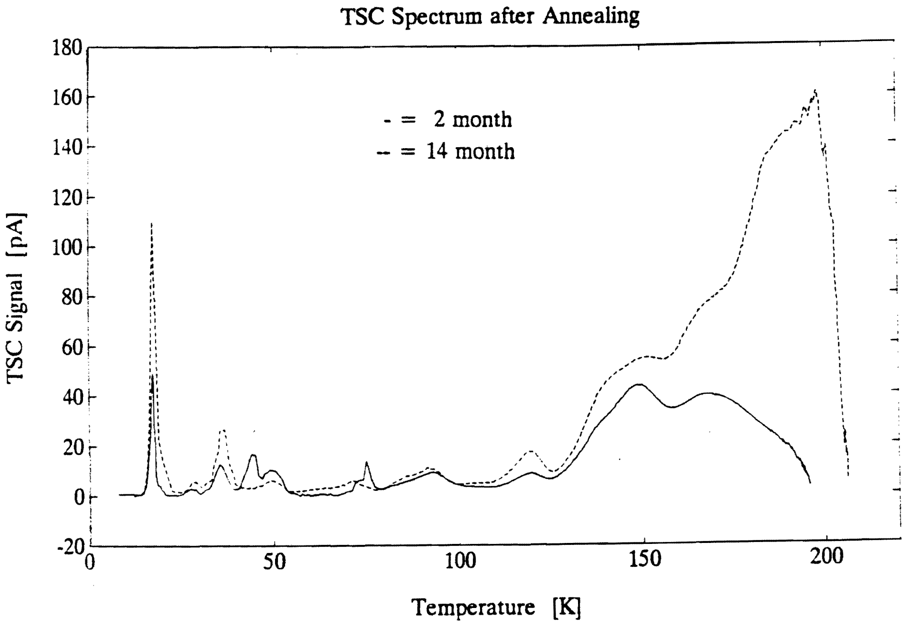


Figure 7

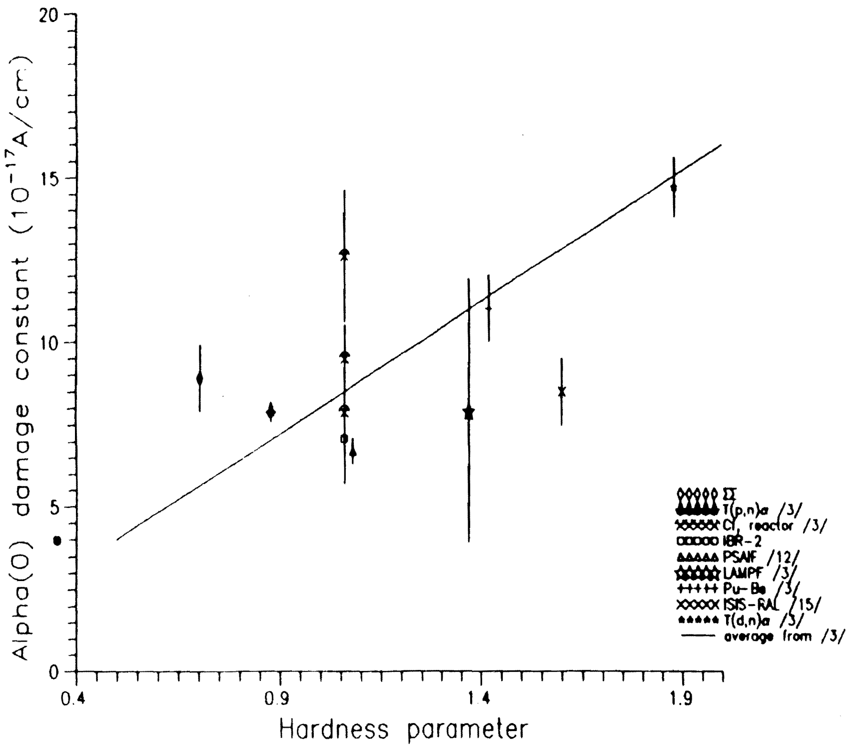
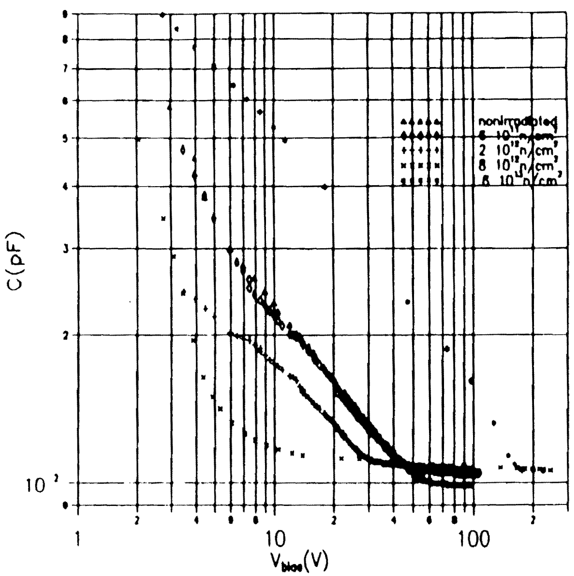
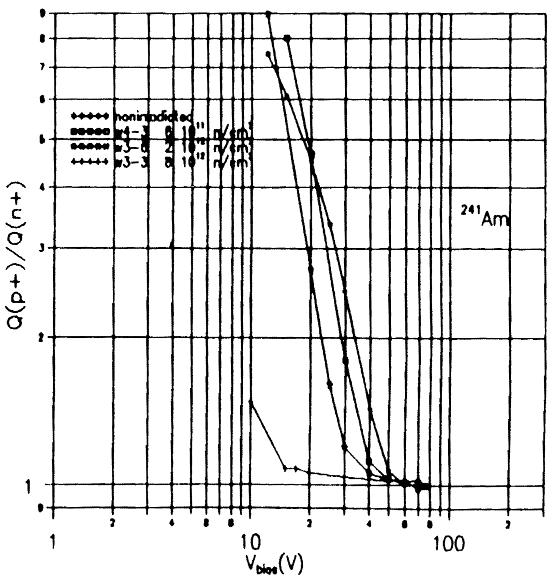


Figure 8



C(pF)
nonirradiated
8 10¹¹n/cm²
2 10¹²n/cm²
8 10¹²n/cm²
8 10¹³n/cm²
10
100
Vbias(V)



Q(p+)/Q(n+)
nonirradiated
w1-3 8 10¹¹ n/cm²
w3-6 2 10¹² n/cm²
w3-3 8 10¹² n/cm²
²⁴¹Am
10
100
Vbias(V)

Figure 9

Table 1

Depletion Depth [µm]				
22	37	50	63	81
Energy Levels [eV]				
0.031	0.031	0.030	0.030	0.029
0.035	0.035	0.034	0.034	0.034
0.058	0.053	0.054	0.054	0.054
0.072	0.069	0.070	0.070	0.070
0.102	0.098	0.099	0.099	0.099
0.168	0.164	0.160	0.165	0.165
0.188	0.182	0.185	0.182	0.182
0.200	0.200	0.203	0.203	0.203
0.210	0.215	0.215	0.215	0.215
0.250	0.250	0.247	0.247	0.247
0.308	0.308	0.308	0.308	0.308
0.337	0.334	0.334	0.334	0.334
0.362	0.358	0.364	0.360	0.364
0.395	0.395	0.407	0.395	0.402
0.423	0.425	0.430	0.430	0.432
0.450	0.440	0.457	0.465	0.453
0.485	0.480	0.490	•	0.500

Table 2

E_t [eV]	Depletion Depth [µm]				
	22	37	50	63	81
	Trap Concentration [10^{12} cm^{-3}]				
0.030	1.8	1.7	1.2	1.1	0.9
0.035	0.3	0.3	0.2	0.2	0.2
0.055	0.1	0.1	0.1	0.1	0.1
0.070	0.5	0.5	0.7	0.5	0.9
0.100	0.2	0.2	0.4	0.3	0.5
0.164	0.2	0.3	0.4	0.2	0.3
0.184	0.1	0.3	0.6	0.4	0.4
0.202	0.3	0.5	0.4	0.4	0.6
0.214	0.1	0.45	0.4	0.4	0.7
0.248	1.0	1.5	1.5	1.5	2.0
0.308	2.0	2.0	1.0	1.0	1.5
0.335	2.2	3.5	4.0	3.0	3.0
0.362	3.0	5.5	8.5	5.0	13.0
0.400	3.4	8.5	14.0	7.5	31.0
0.428	3.0	6.0	7.0	5.0	38.0
0.455	7.0	14.0	32.0	28.0	44.0
0.490	2.0	43.0	63.0	•	200.0