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# Conductive strontium titanate layers produced by boron-ion implantation

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The ion implantation of boron has been found to be an effective method for increasing the conductivity of strontium titanate. A highly conductive layer was formed by implantations at doses of  $3.6 \times 10^{16}$  to  $1.0 \times 10^{17}$  ions/cm<sup>2</sup>, using an accelerating voltage of 100 kV, corresponding to a boron range of about 300 nm. The conductivity of the implanted layer was found to be further enhanced by about four orders of magnitude after annealing at 225°C. The surface resistivity attained was typically 1000 Ω/square at room temperature. The resistance increased with rising temperature between 77 and 500 K. All samples were found to be *n* type, as determined by Hall-effect and thermoelectric measurements. The measured Hall mobility range was from 100 cm<sup>2</sup>/V sec at 77 K, decreasing to 5 cm<sup>2</sup>/V sec at 300 K. The existence of several defect energy levels can be inferred from the electrical data.

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## INTRODUCTION

Transition-metal compounds with the perovskite structure may be insulating or may possess significant conductivity, depending on the compound and its purity. Pure strontium titanate is an insulator, but extrinsic semiconductivity in strontium titanate has been known for many years. The compound also shows many ferroelectric characteristics at temperatures near absolute zero. Tufte and Chapman<sup>1</sup> measured the Hall mobility of *n*-type strontium titanate, made by hydrogen reduction or by niobium addition, and found that the mobility attained high values at low temperature. In later work semiconducting strontium titanate was found to be superconducting at temperatures below about 0.4 K. These facts, and interest in the energy levels and surface states on the crystal, have resulted in continuing work on the material. Extensive tabulations of its properties are given in the Landolt-Bornstein series.<sup>2,3</sup>

The present work reports on highly conductive surface layers made by boron-ion implantation of strontium titanate followed by annealing. Ion implantation is a widely used technique to introduce precisely controlled impurities into surface layers. At boron-ion energies of 100 keV (the energy used in these experiments) the range of the boron in strontium titanate is about 300 nm, a considerably greater range than is attained by implantation of ions more massive than boron. Some previous ion implantations in perovskites or related materials have been reported. A number of studies were concerned primarily with guiding sonic or optical waves at surfaces modified by ion implantation.<sup>4-7</sup> Parker and Kelly<sup>8</sup> investigated the ion implantation of rutile (TiO<sub>2</sub>) with Kr<sup>+</sup> and found conductive layers which they attributed primarily to stoichiometric change. Siskind *et al.*<sup>9</sup> have studied rutile implantation with H<sup>+</sup> and D<sup>+</sup>, and concluded that preferential sputtering was not the dominant mechanism in their experiments. Henrich *et al.*<sup>10</sup> have presented data on low-energy Ar-ion bombardment of strontium titanate, in

which they found evidence for Ti<sup>3+</sup>-O vacancy complexes. Finally Primak<sup>11</sup> has published reports of ion implantation in perovskites, among other insulators. The above studies did not include conductivity measurements as detailed as those given in this report.

A conducting layer is created in an insulator when an implantation results in shallow donors or acceptors within the band gap of the insulator. Inevitably the host crystal sustains considerable radiation damage during the implantation process, and an anneal is usually necessary after implantation. Annealing in silicon, for example, both removes radiation damage and results in some implanted species migrating to substitutional doping sites in the lattice.

In our work a number of ion species were implanted at the same energy and to the same dose level as were used in the experiments with boron ions, but these other ions almost invariably resulted in conductivities at least several orders of magnitude less than those found for boron. The other ions were H<sup>+</sup>, Ar<sup>+</sup>, C<sup>+</sup>, N<sup>+</sup>, Fe<sup>+</sup>, As<sup>+</sup>, Ta<sup>+</sup>, and Nb<sup>2+</sup>. All of these ions cause radiation damage, and those close to boron in mass, such as carbon, cause damage quantitatively comparable to boron. The markedly lower conductivity caused by almost all implants other than boron showed that the boron atom is important to conduction. The only other ion which we found to cause comparable conductivity to boron was niobium, an ion already well known as causing conductivity in strontium titanate. This report is solely concerned with boron implants.

The implantation of strontium titanate differs from that of silicon in several important ways. Because strontium titanate is initially insulating, the implantation of ions is accompanied by the appearance of strong electric fields around the insulator. These fields disappear, however, as the conductivity of the material increases because the bombarded surface is then connected to ground. Sputtering of surface atoms

occurs during ion bombardment and may not be a uniform process. Strontium, titanium, and oxygen atoms may leave the surface at different rates, resulting in high vacancy concentrations which differ among the elements. Vacancies may act alone or in combination with boron as donor or acceptor centers and as scattering centers limiting the mobility of charge carriers.

## EXPERIMENTAL

The strontium titanate samples used for implantation studies were cut from boules grown by the National Lead Company. The samples were cut into thin slices, typically 1 mm in thickness. The surface to be implanted was lapped and polished to a 0.5- $\mu\text{m}$  finish, then heated at 600 °C in air for 12 h and oven quenched. Boron ions were implanted through a mask having rectangular dimensions of 1 cm in length and 1.5 mm in width, and having four narrow side arms which were used as contacts for making Hall and conductivity measurements.

100-keV boron ions were used for implantation. Magnetic separation was used to establish a  $^{11}\text{B}$  beam. The accelerator was of conventional Cockroft-Walton design. Samples of different doses were prepared within a range from  $6 \times 10^{15}$  to  $1 \times 10^{17}$  ions/cm<sup>2</sup>. During implantation the substrate was at room temperature. The ion current was typically 0.5  $\mu\text{A}$ , and the beam was electrically rastered to improve the uniformity of the implant. As previously mentioned, strontium titanate is an insulator at room temperature, and a conducting path was provided from the implanted surface to ground, thus preventing the buildup of strong electric fields above the surface.

The implanted area was black in color. Spectral analysis at optical and ir frequencies showed no strong absorption peaks, indicating a very broad spectral range for the absorption. After implantation the samples were heat treated at 200 °C in a hydrogen atmosphere and then oven quenched. This anneal treatment reduced the measured resistivity of the implant region by about four orders of magnitude. Annealing in air, in vacuum, or in hydrogen produces the same results for annealing temperatures below 275 °C. Above that temperature the resistance of the layer increases rapidly for most of the implants we have studied. The optimum annealing temperature was not the same for all crystals, but a reduction in room-temperature surface resistivity by at least three orders of magnitude was found after annealing in all crystals implanted under conditions used in this paper. The annealing process probably involves some regrowth of the underlying crystal into the implanted layer, a process whose time constants seem to depend on the impurities present in the original SrTiO<sub>3</sub> boule. The purity of the boules is 99.95%.

Electrical contacts to the samples were made using conducting silver paste, painted onto either the sidearms or the ends of the implanted area. The conductivity (four-point method) and the Hall and Seebeck voltages were determined by dc techniques with a Keithley 616 electrometer. The high impedance of this instrument (above  $10^{14} \Omega$ ) was necessary

TABLE I. Properties of implanted samples at room temperature, after annealing.

Dose (ions/cm <sup>2</sup> )	Surface resistivity ( $\Omega$ /square)	Hall mobility (cm <sup>2</sup> /V sec)	Seebeck coeff. (mV/K)
$5.8 \times 10^{15}$	$1.3 \times 10^5$	...	40
$1.1 \times 10^{16}$	$2.9 \times 10^3$	11.4	120
$3.6 \times 10^{16}$	$5.0 \times 10^2$	5.2	300
$7.3 \times 10^{16}$	$4.4 \times 10^2$	5.1	180
$1.0 \times 10^{17}$	$5.9 \times 10^2$	5.6	60

because of the highly nonlinear contact resistances at the silver paste-conductor interface. All of the measurements were determined during the same run. The Hall voltage was found to be linear to a field of 1.5 T. The temperature dependence of the Hall coefficient was measured using a field of 0.9 T. The measurements were made at a pressure below  $10^{-3}$  Torr and at temperatures between 77 and 480 K.

The implanted surface was approximately a (155) plane. During implantation the sample was tilted at 7° to the beam to avoid channeling, and no evidence for channeling was seen. The conductive layers studied were at least ten orders of magnitude more conductive than the substrate, so that the electrical behavior of the implanted layer dominated the measurements. Two independent implantations and sets of measurements were done at each dose. In most cases the disagreement between the two measurements did not exceed 10%, which was also the level of disagreement between runs on the same sample. The variability was caused by thermal hysteresis, and tended to be reduced after several thermal cycles.

Because of the sputtering action of the ion beam during implantation, the exact chemical nature and phase of the implanted layer is not known. In most materials subjected to ion-bombardment doses as high as those used in this study, the bombarded layer becomes amorphous. HEED and SIMS are two surface tools which could be applied to the bombarded layers to help characterize them, but we have not made these measurements as yet.

## RESULTS AND DISCUSSION

Table I presents a summary of the values of resistivity, Hall coefficient, mobility, and Seebeck coefficient (with respect to copper) at room temperature as a function of ion dose. For all of the measurements, the signs of both the Hall and Seebeck voltages indicated *n*-type conductivity. In all calculations a homogeneous conducting layer of 300 nm thickness (calculated by the Lindhard-Scharff-Schiott method) was assumed. In fact, ion implantation does not produce a completely homogeneous layer. Thus the values presented here are really averages for the conducting volume of the sample.

As seen from Table I, the resistivity of the sample decreases, in general, with increasing ion dose. At the highest dose, however, the resistivity increased somewhat. The value of the mobility is relatively constant for the three highest

doses and is very close to that given by Tufte and Chapman<sup>1</sup> and by other authors<sup>12</sup> for reduced and for niobium-doped SrTiO<sub>3</sub>. On the other hand, the mobility of the sample with the implant concentration of  $1.1 \times 10^{16}/\text{cm}^2$  is about twice as high as those of the more heavily implanted samples. As will be seen below, this sample is quite different from the others in its behavior. The sample with the lowest implant dose ( $5.8 \times 10^{15}$  ions/cm<sup>2</sup>) did not have a measurable Hall effect.

The temperature dependence of the resistivity for all of the samples is given in Fig. 1. Several different types of behavior are evident.

(1) Lowest dose,  $5.8 \times 10^{15}$  ions/cm<sup>2</sup>. Below 400 K the resistivity decreased with increasing temperature, and above 400 K the reverse was true. This behavior is characteristic of an extrinsic semiconductor whose impurities are fully ionized above 400 K. Tufte and Chapman<sup>1</sup> obtained similar results for their compositions with low carrier concentrations.

(2) Intermediate dose,  $1.1 \times 10^{16}$  ions/cm<sup>2</sup>. At all temperatures the resistivity increased as the temperature rose. For this sample an approximately linear relationship between log resistivity and reciprocal temperature was found below 175 K, and a second linear relationship of higher slope was found above that temperature.

(3) Higher doses,  $3.6 \times 10^{16}$  to  $1.0 \times 10^{17}$  ions/cm<sup>2</sup>. At all temperatures the resistivity increased as the temperature rose. The slope of the log resistivity versus reciprocal temperature curve increases as the temperature rises. The curve corresponding to the highest implantation dose ( $1.0 \times 10^{17}$

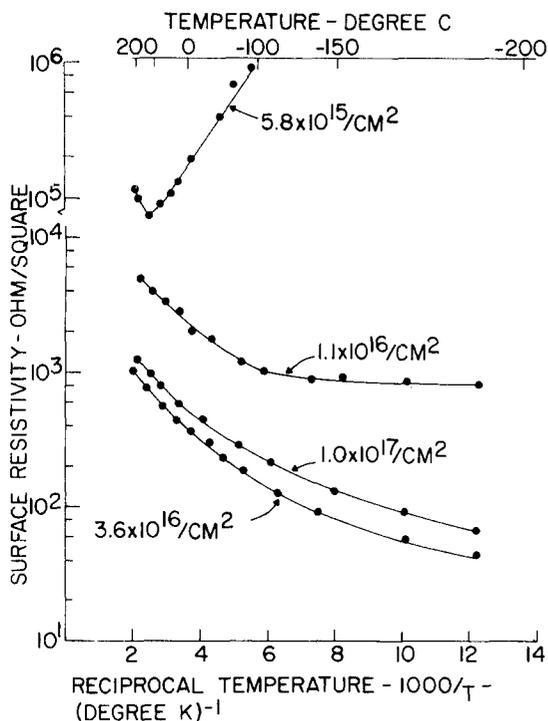


FIG. 1. Surface resistivity of boron-implanted strontium titanate as a function of temperature. The data for the implant of dose  $7.3 \times 10^{16}$  ions/cm<sup>2</sup> were so close to those for the dose  $3.6 \times 10^{16}$  ions/cm<sup>2</sup> that only the latter are shown on the drawing. The surface resistivity may be converted to a volume resistivity, in  $\Omega$  cm units, by multiplying by the layer thickness,  $3.0 \times 10^{-3}$  cm. Note that the vertical axis is broken between  $10^4$  and  $10^5$   $\Omega$ /square.

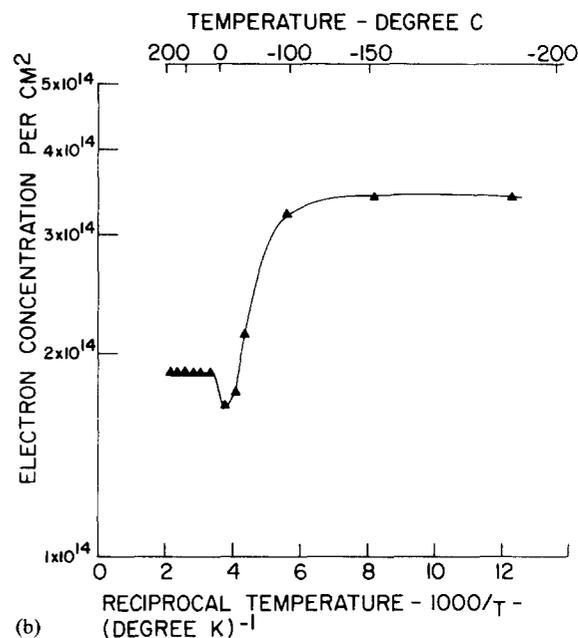
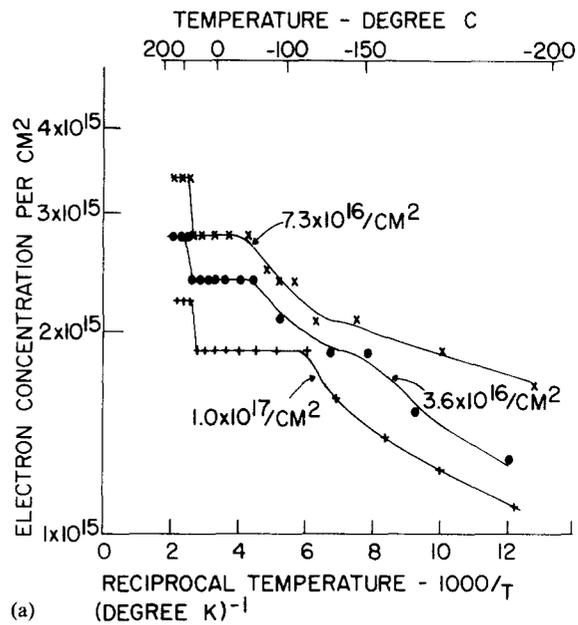


FIG. 2. (a) Electron concentration per unit area as a function of temperature for the three highest implant doses. The abrupt changes are due to ionization of new levels, while the temperature intervals of constant concentration are due to complete ionization. (b) Same data for the dose  $1.1 \times 10^{16}$  ions/cm<sup>2</sup>. The decrease in electron concentration with rising temperature between  $-150$  and  $0^\circ\text{C}$  indicates compensation. All concentrations/unit area may be converted to concentrations/unit volume by dividing by the layer thickness,  $3.0 \times 10^{-3}$  cm.

ions/cm<sup>2</sup>) is higher in resistivity than the other two, so that there is a broad minimum in resistivity at a dose between  $3 \times 10^{16}$  and  $7 \times 10^{16}$  ions/cm<sup>2</sup>. The interpretation of these data is given below.

The data on carrier concentration as a function of temperature, calculated from Hall data for all of the samples showing a Hall effect, are given in Fig. 2. The carrier concentration and mobility were calculated by well-known methods, using a one-carrier model. Even at the highest temperatures, the electron concentration is less than 1% of the

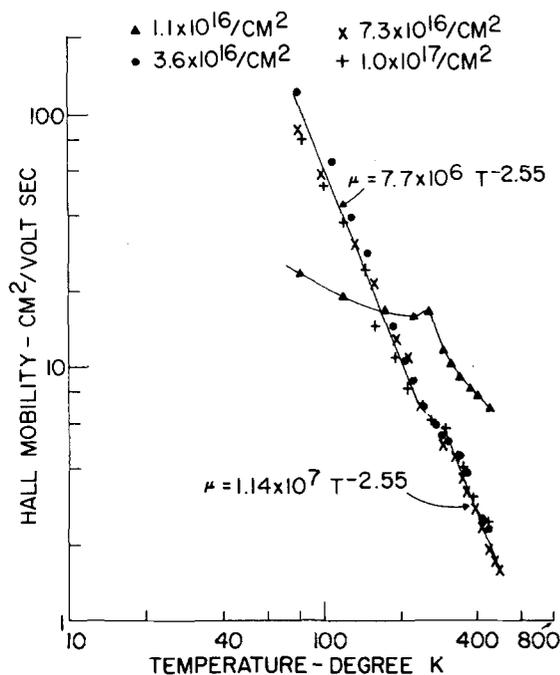


FIG. 3. Electron Hall mobility,  $\mu$ , as a function of temperature for annealed implants. The low-temperature data for the dose  $1.1 \times 10^{16}$  ions/cm<sup>2</sup> probably do not represent the drift mobility.

implanted-ion concentration. The samples with higher implant doses all show similar behavior. An electronic conductivity is seen with the electron concentration increasing as the temperature rises. The stepwise increase (Fig. 2) suggests at least two important donor levels, one ionizing below 77 K and the other at about 360 K. A qualitative difference between the sample with dose  $1.1 \times 10^{16}$  ions/cm<sup>2</sup> (the lowest dose for which the Hall effect could be measured) and the others is apparent. Over most of the measured temperature range the carrier concentration for the low-dose sample is either temperature independent or the carrier concentration decreases, indicating significant compensation as the temperature rises. It is noteworthy that the major breaks in the conductivity temperature relationship and in the carrier-concentration-temperature relationship occur at about the same temperature, 170 K. It appears that there is an important change in the conductivity mechanism for this sample at 170 K.

Figure 3 gives the mobility, calculated from the Hall effect data and conductivity on the basis of electronic conduction only, for samples showing a measurable Hall effect. The values of the mobility are high enough to assure that the conduction phenomena observed in this work are not due to hopping processes or ionic conductivity. For implantation doses of  $3.6 \times 10^{16}$ /cm<sup>2</sup> or above, the mobility data are very similar to the bulk data of Tuft and Chapman.<sup>1</sup> These authors found that the mobility showed a  $T^{-2.7}$  behavior, while in our case we find  $T^{-2.55}$ , where  $T$  represents absolute temperature. Our measured mobilities are about a factor of 2 below those of Tuft and Chapman.

The  $1.1 \times 10^{16}$  ions/cm<sup>2</sup> sample behaves like none of the others. Its mobility at room temperature and above is higher than those of the others, but below room temperature the

mobility rises comparatively little as the temperature drops. At liquid-nitrogen temperature the Hall mobility still has the comparatively low value of 23 cm<sup>2</sup>/V sec. We believe that the low calculated Hall mobility is not really the correct drift mobility. In this sample we believe that  $p$ -type conduction is important at lower temperatures, and that the mobility cannot be calculated on the simple model we have applied. Evidence for this conclusion is seen below in the Seebeck data.

The data on the Seebeck coefficient of the samples are given in Fig. 4. This coefficient is a very sensitive indicator of small changes in the Fermi level as the temperature varies. The rather complex variation of the Seebeck coefficient of all of the samples above 200 K again indicates a number of ionization energies for donor and acceptor centers. Small Fermi-level changes may affect the Seebeck coefficient substantially, but could result in relatively minor carrier-concentration changes. Tuft and Chapman<sup>1</sup> postulated a high degree of compensation of donor and acceptor centers in their bulk samples as an explanation for their experimental mobility values, which were well below the values predicted by theory. These authors found that their samples with niobium impurity had mobilities about an order of magnitude higher than hydrogen-reduced samples at low temperatures, although at higher temperatures the two semiconductors had comparable mobilities. As previously mentioned, the electron mobility in most of our surface layers was lower than that of Tuft and Chapman, leading to the conclusion that lattice imperfections were still abundant in our materials, even after annealing.

If an extrinsic semiconductor has only one donor level, the conductivity will decrease if that level becomes deeper, and the Seebeck coefficient will increase. Considering the

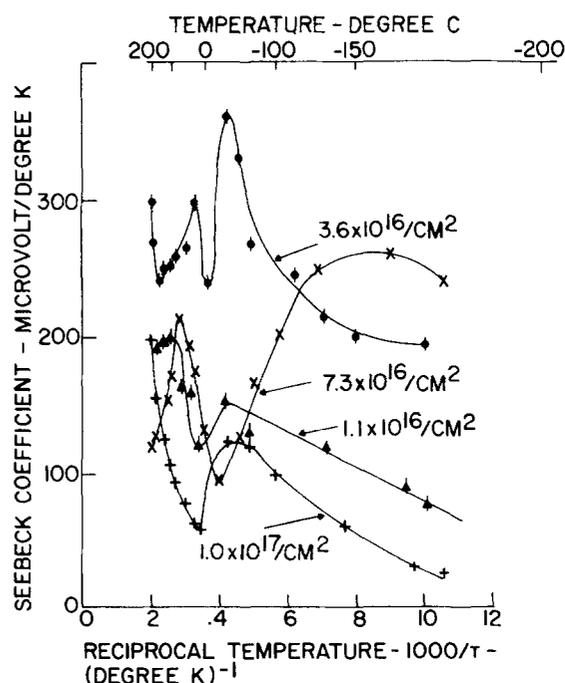


FIG. 4. Seebeck coefficient as a function of temperature for annealed implants. The complex behavior indicates several energy levels.

sample with  $1.1 \times 10^{16}$  ions/cm<sup>2</sup>, we find that the Seebeck coefficient at low temperatures is very low compared with other samples of much higher conductivity. The simplest explanation of this phenomenon is that compensation is particularly high in the sample in question ( $1.1 \times 10^{16}$  ions/cm<sup>2</sup>). It may be that the hole concentration at low  $T$  is very appreciable with respect to the electron concentration, or that the hole mobility is high at low  $T$ , leading to the observed low values for the Seebeck coefficient and reducing the Hall field considerably. It is probable that the carrier concentration and mobility calculated for this sample on the basis of a one-carrier model are inaccurate. It should be pointed out that the surface layer of a compound which undergoes ferroelectric transitions is a very complex system.<sup>13</sup> We did not find in this work conductivity effects which could be associated with the low-temperature ferroelectricity in SrTiO<sub>3</sub>.

The data for the more highly doped samples appear reasonable compared to earlier work. The assumption of one dominant carrier type at low temperatures for those samples is probably accurate, although the assumption fails to explain the Seebeck data above 200 K. The complexity of these data is probably due to the high sensitivity of the Seebeck coefficient to relatively minor changes in the carrier concentrations, particularly when the latter occur over a small temperature range.

The data of Fig. 2 permit the evaluation of the Fermi energy of the semiconducting layers using well-known methods.<sup>14</sup> Again the assumption is made that there is one type of charge carrier, and, following the results of Frederikse and Candela<sup>15</sup> for SrTiO<sub>3</sub>, we shall assume the electron effective mass is  $6m_e$ , where  $m_e$  is the free-electron mass. A plot of the Fermi energy as a function of temperature for the layer implanted with  $3.6 \times 10^{16}$  ions/cm<sup>2</sup> is given in Fig. 5.

The data show that the Fermi energy at liquid-nitrogen temperature is within a few millivolts of the conduction band, and that this interval has increased to about 50 mV at room temperature. This sort of behavior is characteristic of a semiconductor with a high concentration of impurities having an activation energy of a few millivolts. Above about 300 K, Fig. 5 shows that the Fermi energy does not drop as rapidly with temperature as would be predicted by extrapolation of the lower-temperature data. This is due to ionization of another donor level with a higher activation energy. The charge carriers due to this donor level appear at 360 K on all of the carrier-concentration-temperature curves (Fig. 2).

In summary, a conducting layer can be made on insulating strontium titanate by ion implantation. Boron is an exceptionally effective implant ion for this purpose. Electrical measurements show that several defect levels are present in this layer. Our measurements do not show whether boron itself is a donor or whether it is associated with other defects in the layer. It seems reasonable from the data presented above (Figs. 2 and 5) that the measured conductivity of the boron-implanted layers in the implantation range between  $3.7 \times 10^{16}$  and  $1.0 \times 10^{17}$  ions/cm<sup>2</sup> is associated primarily with one type of donor center between room temperature and 77 K. At higher temperatures another type of donor

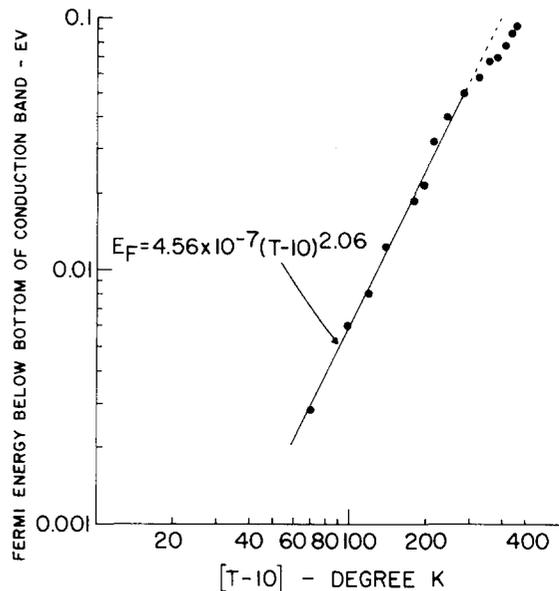


FIG. 5. Fermi energy as a function of temperature for an annealed boron implant of dose  $3.6 \times 10^{16}$  ions/cm<sup>2</sup>. The Fermi level drops lower into the energy gap as the temperature rises. This dominant low-temperature behavior is that of a shallow donor level.

center is ionized. For samples with low implant doses ( $1.1 \times 10^{16}$  ions/cm<sup>2</sup>)  $p$ -type conduction significantly modifies the dominant  $n$ -type conduction [Figs. 2(b) and 3]. The conductivity associated with boron implantation decreases permanently for most samples heated above 275 °C. This change may be due to migration of the boron or to a stoichiometric change in the implanted layer.

Finally we point out that this is an example of a system prepared by ion implantation which probably could not be duplicated by other methods, such as diffusion or conventional chemical reaction.

## ACKNOWLEDGMENT

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