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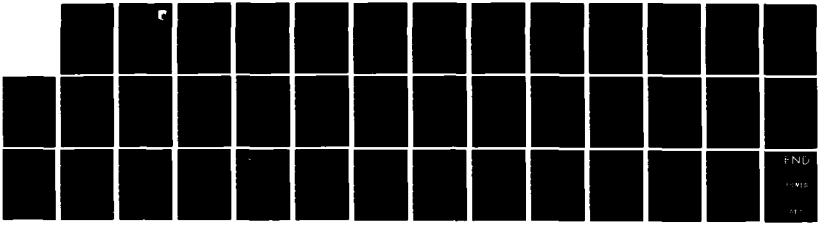

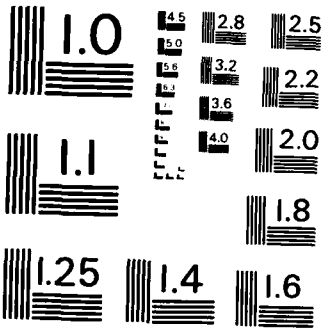

AFWAL-TR-85-4048

ELECTRICAL PROPERTIES OF NITROGEN DOPED FLOAT ZONE SILICON

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Laser and Optical Materials Branch
Electromagnetic Materials Division

July 1985

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Examination of nitrogen doped float zone silicon indicates that electrical properties are similar to commercial Czochralski material. This material deserves further consideration for use in high power device and intrinsic infrared detector applications. Temperature dependent Hall effect measurements have been made on as received and neutron transmutation doped (NTD) sample of a nitrogen doped float zone silicon crystal to determine its electrical properties. Samples were studied in both as-received state and after various high tempera-

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ture anneals. Results were compared with commercial n-type Czochralski silicon and conventional neutron doped float zone silicon. Undoped, annealed samples of Si:N showed signs of inhomogeneities believed to be related to precipitation of nitrogen related complexes. These inhomogeneities were not seen in lightly NTD's and annealed nitrogen doped material, indicating that even light doping will mask the effects of the proposed precipitation. No evidence was detected for any electrically active level that could be directly related to the nitrogen.

FOREWORD

This report describes an in-house effort conducted by the personnel of the Department of Electrical Engineering of the Air Force Institute of Technology (AFIT), and of the Laser and Optical Materials Division (MLP), WPAFB, Ohio. It was performed under Program Element 661102F, Project 2306, Task 2306Q1, Work Unit 2306Q106.

The work was performed during the period March 1984 to March 1985 under the direction of Mary A. O'Leary of AFIT and W. C. Mitchel of AFWAL/MLPO.

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SECTION I

INTRODUCTION

Nitrogen doping of float zone silicon has recently received new interest as this dopant has been proposed as an alternative to oxygen for eliminating the adverse effects of processing of high purity float zone silicon. Oxygen is used in Czochralski grown silicon, which is the dominant material for integrated circuit applications, as a hardening agent and for internal gettering of deleterious impurities such as iron. It has been reported that nitrogen doping prevents slip and warpage of wafers during high temperature processing (Reference 1) thus making float zoned (FZ) material easier to process. This material should thus improve the yield and performance of power devices, traditionally fabricated from FZ material because of its superior uniformity and purity. Another possible application is for intrinsic infrared detectors, where purity is most important. Fortunately, it is a well established fact that nitrogen does not behave as other group V elements when incorporated into silicon crystals (References 2-5). All other group V elements have rather high solid solubilities in silicon and enter the lattice substitutionally, producing shallow donor states in the band gap, thus the prevalence of these elements as n-type dopants. Nitrogen, however, has a very low solubility and produces a plenitude of levels in the gap, if all levels attributed in the literature to nitrogen are to be believed (References 2-6). It does seem certain that nitrogen does produce donor states and not acceptor states, but the position of the donor states in the gap and the atomic configurations that produce the states are still a matter of some controversy, particularly whether the states are very shallow or deep (References 7-13). The method of introducing the nitrogen does appear to play a strong role in determining the nature of the electrically active levels; doping from the melt with either nitrogen gas or solid silicon nitride and ion implantation have all been reported. The Materials Laboratory has obtained a boule of nitrogen doped float zoned silicon for examination from Shin-Etsu Handotai Co. through Dr. T. Abe. The boule is typical of such material grown by Shin-Etsu and nitrogen doping was accomplished by adding silicon nitride to the poly-silicon charge before the final pass. Other than the nitrogen, the crystal had no intentional dopants. In this technical report we present the results of a study of the electrical properties of this crystal and compare these properties with commercially supplied n-type CZ crystals.

We have had a portion of the Si:N boule irradiated for neutron transmutation
g (NTD) to increase the conductivity. In this process silicon is placed in the
on flux of a reactor. Some of the thermal neutrons are absorbed by silicon
and the resulting nuclear reaction results in the emission of a beta particle
he transmutation of the silicon atom into a phosphorus atom, the desired dopant.
technique has several advantages over doping during crystal growth, the most
tant of which is the extreme uniformity of the doping due to the small cross
on for absorption of thermal neutrons by silicon. The irradiated material does
to be thermally annealed at around 800°C to remove lattice damage introduced by
ransmutation process and by the high energy neutrons that are always present in
eactor flux. We have compared the electrical properties of NTD Si:N with NTD CZ
con.

We describe our experimental procedures in Section II of this report. Included
his section is a description of a modification of the standard high impedance
effect system used in our laboratory for use in examining the electrical
erties of n or p-type thin layers on opposite type substrates. This modification
not used in this experiment but is included for future use, particularly for
be implants into undoped Si:N wafers. The results of standard Hall effect
riments on various doped samples are presented in the next section. The results
discussed, and conclusions and recommendations are made in the final section.

SECTION II

EXPERIMENTAL DETAILS

Samples were fabricated from the nitrogen doped, float zoned crystal, from an ıdoped float zoned crystal and from two commercial Czochralski crystals, one lightly ɔped and the other moderately doped, both with phosphorus. The as-received ıaracteristics of the crystals are given in Table 1. Segments of the Si:N crystal, ıe lightly doped Cz crystal and the undoped FZ crystal were neutron transmutation ɔped (NTD). The irradiations were performed at the University of Missouri Research ɛactor (MURR), a light water reactor with a relatively high flux of thermal ɔutrons. The amount of phosphorus added to the nitrogen doped and the Cz crystals ıs $1x10^{14}$ cm^{-3}. That added to the undoped FZ crystal was $1.2x10^{14}$ cm^{-3} and $3x10^{13}$ $)^{-3}$.

TABLE 1

MATERIAL PROPERTIES

Supplier	Growth Technique	Dopant	Type	Resistivity Ω-cm
Shin-Etsu Handotai	FZ	Nitrogen	n	10^3
Hughes Aircraft	FZ	residual Boron	p	10^5
Monsanto	CZ	Phosphorus	n	200
Monsanto	CZ	Phosphorus	n	2.0

Several 3/8" x 3/8" samples were cut from various wafers of both as-received aterial and NTD'd material for use in the Hall effect experiments. After cutting, ll samples were cleaned. This procedure consisted of rinses in soapy water, warm richloroethane, warm acetone and warm alcohol. The rinses were followed by the tandard NH_4OH-H_2O_2 and HCl-H_2O_2 washes and a final rinse in distilled water. everal samples, including all the NTD'd samples, then received various anneals. nnealing was performed in a small tube furnace with a high purity polycrystalline ilicon furnace tube. The samples were placed between two high purity silicon wafers nd on a poly-silicon platform to prevent inadvertent contamination during annealing.

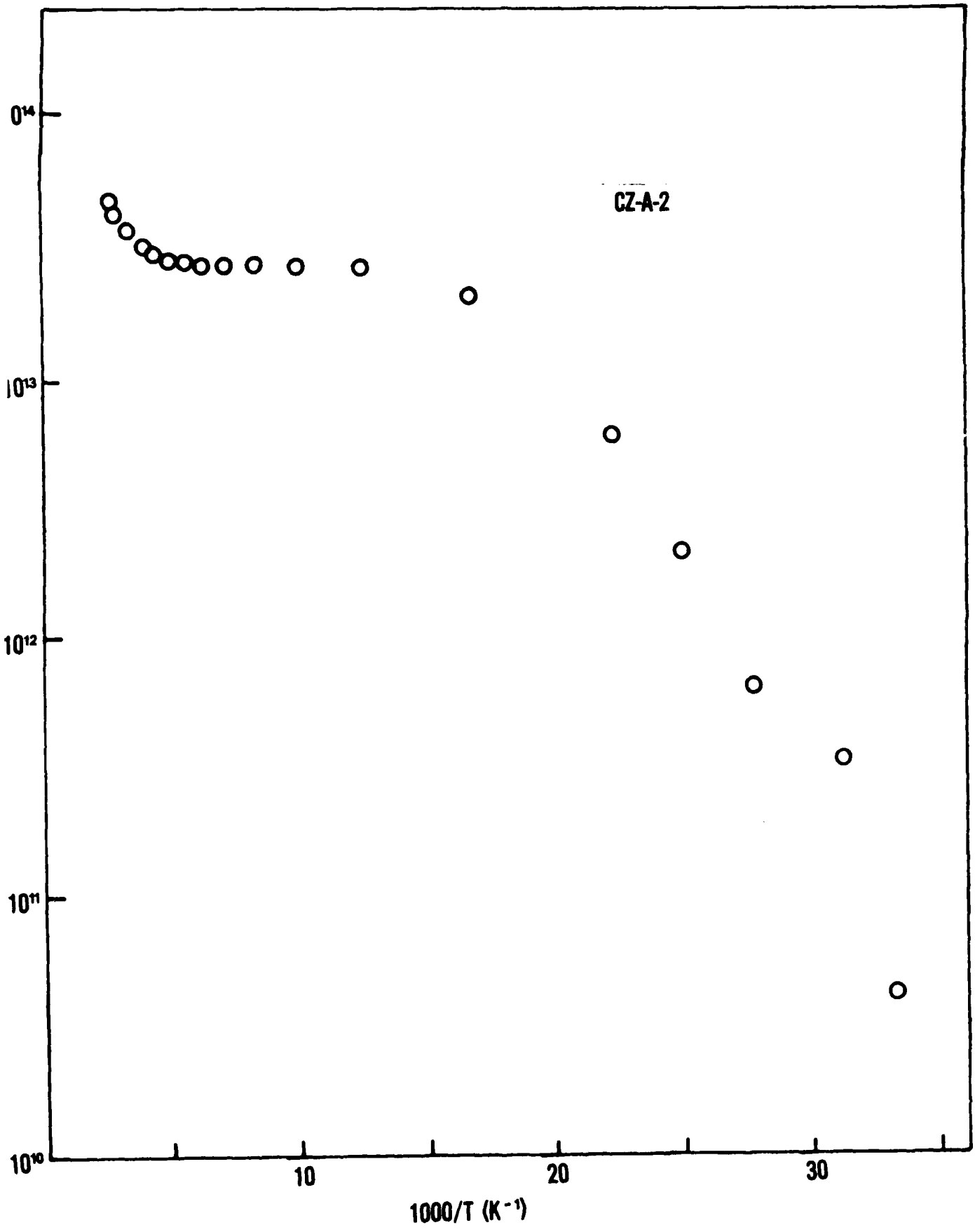

Figure 10. Carrier Concentration vs. Inverse Temperature for Annealed, High Resistivity Commercial Czochralski Sample CZ-A, Annealed at 800°C for One Hour

ple, shown in Figure 10, but not in the lower resistivity CZ sample, shown in ure 11. This shoulder is most likely due to the presence of deep donor levels at concentrations, so they are overshadowed by the phosphorus in the more heavily ed sample. It was at first thought that these deep levels might have been roduced during annealing as contaminants so two samples of neutron transmutation ed float zone silicon without nitrogen doping were run for comparison. The ults are shown in Figures 12 and 13. There is only the slightest trace of a ulder in one of these and no shoulder at all in the other, so contamination, while completely ruled out, is unlikely and the effect in the Si:N and CZ samples is bably a real effect of the material, although not necessarily due to the same se in both types of material.

The settling time problem was significantly reduced in all cases by annealing several other disturbing effects developed after annealing in some samples. haps the most damaging was a serious degradation in the van der Pauw ratio, R_1/R_2. ratios tended to show strong temperature dependences after annealing. A typical nple is given in Figure 14, where the ratio R_1/R_2 of the annealed sample as pared with that for the same sample before annealing. The carrier concentrations resistivities for the samples with large fluctuations in the ratio showed alous effects as well. A plot of n vs. 1/T for two of these samples is shown in ure 15. The anomalous behavior appears in the same temperature region for both les and might be thought to be due to some intrinsic effect in the material, but one examines the mobility (Figure 16) the anomalies are no longer visible. The alous structure in the carrier concentration is reflected in the resistivity ts and since they cancel out in the mobility, it is likely that these effects are to strong inhomogeneities in the samples induced by annealing. These samples not analyzed further for this reason.

NEUTRON TRANSMUTATION DOPED SAMPLES

Samples from the portions of the two boules that were neutron transmutation d (NTD) were annealed and analyzed. The results for a nitrogen doped NTD sample shown in Figures 17 and 18. The results for the CZ sample are given in Figures nd 20. The two samples have almost identical properties, as would be expected two boules that were irradiated together. The carrier concentrations are almost tical as are the mobilities, which are both indicative of high quality material. van der Pauw resistance ratio did show a degradation below about 35K, but above temperature, it was very nearly constant. Low temperature degradation of the ratus and not the sample.

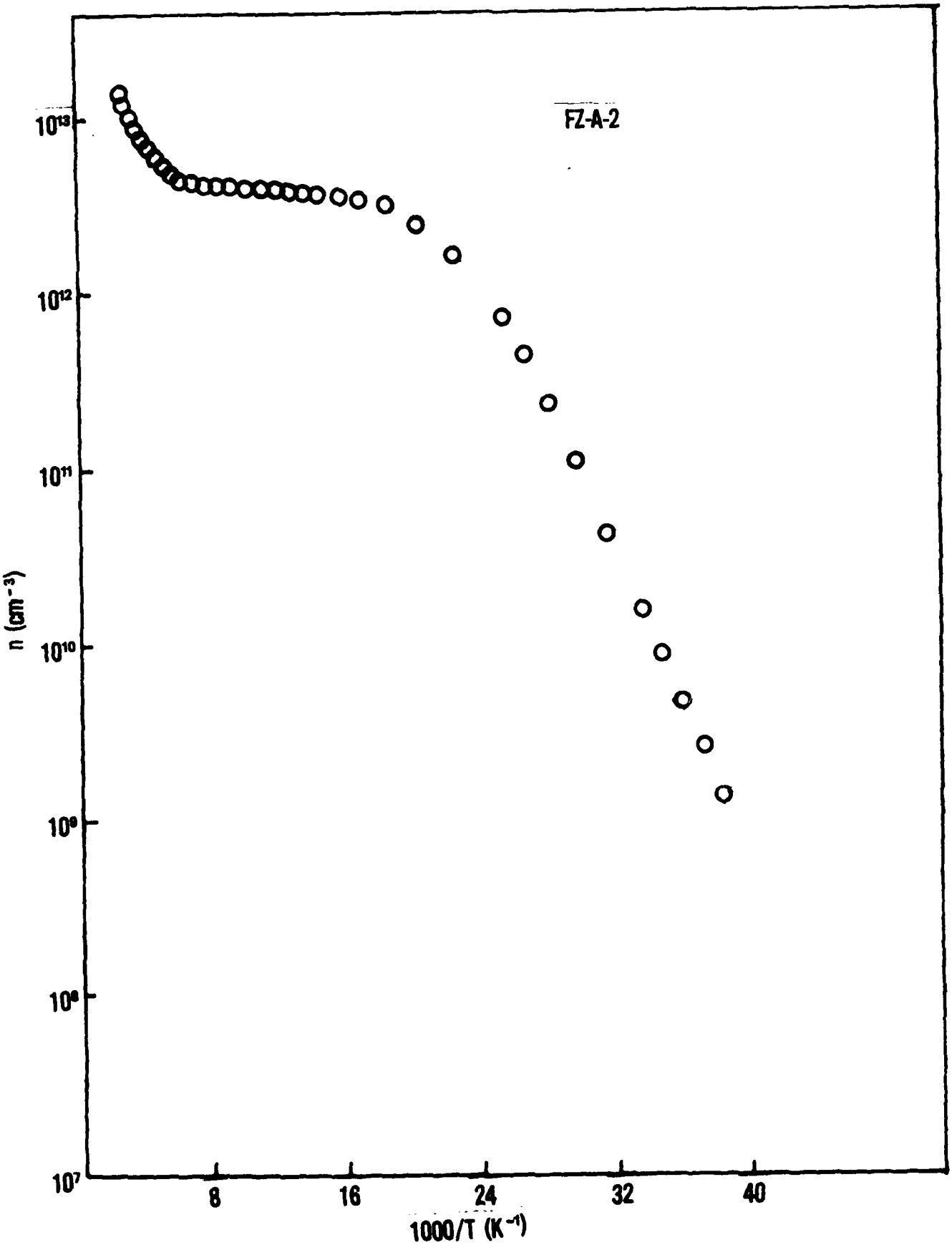

Figure 9. Carrier Concentration vs. Inverse Temperature for Annealed Si:N Sample FZ-A, Annealed at 900°C for 0 . Hour

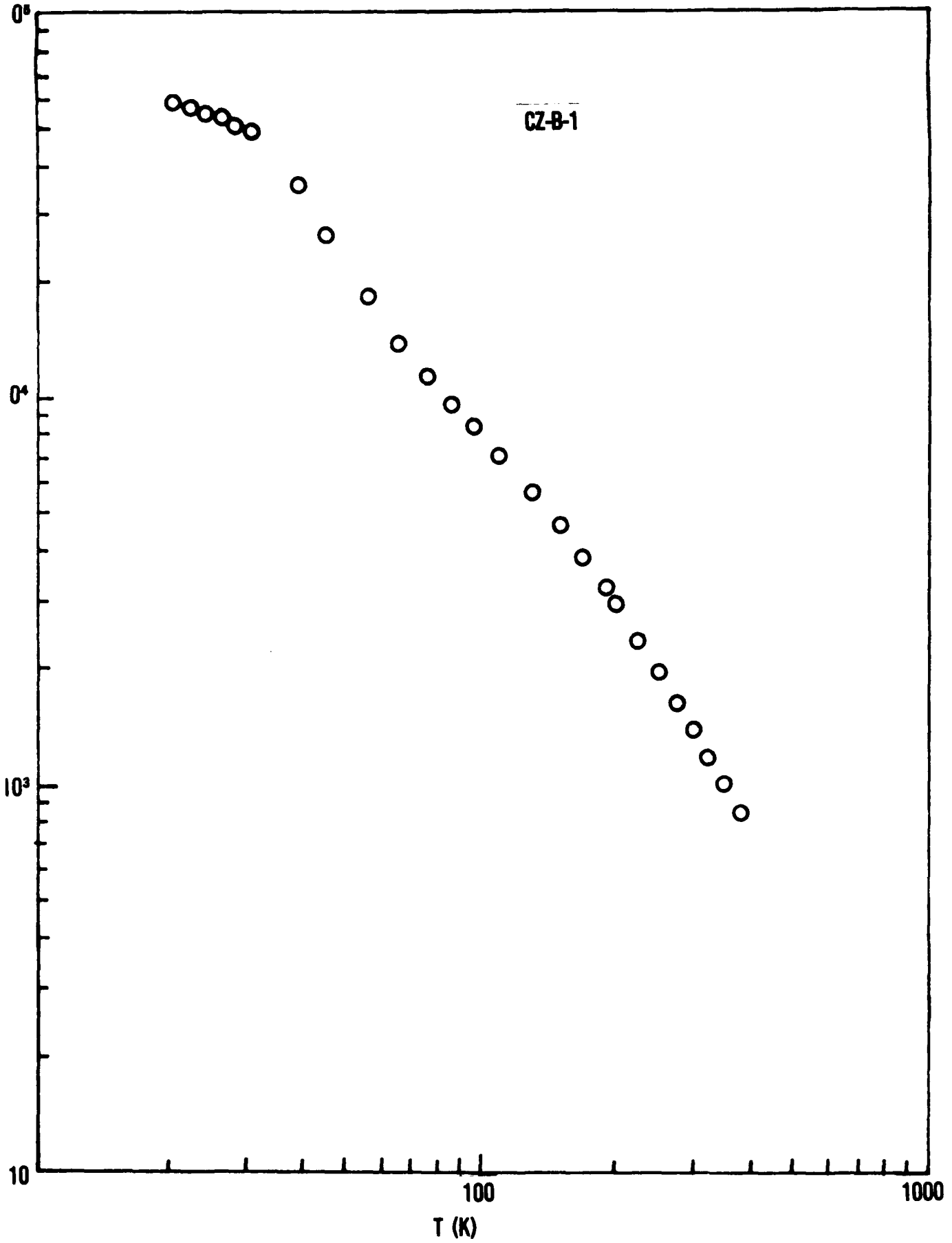

Figure 8. Mobility vs. Temperature for Low Resistivity Commercial Czochralski Sample CZ-B

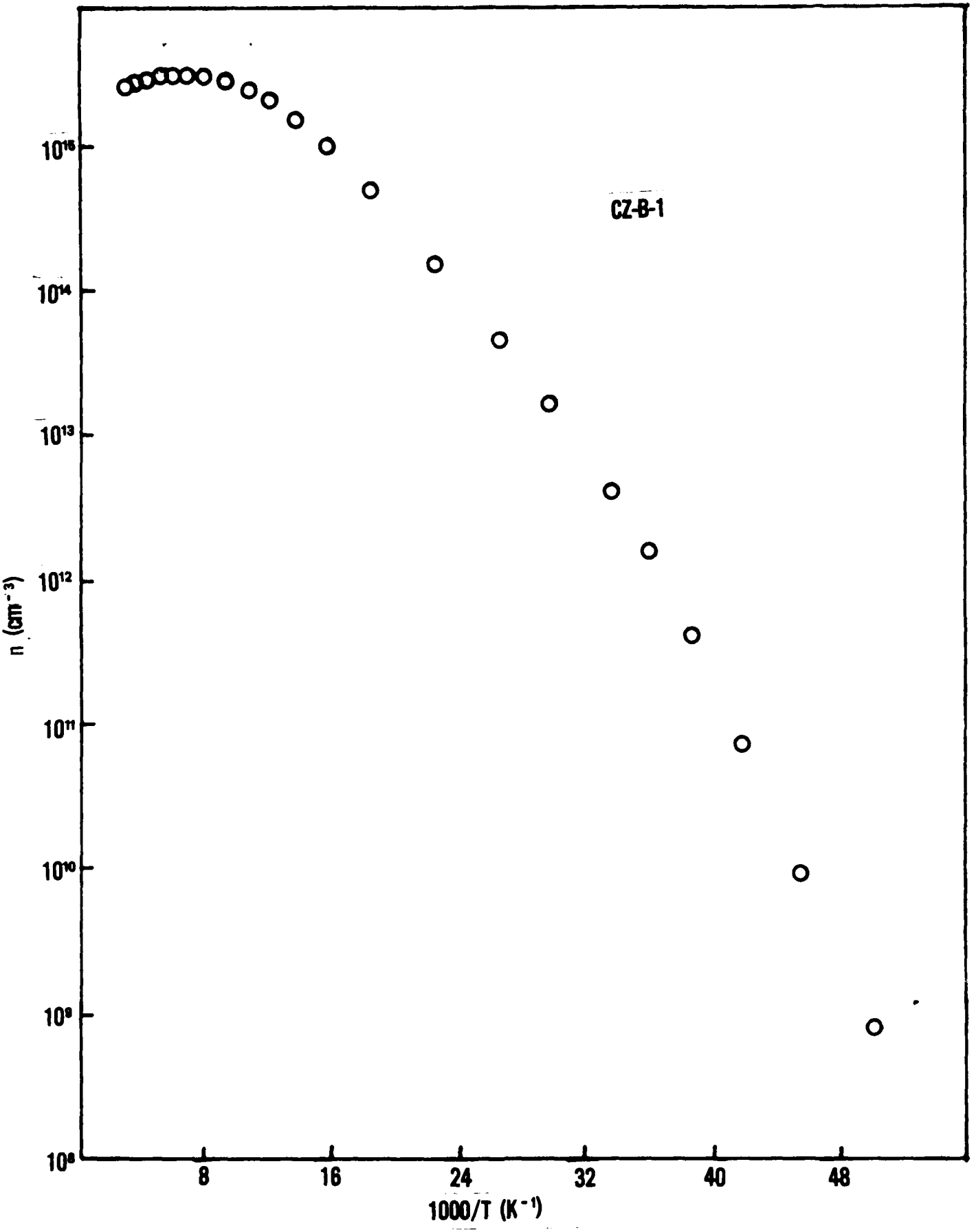

Figure 7. Carrier Concentration vs. Inverse Temperature for Low Resistivity Commercial Czochralski Sample CZ-B

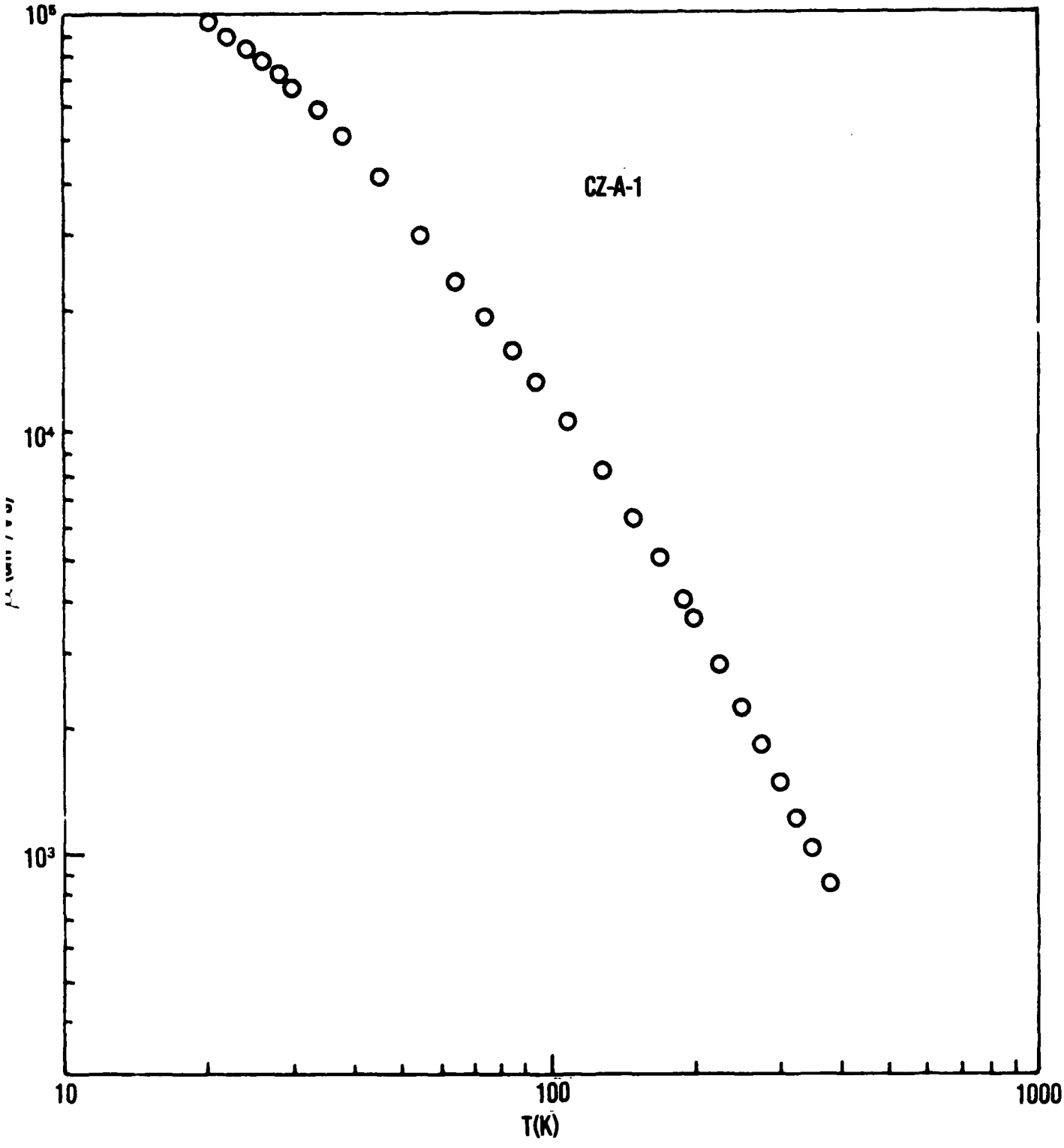

Figure 6. Mobility vs. Temperature for High Resistivity Commercial Czochralski Sample CZ-A

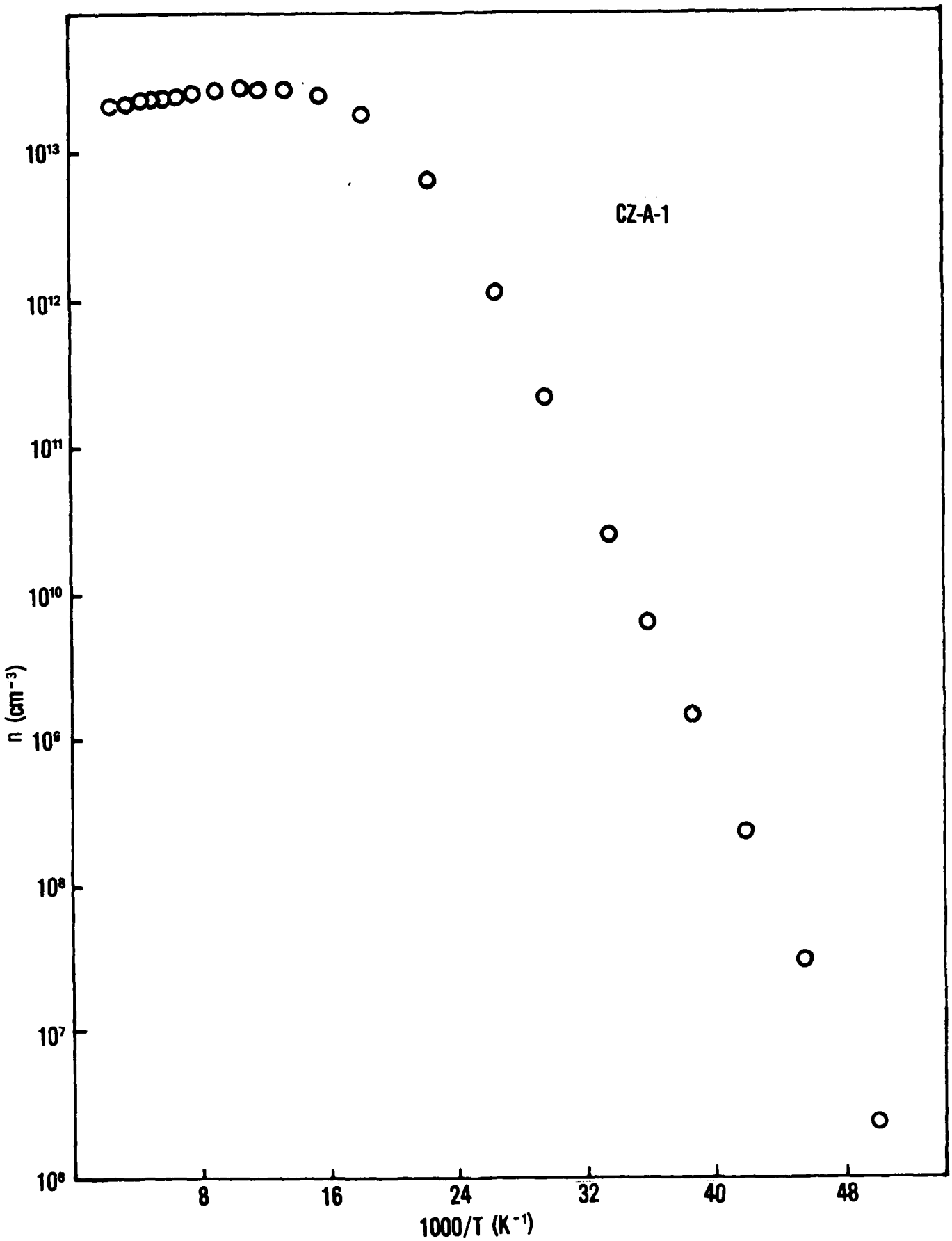

Figure 5. Carrier Concentration vs. Inverse Temperature for High Resistivity Commercial Czochralski Sample CZ-A

really the donor concentration minus the total concentration of acceptors. Boron is the usual acceptor in high purity float zone material and is usually present in concentrations around 10^{12}. Infrared absorption experiments were performed on this material and no impurity related levels were detected; so it is reasonable to assume that the shallow donor concentration in this sample is close to the value of n at saturation although the identity of the donors is not confirmed. We note here that the sudden increase in n at the highest temperature is reproducible and is most likely due to the onset of intrinsic conduction, that is, conduction due to electrons excited from the valence band and not from states in the forbidden gap. The slight decrease in n with increasing temperature just after saturation is also reproducible and is most likely due to the temperature dependence of the scattering factor, r. Figures 5 through 8 show n vs. 1/T and (μ) vs T for two commercial, phosphorus doped samples. Figures 5 and 6 are results for a high resistivity sample and Figures 7 and 8 are for a moderately doped sample. These are given for comparison. Note that the mobilities for the high resistivity sample are essentially identical to the nitrogen doped sample and that the shape of the n vs. 1/T curve is similar except for the lack of intrinsic conduction, the absence of which is expected in samples with this high of doping. The dip in n in the saturation region is even more pronounced for both of these samples, indicating a concentration dependence to the scattering factor that has not been previously emphasized in the literature.

As mentioned above, infrared absorption measurements were made on the nitrogen doped material. These measurements were made in our laboratory on 2mm thick samples. The only structure seen was that due to lattice absorption and no indication of the presence of either nitrogen or any other impurity was detected. The absorption measurements reported in Reference 1 used thicker samples than we did. Photoluminescence measurements were also made on as-grown nitrogen doped samples and excitations attributed to nitrogen were detected at low levels, confirming the presence of nitrogen.

2. ANNEALED, UNIRRADIATED SAMPLES

Several Si:N samples and commercial CZ samples were annealed at various temperatures for either one-half or one hour. The principal change in both types of material was the appearance of a high temperature shoulder in the n vs. 1/T plots. This was clearly visible in the Si:N sample shown in Figure 9 and in the high resistance CZ

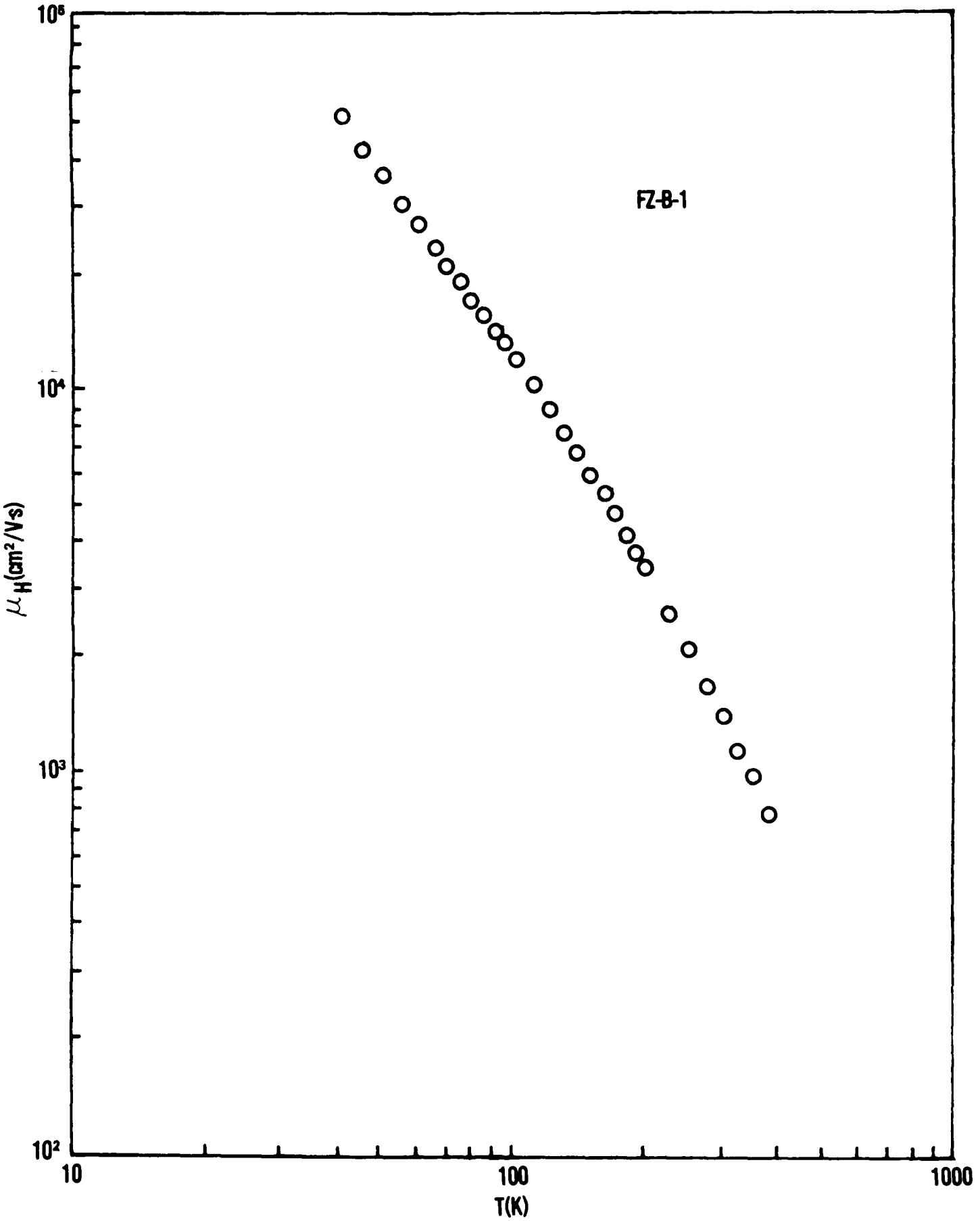

Figure 4. Mobility vs. Temperature for As-received Si:N Sample FZ-B

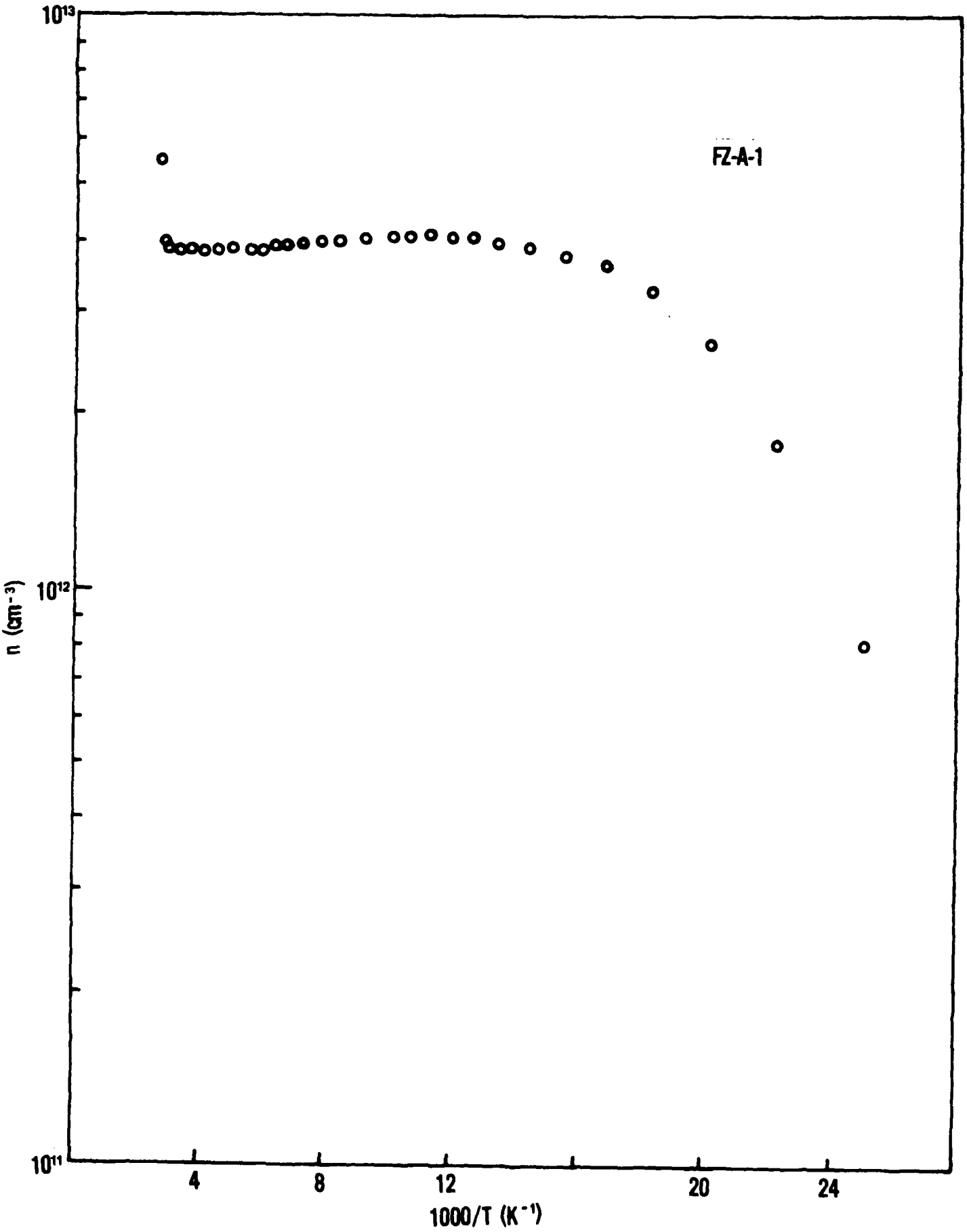

Figure 3. Carrier Concentration vs. Inverse Temperature for As-received Si:N Sample FZ-B

SECTION III

RESULTS

1. AS-RECEIVED

Several samples from the nitrogen doped crystal were run without any anneals or irradiations. The results for the best of these are shown in Figures 3 and 4, Figure 3 is the carrier concentration versus inverse temperature plot and Figure 4 is the mobility versus temperature plot. We were only able to take data down to 40K before the settling time for the sample voltages present during the voltage/current lead switching required for the van der Pauw technique became too long. The settling time was unmeasurable at room temperature, but became progressively worse as the temperature was decreased. This was a problem with all the unannealed Si:N samples and the cause is as yet undetermined. A similar effect at these low temperatures is often a problem with heavily damaged samples and in this case is related to poor mobilities. Examining Figure 4, we see that this cannot be the case for the Si:N samples because the mobility shows no sign of degrading at low temperatures and shows no sign at all of anything but phonon scattering, as would be the case in high quality undoped material. Another possible cause for excessive settling times is poor ohmic contacts. New contacts were laser annealed and the samples were rerun but there was no noticeable improvement. Different dopants were tried for the contacts and again there was no significant change. Phosphorus was the original contact dopant and antimony was the second dopant used. The van der Pauw resistance ratio for these samples, while not completely devoid of any temperature dependence, was still satisfactory, changing from 0.35 to 0.43 in the worst case. A temperature dependence in this ratio or a strong deviation of the value from unity for a highly symmetrical sample is often an indication of either inhomogeneities in the sample or bad contacts.

The settling time problem prevented us from obtaining an accurate determination of the activation energy from the log(n) vs 1/T data, but we were able to obtain a rough estimate which indicated that phosphorus (E = 0.045eV) was the dominant shallow level. An estimate of the concentration of shallow donors can be obtained from the value at which n saturates, about 4×10^{12} or this sample. The donor concentration must be at least this high but can be larger because the value at saturation is

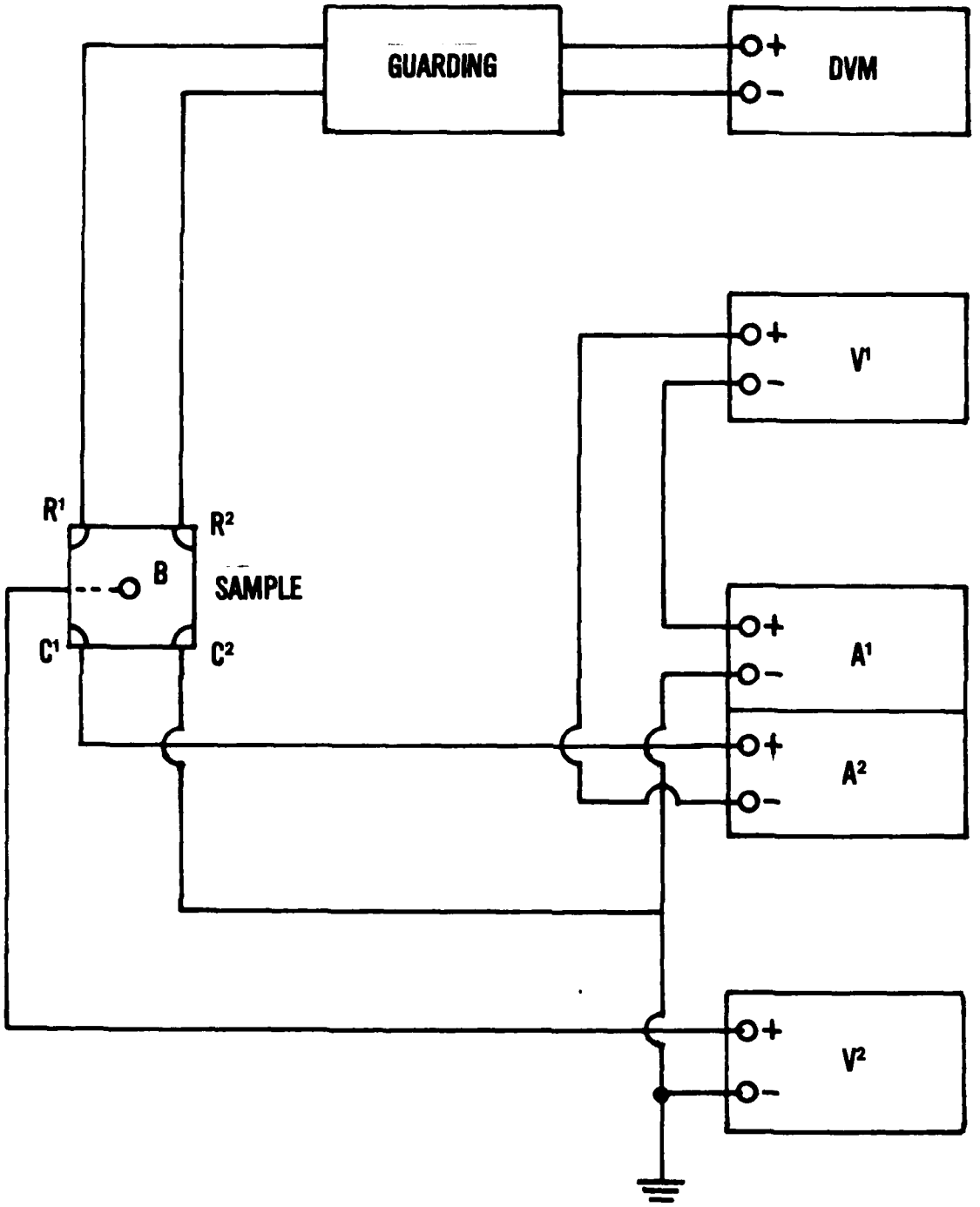

Figure 2. Experimental Set-up for Reverse Biasing the Substrate of an Ion Implanted Sample

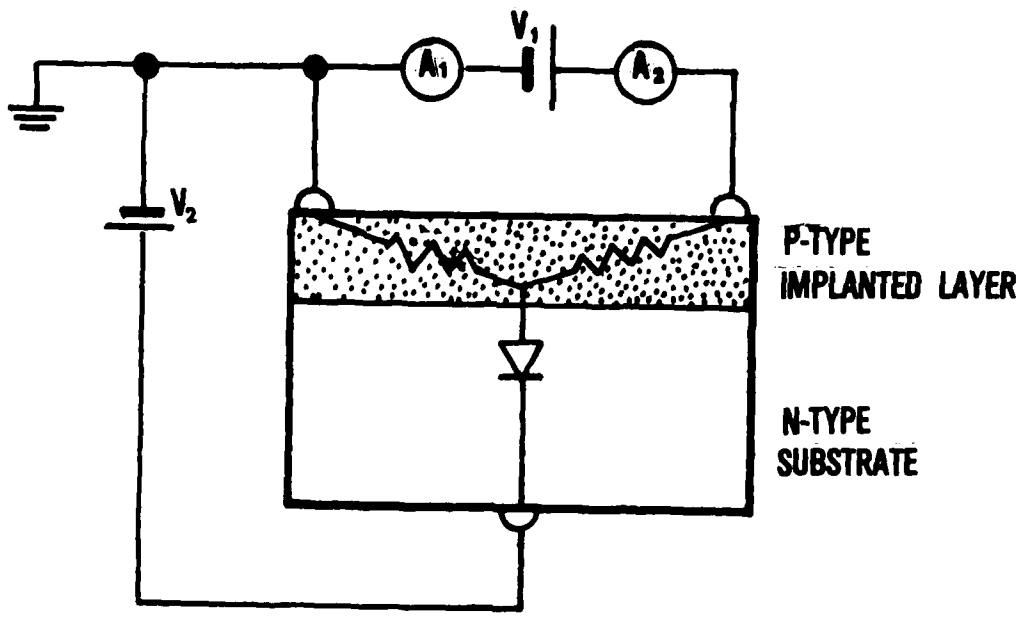

Figure 1. Proposed Technique for Reverse Biasing the Substrate of an Ion Implanted Sample for Hall Effect Measurements

The furnace was first brought up to temperature and then the sample platform was inserted into the hot zone at the center of the tube. The ambient was flowing, high purity argon.

The Hall effect samples were laser annealed to put ohmic contacts on the four corners (Reference 14). Contact dopants were either phosphorus or antimony. Doping was restricted to an area of approximately 2 mm^2. Copper leads were soldered to the contact regions with indium solder after cleaning. The Hall effect apparatus used for these experiments was the guarded, high impedance system of AFWAL/MLPO described elsewhere (Reference 14).

In order to investigate the effects of ion implantation of p-type dopants in n-type Si:N material, a modification of the standard Hall effect set-up was developed. Due to experimental difficulties involved with the ion-implantation, this experiment was not performed, but a description of the modifications is included for future reference. Even though the resistivity of the substrate is high and that of the implanted layer is considerably lower, extra precautions must be taken when attempting to make measurements on the implanted layer to prevent leakage currents through the substrate that would invalidate any results. To this end, an arrangement for back biasing the substrate was developed as well as a method for determining the magnitude of the leakage current. Figure 1 shows schematically how this is done. V_1 supplies the current to the implanted layer while V_2 supplies the bias voltage to the substrate. V_1 and V_2 are arranged in such a manner so that if V_2 is greater than V_1 all regions of the substrate are reverse biased. If there is any leakage current V_2 will draw current and so the two ammeters A_1 and A_2 will read different values. A difference of a percent or so in A_1 and A_2 will indicate a significant leakage current. If the ammeters are the two portions of a dual channel electrometer the difference can be displayed directly. Figure 2 shows a possible realization of this arrangement. A1 and A2 are the two channels of a Keithley model 619 electrometer. V1 can be either a constant current source or a standard laboratory power supply. V2 is a standard power supply and DVM is a high resolution differential voltmeter.

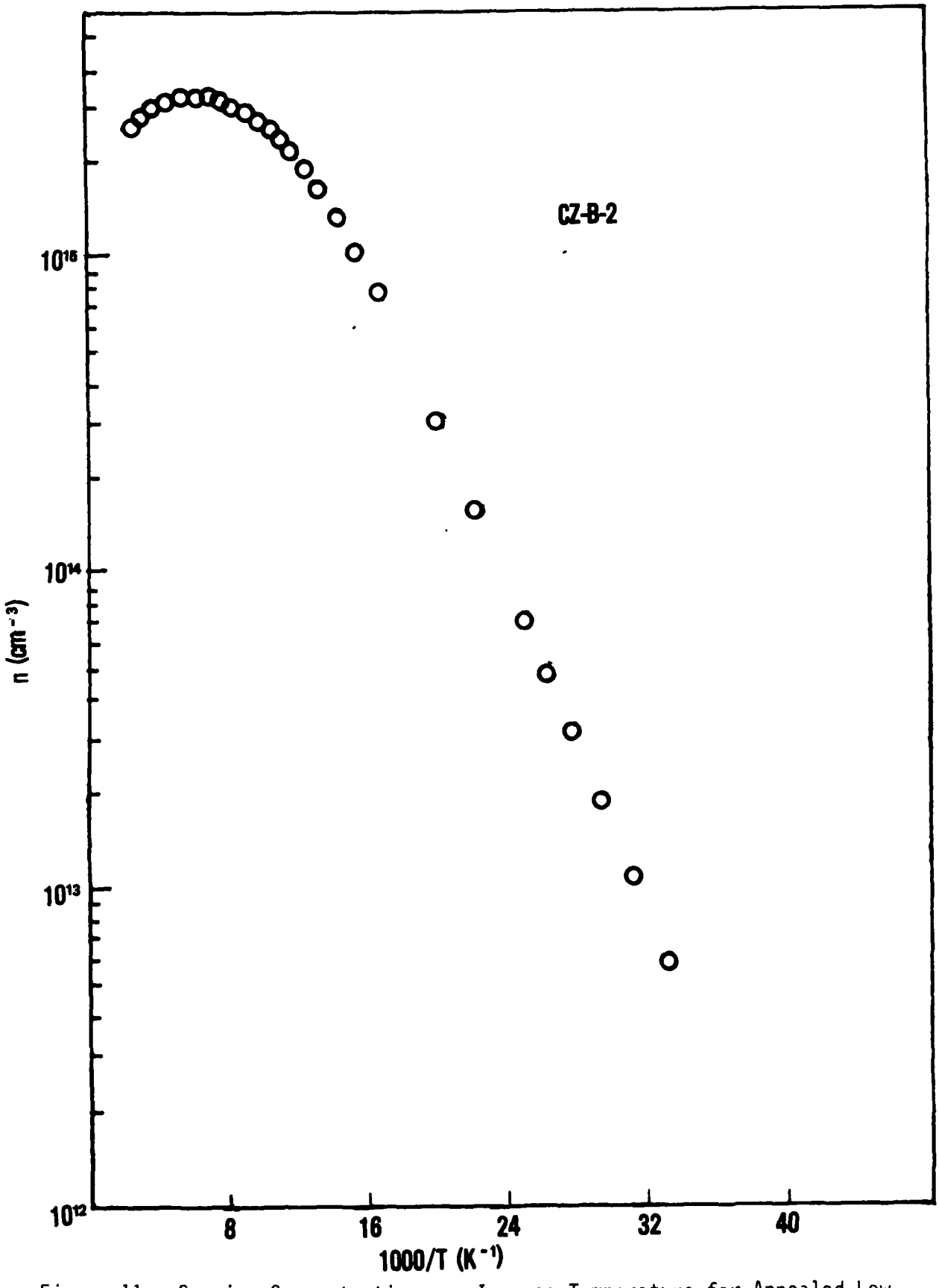

Figure 11. Carrier Concentration vs. Inverse Temperature for Annealed Low Resistivity Commercial Czochralski Sample CZ-B, Annealed at 900°C for One Hour

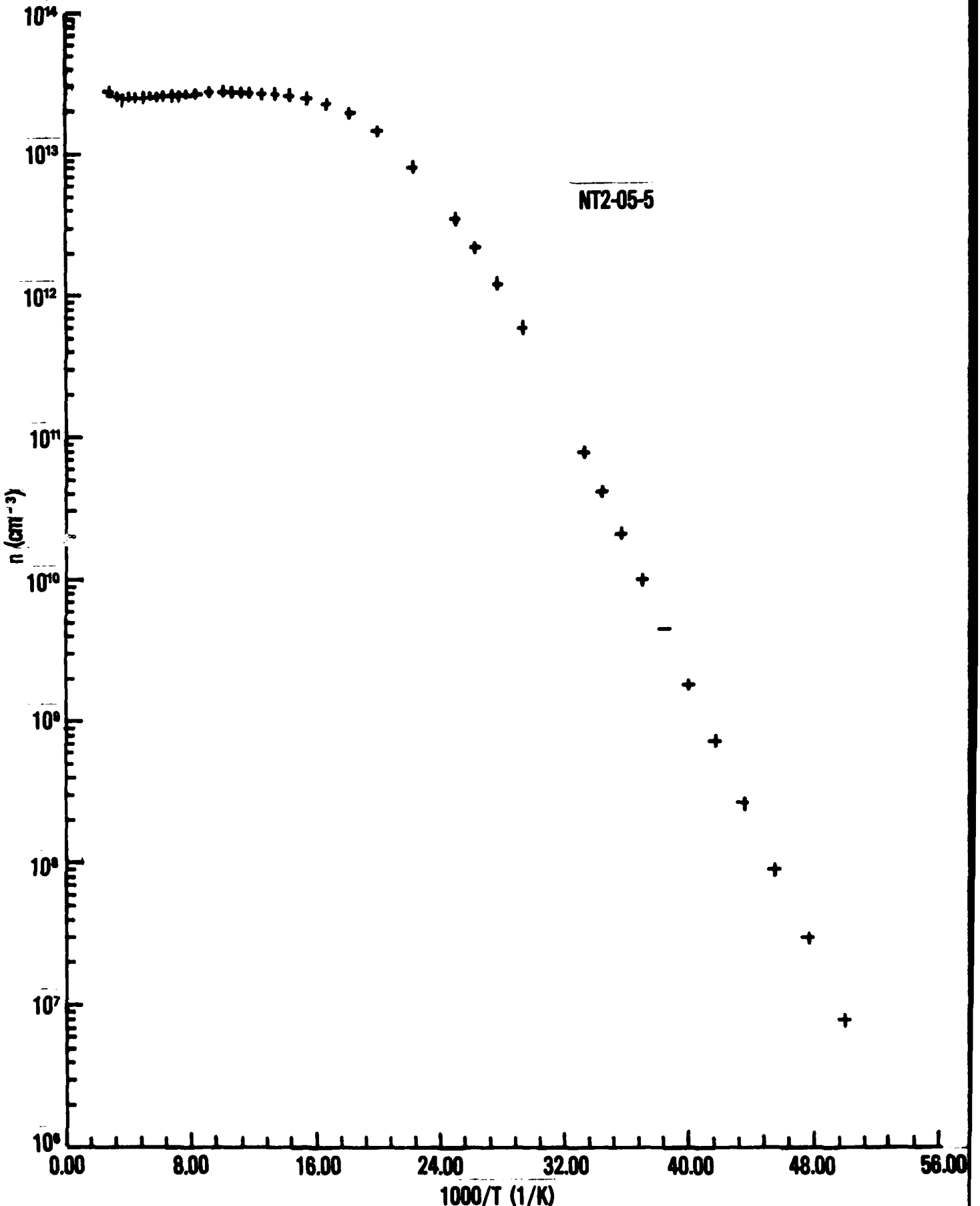

Figure 12. Carrier Concentration vs. Inverse Temperature for Annealed, NTD'd, Undoped Float Zone Sample NT2-05, Annealed at 800°C for One Hour

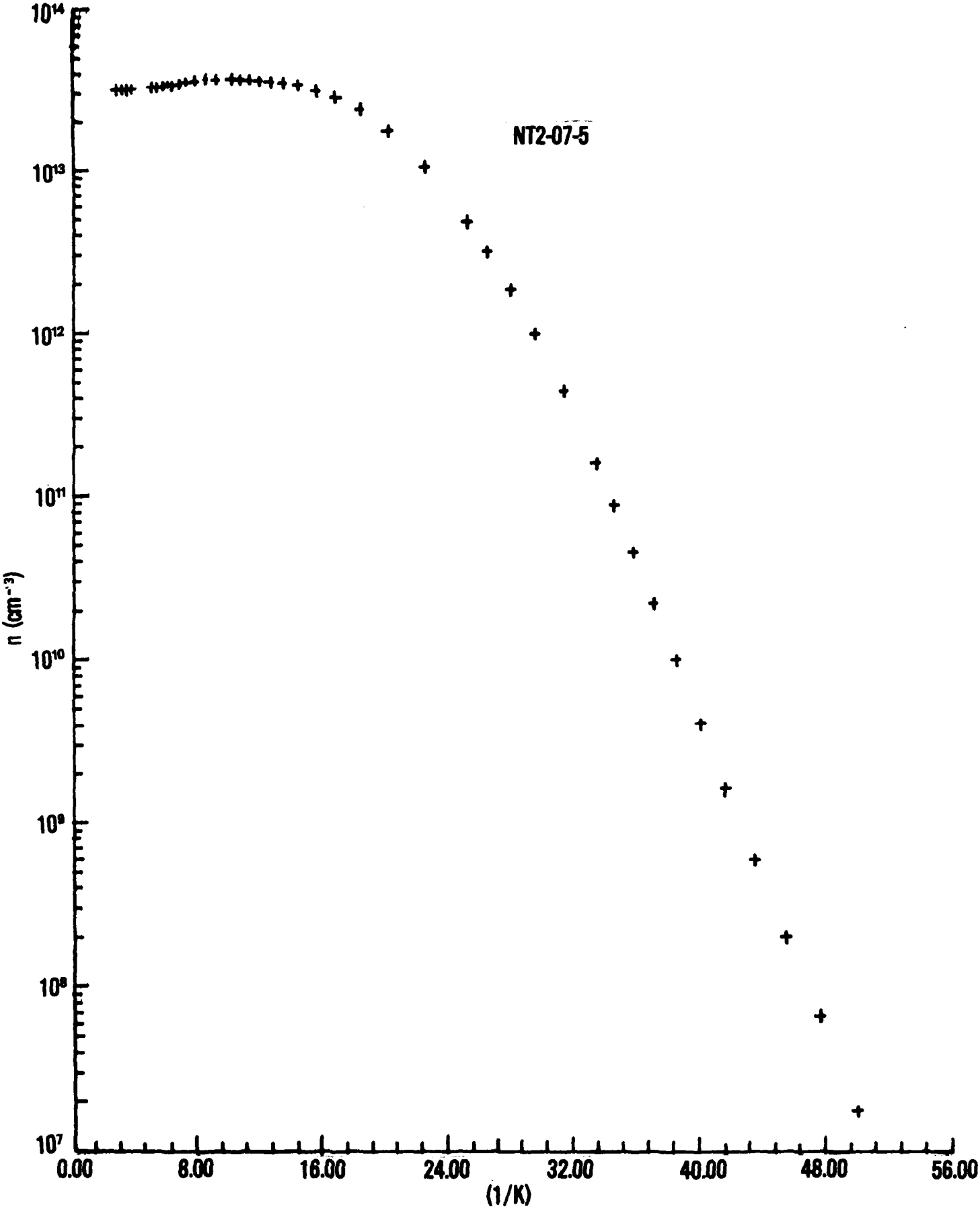

Figure 13. Carrier Concentration vs. Inverse Temperature for Annealed, NTD'd, Undoped Float Zone Sample NT2-07, Annealed at 800°C for One Hour

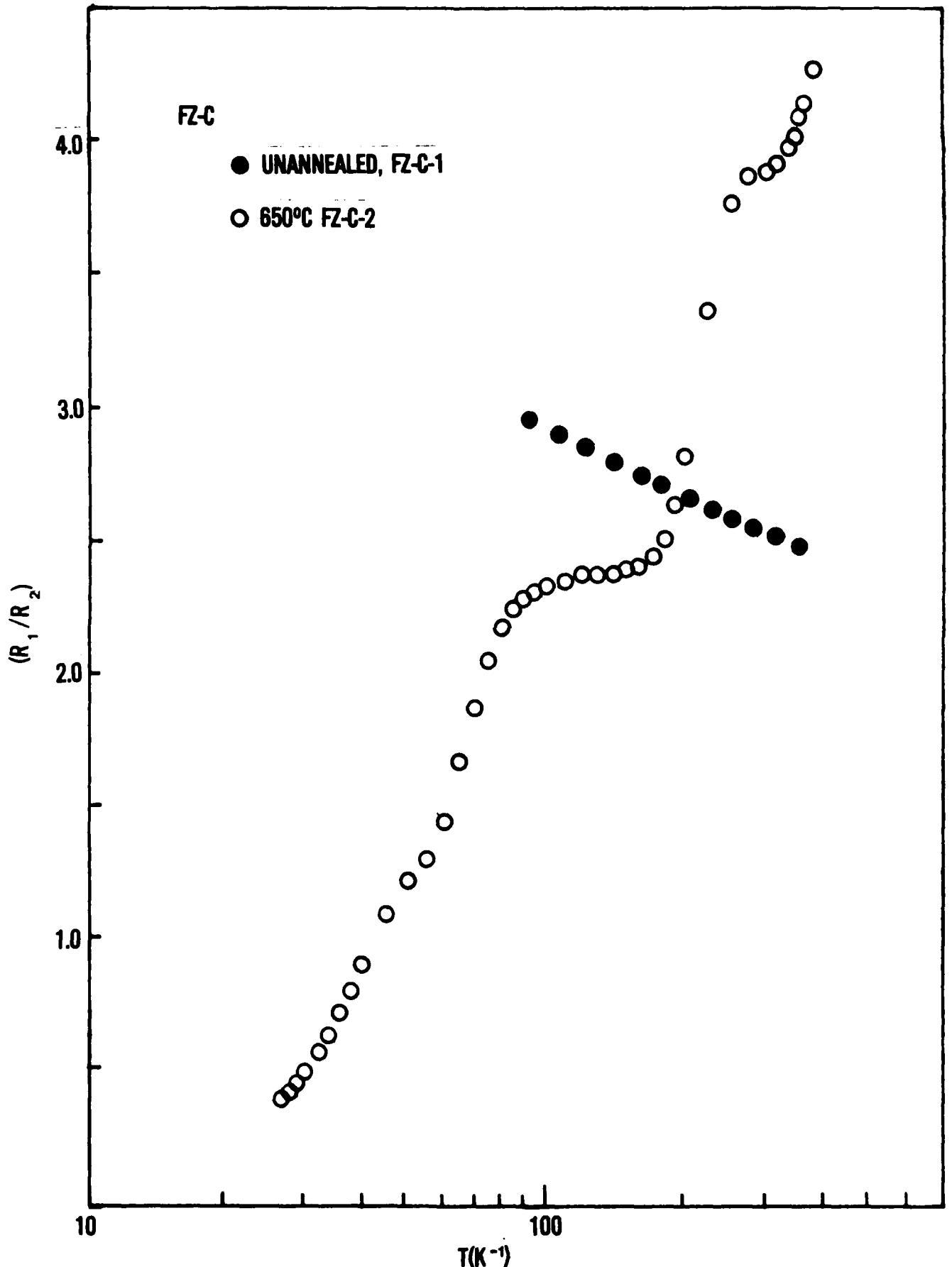

Figure 14. van der Pauw Resistance Ratio for Si:N Sample FZ-C before and after a One Half Hour Anneal at 650°C

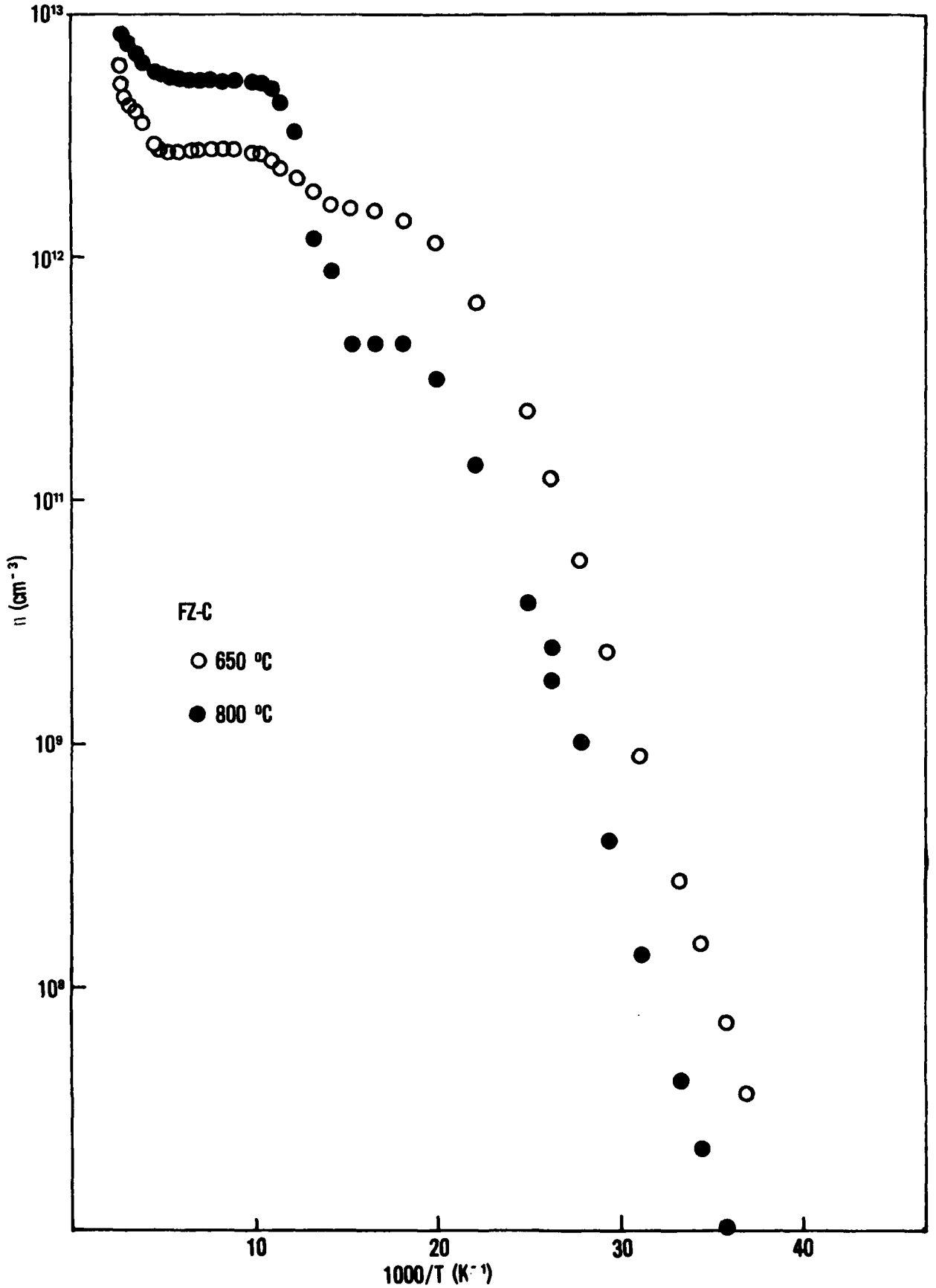

Figure 15. Carrier Concentration vs. Inverse Temperature for Si:N Sample FZ-C before and after One Half Hour Anneal at 650°C

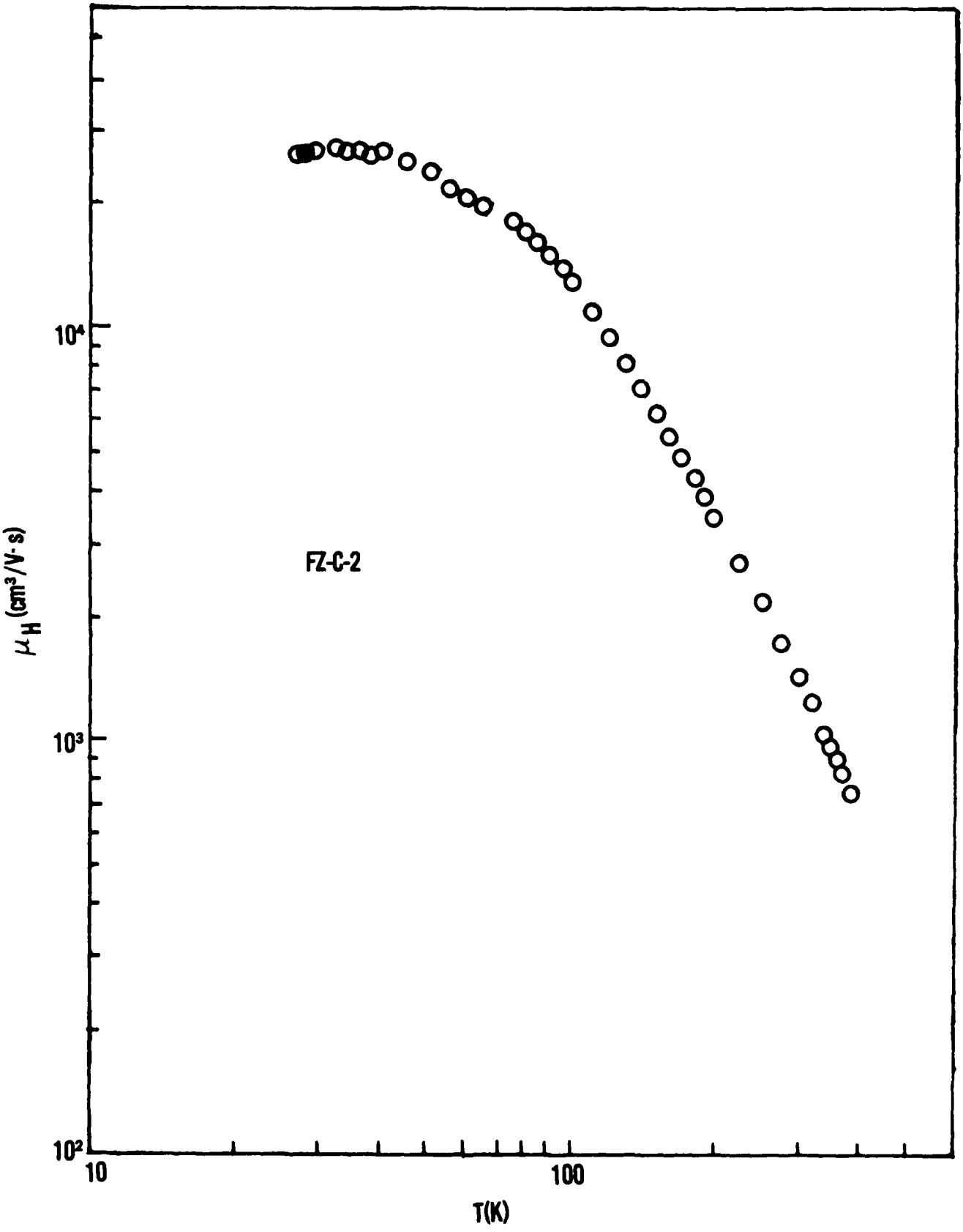

Figure 16. Mobility vs. Temperature for Si:N Sample FZ-C after 650°C Anneal

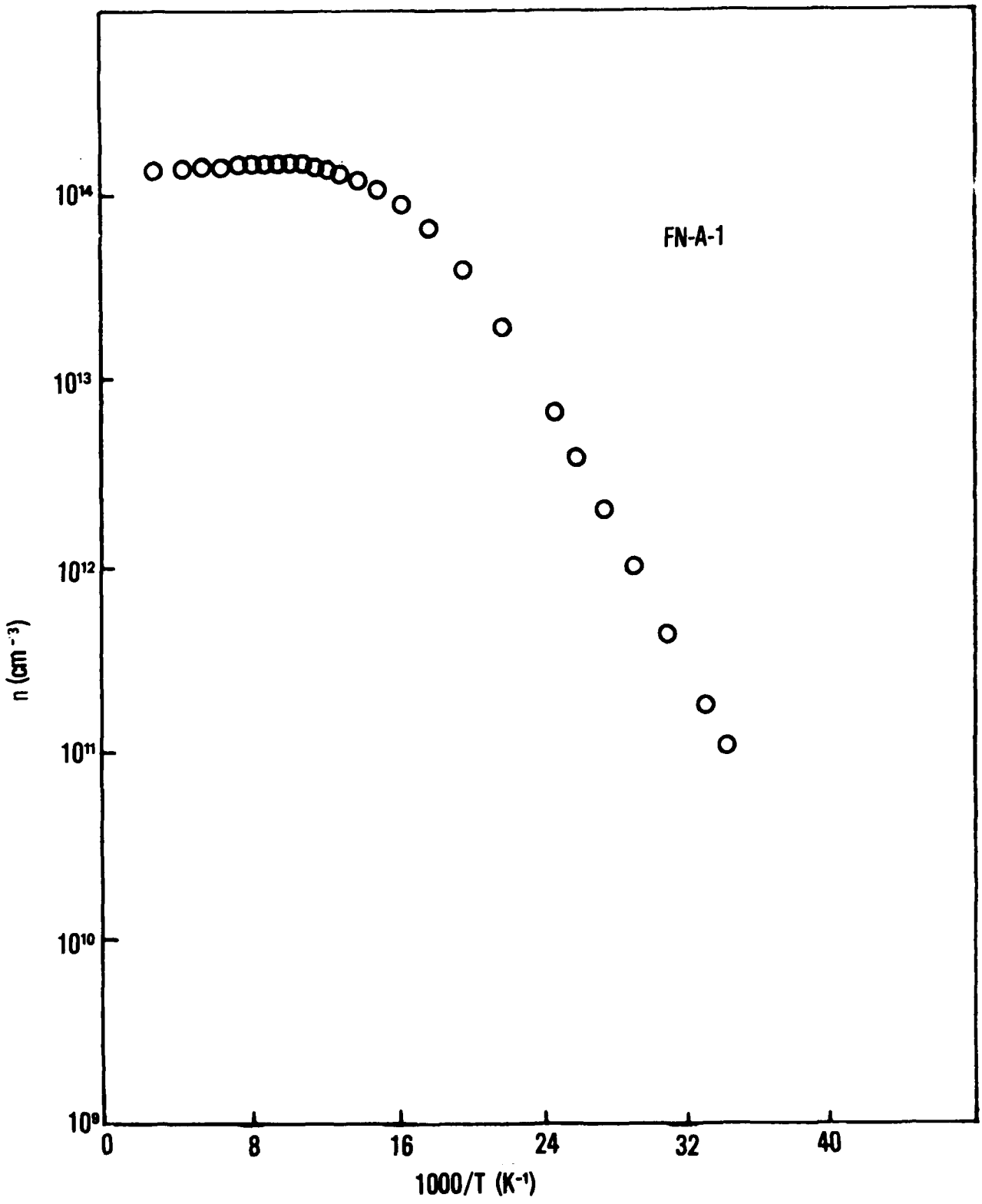

Figure 17. Carrier Concentration vs. Inverse Temperature for NTD Si:N Sample FN-A after Annealing at 800°C for One Hour

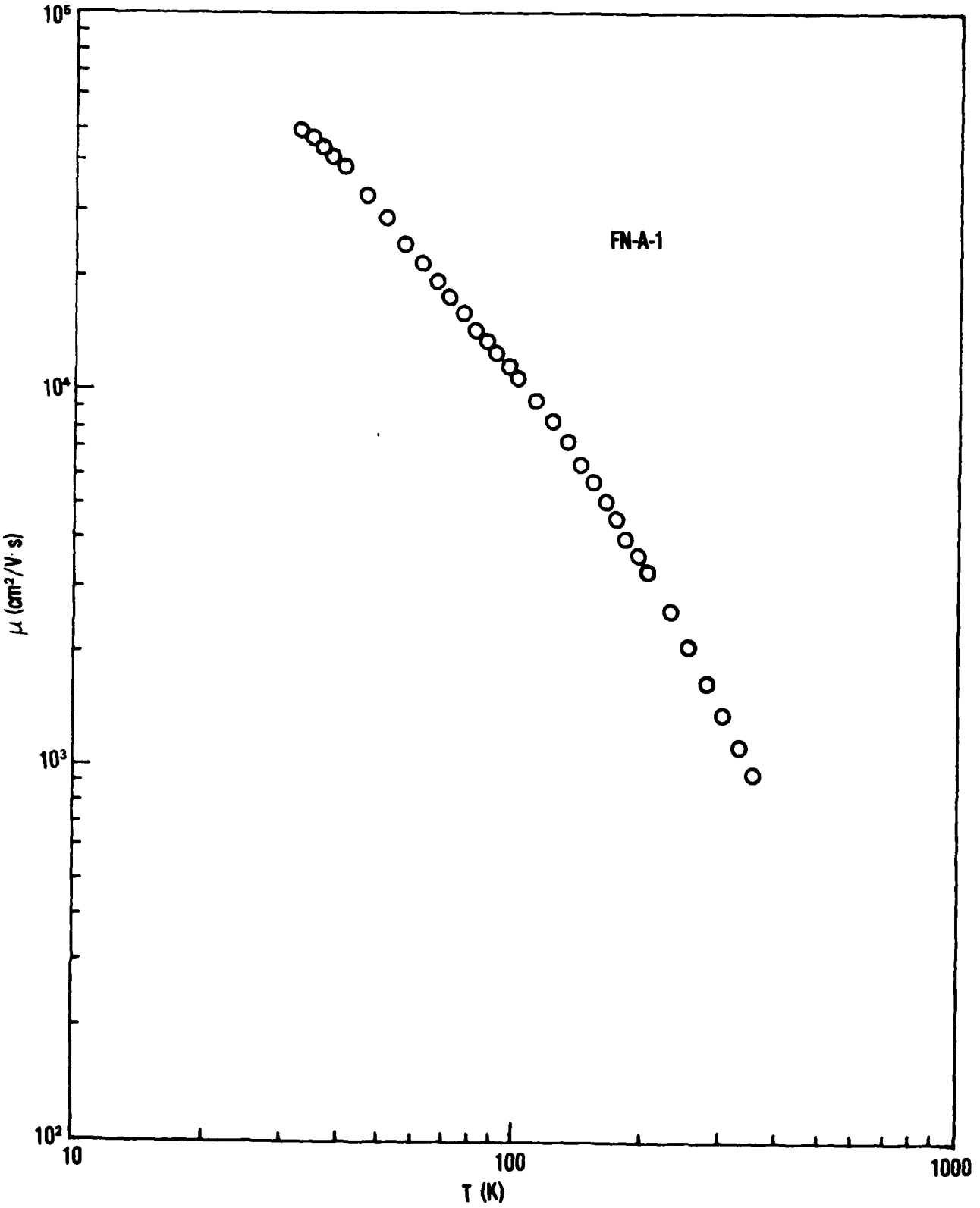

Figure 18. Mobility vs. Temperature for NTD Si:N Sample FN-A after Annealing at 800 C for One Hour

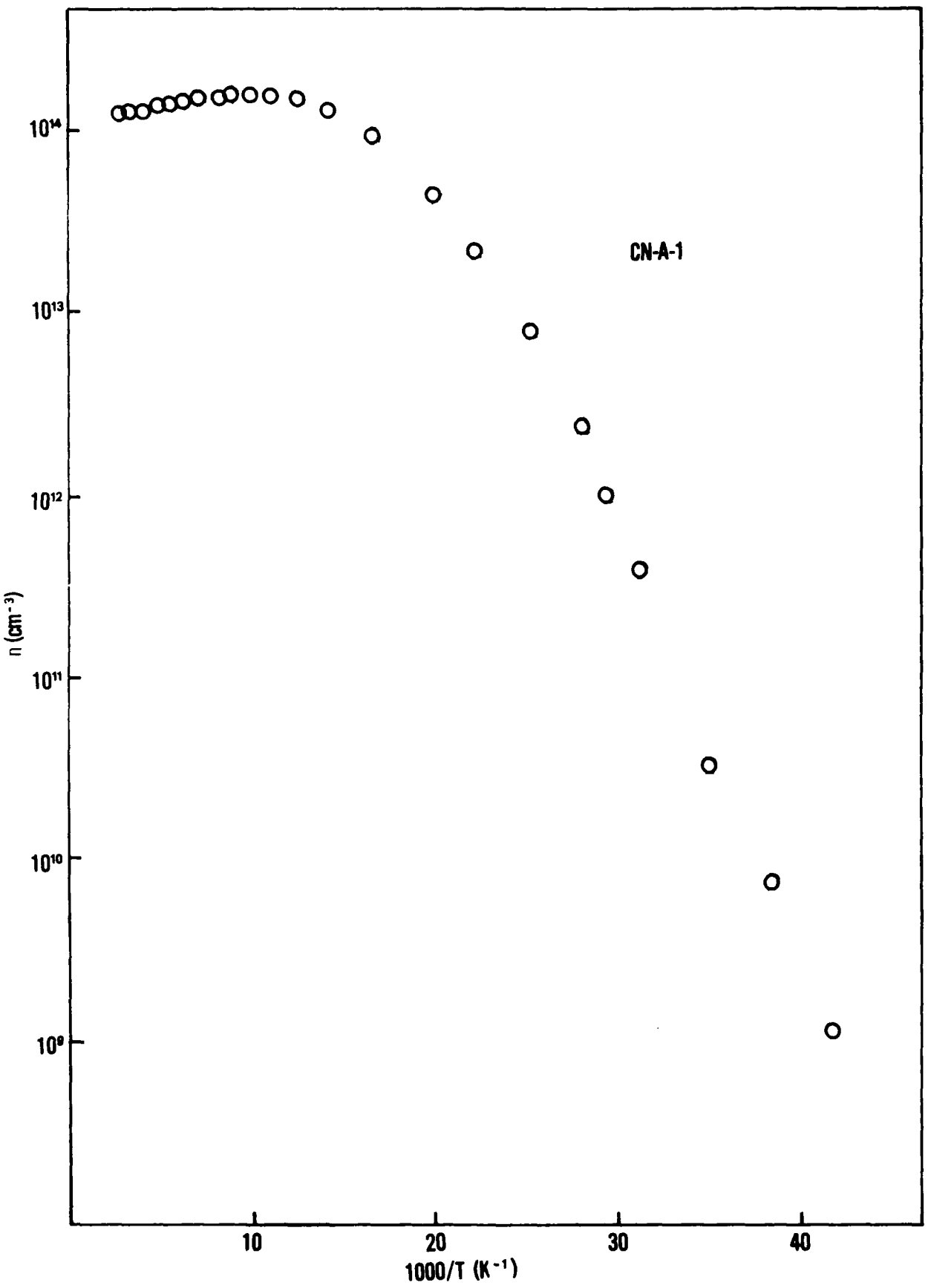

Figure 19. Carrier Concentration vs. Inverse Temperature for NTD Commercial Czochralski Sample CN-A after Annealing at 850°C for One Hour

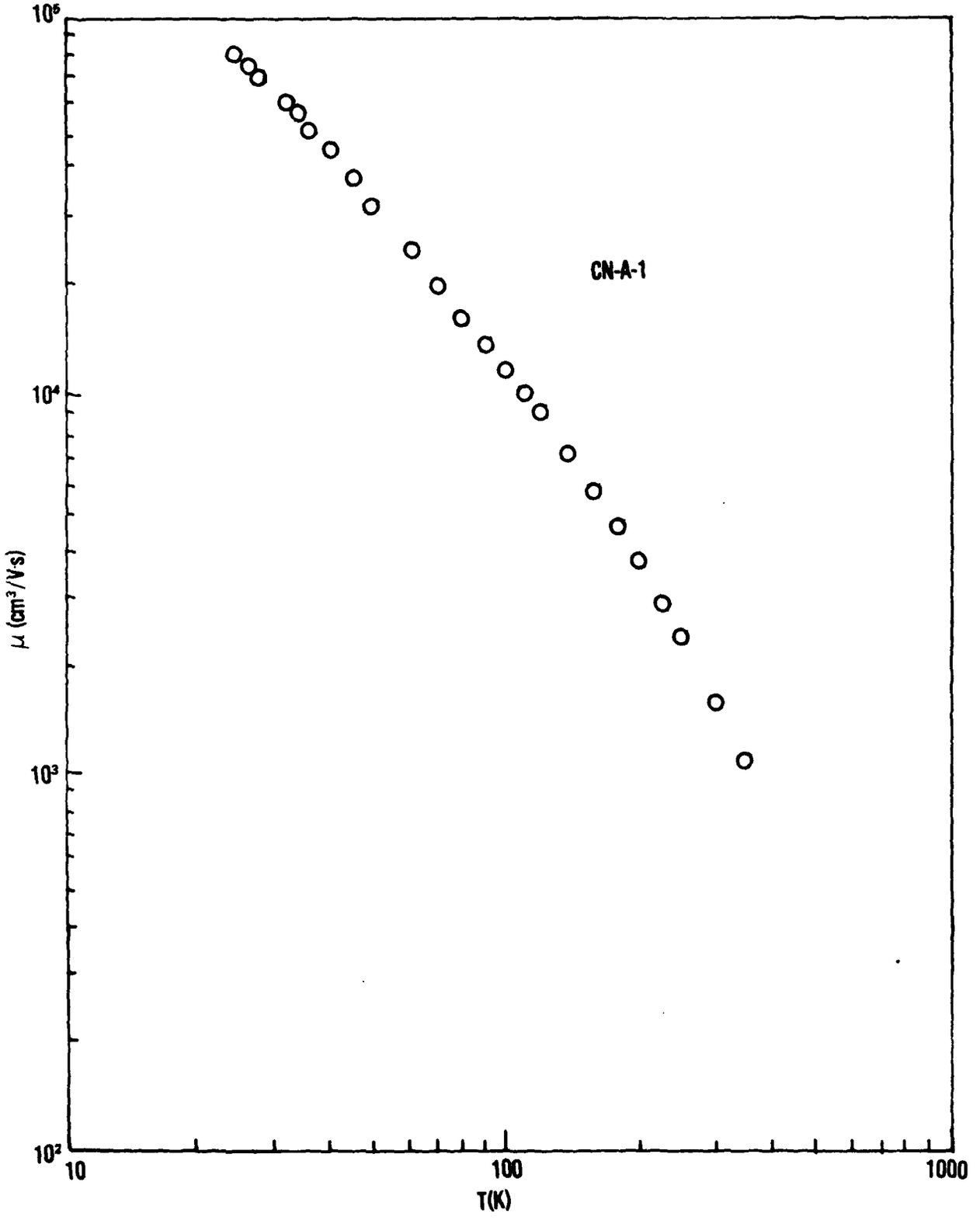

Figure 20. Mobility vs. Temperature for NTD Commercial Czochralski Sample CN-A after Annealing at 850°C for One Hour

SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

We have examined the electrical properties of nitrogen doped float zone silicon and compared the results with those for commercial Czochralski grown silicon. We have found no evidence for the presence of any shallow levels that might be related to nitrogen doping. The deep levels that appear to form during high temperature anneals might be related to nitrogen, although similar effects are seen in Cz material with no nitrogen. It is well known that oxygen in Cz material produces a multitude of electrically active levels after various anneals. The deep levels seen in the Cz samples are probably due to oxygen but this is probably not the case in the Si:N samples because infrared absorption measurements detected only minimal amounts of oxygen. The disturbing inhomogeneities that occur after annealing might be related to the deep levels as well, and it is possible that the anneals form some kind of extended complex such as silicon nitride precipitates. Further investigation is needed before these statements can be considered any more than speculation. Experiments such as deep level transient spectroscopy (DLTS) would give considerable information on the deep levels that are produced and advanced microscopy experiments might be able to see any precipitates or extended defects.

These problems mentioned above should be investigated but the experiments on the neutron transmutation doped material are very encouraging. The problems seen in the high resistance Si:N samples are all but eliminated by the very light doping that the NTD introduced. Most electronic device applications require considerably higher doping concentrations than those used in the NTD experiment. Therefore, it can safely be assumed that any inhomogeneities will be masked by the doping and so Si:N appears to be identical in electrical properties to Czochralski, at least for the single boule studied here. More experiments on different boules, including p and n-type doped material as well as intrinsic, are clearly needed. In addition to Hall effect measurements, DLTS and electron microscopy experiments, mechanical properties should be further investigated. The experiments reported here are successful enough to warrant the use of nitrogen doped material, particularly n and p-type material, in the fabrication of actual devices to compare the yields and performances with more conventional material. Investigations of the internal gettering properties of nitrogen or nitrogen related complexes should be investigated to determine if this

material can be used as a replacement for oxygen doped, Czochralski grown silicon for very large scale integration (VLSI) applications. At present, the processing of VLSI circuits introduces small but significant quantities of such impurities as iron which degrade device performance but are gettered to oxygen precipitates and removed from the active region of the substrate by same processing steps. If nitrogen proves as effective as oxygen, or if cleaner, low temperature processing steps can be developed, then this nitrogen doped, float zone material might become a significant competitor in the VLSI field.

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