Teller tetragonal distortion due to the interaction with the ϵ_g mode and of the depolarization due to the interaction with the τ_{2g} mode. It should be noted that, since the polarization of luminescence is caused by the JTE, the degree of polarization as a function of azimuthal angle gives little information on the direction in which the vacancy exists. In fact, since $d<0$ as concluded above, the vacancy may exist in the nearest-neighbor position although the degree of polarization as a function of azimuthal angle in the (100) plane becomes zero in the [011] and [0$\bar{1}$1] directions.

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Electron-Spin-Resonance Measurements of the Spin Susceptibility of Heavily Doped *n*-Type Silicon*

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The results of relative spin-susceptibility measurements as a function of donor concentration are reported for heavily doped *n*-type silicon. It is shown that, at liquid helium temperatures, the susceptibility decreases with impurity concentration for samples in the impurity-band region. At still higher concentrations, the susceptibility is well described by the (concentration)$^{1/3}$ law expected for a degenerate electron gas.

Results of ESR measurements of the spin susceptibility associated with heavy dopings of phosphorus impurities in silicon have been reported[1-3] and interpreted in terms of combined impurity-band and localized-electron models. Several anomalous features of these results, which appeared at liquid helium temperatures, have been treated with some skepticism[4] primarily because of (1) the inherent inaccuracy of an ESR method, which in principle requires a double integration of the observed resonance line and a comparison with the broad line of a CuSO$_4$·5H$_2$O "standard" sample, and (2) the absence of similar anomalies in subsequent static susceptibility measurements.[2,5]

This Letter presents the results of our own ESR susceptibility measurements which are in disagreement with those previously reported[1] but are qualitatively consistent with other related experimental data.[4] Our data were obtained through the use of an X-band (~9.1 GHz) TE$_{104}$ double-sample cavity equipped with a modulation "switch." The latter device utilized the fact that a reduction in the amplitude of the ~2-kHz magnetic modulation field at one of the two sample sites reduces the contribution of the sample at that site to the total observed signal (our spectrometer detected synchronously to this modulation). Thus by recording the two overlapping signals first in the absence of this attenuation and then with each of the individual sample signals in turn attenuated, it is possible to separate out the relative signal intensities of the two samples. In practice, since the two sample sites could not be exactly equivalent, as additional set of measurements was necessary in which the sample positions were interchanged.

The "switch" was a length of copper tubing which could be moved vertically over the length of the cavity by an attached rod operable from the outside of the cryostat. This tubing mated with flanges fixed to the cavity's exterior walls so that it could (the 2-kHz skin depth was much less than the tubing wall thickness) attenuate the modulation field at either the lower sample site ($\frac{1}{2}\lambda$ above the cavity bottom) or at the upper site ($\frac{1}{2}\lambda$ below the cavity top or iris). It was also possible to leave the fields unattenuated at both sites.

The phosphorus-doped silicon samples used in these experiments were in powder form and ranged in concentration from $N_0 = 2.5\times10^{18}$ to $N_0 = 10^{20}$ donors/cm^3. The powders were obtained by mortar-and-pestle grinding of slices cut from purchased single-crystal boules.[6] The "surface" ESR line[7] produced by this grinding was removed by a HF-HNO$_3$ etch and a 400°C anneal.[8] The powders were separated into three portions, each of more or less uniform particle size, by a sedimentation process. The finest of these powders consisted of particles generally smaller than 1 μm and were not used in this study because of their relatively large surface-to-volume ratio. Samples were taken from each of the other portions and put into uniformly sized, Lucite-capped sample tubes which could be positioned reproducibly in Lucite sample holders mounted inside the cavity.

Susceptibility comparisons, by our modulation switch method, of samples of identical concentration but different particle size indicated no difference outside of our experimental error. This error was estimated as ≈7% by comparison measurements of samples of known weights taken from the same portion of the same material. Similarly our results were shown to be independent of the screening of the microwave field by the outer portions of the sample and of the annealing used to eliminate traces of the surface line. Data were, of course, taken at microwave powers sufficiently low to avoid saturation effects.

Comparison data were taken at 1.1, 4.2, and 77°K with a standard X-band spectrometer, klystron-locked to the sample cavity frequency and adjusted to minimize dispersive (χ') mixing into the desired absorptive (χ'') signal. The quantity $\chi_p \propto \int_0^\infty \chi''(H)dH$ was determined by a computer double integration[9-11] of the digitally recorded spectrometer output signal which is proportional to $d\chi''/dH$.

In view of the fact that our technique yielded relative intensity measurements only in a pairwise fashion, the deduction of a meaningful relative susceptibility-versus-concentration curve required a selection of these pairings which would minimize pyramiding errors. Inasmuch as our technique was most sensitive for samples of similar linewidths and intensities, our procedure compared all samples of concentration greater than 1.3×10^{19} donors/cm^3 to the sample of this concentration. Similarly all samples of concentrations less than 7×10^{18} donors/cm^3 were compared against a sample of this concentration. These data together with comparison of the two "standards" (7×10^{18} and 1.3×10^{19} donors/cm^3) gave a direct comparison, relative to the 7×10^{18}-donor/cm^3 sample, for concentrations less than or equal to 1.3×10^{19} donors/cm^3 and a two-step comparison for more highly concentrated samples. Susceptibility ratios obtained from direct comparisons of samples of adjacent concentrations as well as those obtained from comparisons against a common (3.5×10^{18} donors/cm^3) sample were in agreement, within experimental error, with the values obtained from our previously described scheme. Thus, at a given temperature, the susceptibility relative to that of an aribtrarily chosen sample can be specified to an accuracy of about 10%.

The three sets of constant-temperature data were then related by a determination of $\chi_p(T)$ for two samples relative to a "standard" with a known susceptibility temperature dependence. We eschewed the use of single-crystal CuSO$_4\cdot$5H$_2$O samples in this capacity because of their large linewidths and the disagreement between the two sets[1, 12] of $\chi_p(T)$ data obtained with this standard. Our calibration was done against the signal[13] obtained from a 700°C-annealed crystal of neutron-irradiated LiF. The latter signal has been shown to be due to microscopic lithium globules and should, for the temperatures of interest, have a nearly temperature-independent Pauli-like susceptibility.[14-16] We have not assumed any particular value for this susceptibility but merely have assumed it to be independent of temperature. This is sufficient to allow relative measurements of χ_p as functions of concentration and temperature. This comparison was made for both the 2.5×10^{18}- and 1.3×10^{19}-donor/cm^3 samples. The ratios of the 2.5×10^{18}-donor/cm^3 susceptibilities, at each of the three temperatures, obtained by direct comparison against LiF agreed, within experimental error, with those obtained by similar "direct" measurements on the 1.3×10^{19}-donor/cm^3 sample and the use of the single-temperature susceptibility comparison data. This calibration method allows us to relate the three single-temperature relative susceptibility results with an estimated maximum error of 10%.

Figure 1 represents our results for $\chi_p(T)$ as a function of concentration (N_0). The units of the ordinate axis refer all susceptibilities to that of the 2.5×10^{18}-donor/cm^3 sample at 1.1°K. The dashed curve represents the relative susceptibility results of Maekawa[1] at $T=1.5$°K. This curve is derived from the reported absolute susceptibility measurements by arbitrarily requiring agreement of the two sets of data at 4.2°K for a $N_0=2.5\times10^{18}$-donor/cm^3 sample. It can be observed that the previously reported susceptibility peak at roughly 2×10^{19} donors/cm^3, and the subsequent falloff at higher concentrations, are absent from our results. In fact our data for $N_0\gtrsim2\times10^{19}$ donors/cm^3 qualitatively verify the $N_0^{1/3}$ susceptibility dependence inferred from Knight-shift measurements[17] and expected on the basis of a Fermi level located within the conduction band of the silicon host.[4, 18] The small differences, in this concentration region, between the measured susceptibilities at the three temperatures are within our experimental error.

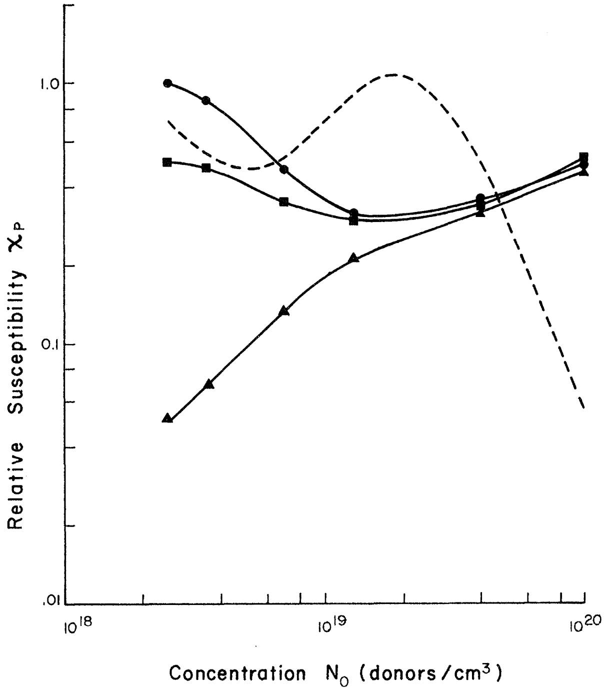

FIG. 1. The relative spin susceptibility χ_p, with respect to that of a 2.5×10^{18}-donor/cm^3 sample at 1.1°K, plotted as a function of phosphorus-donor concentration at $T=1.1$°K (circles), $T=4.2$°K (squares), and $T=77$°K (triangles). The dashed curve represents the relative susceptibility at $T=1.5$° inferred from the results of Maekawa (Ref. 1).

At 1.1 and 4.2°K the falloff of χ_p with increasing donor concentration in the low-concentration region of Fig. 1 is evidently due to the "condensing" of somewhat localized spins into a degenerate "impurity band" at higher concentrations. Some evidence of this behavior has been seen previously in static susceptibility measurements.[5] However, we feel that the previously proposed[5] explanation of this effect, in terms of the existence at these concentrations of "isolated" donors, is unrealistic. Such a model requires that donors as close as 55 Å act as "isolated" spins. This requirement is not in accord with recently published measurements[19] of the exchange energies of donor pairs as a function of the interdonor separation. In any case the postulated number of these isolated spins is sufficient to allow their distinct spectrum to be observed. We have observed no evidence of such a spectrum. It would appear to us more fruitful to develop models in this region either in terms of large "clusters" of impurity atoms or on the basis of incompletely degenerate impurity bands.

We suspect that the large differences between our results and those of Maekawa[1] could be due to the shortcomings of the single-crystal samples used in the earlier experiments. At high concentrations, the sample thicknesses were no longer small compared to the microwave skin depth and the application of corrections for the effective sample volume is questionable. The etching technique used to produce sufficiently thin samples may have affected the sample susceptibility since etching often proceeds preferentially in the more heavily doped regions of a sample. Because of these difficulties, as well as the need for an individual calibration of each sample against the broad $CuSO_4\cdot5H_2O$ line, we feel the previous experiments to be inherently less accurate than our direct comparison of powder samples. The possibility that our powder results are not truly representative of bulk properties appears to have been eliminated by the independence of these results with respect to particle size (as long as the particle sizes are approximately less than the microwave skin depth).

These experiments are now being extended to other temperatures and a study of a number of calibration standards is being carried out which, it is hoped, will allow accurate absolute susceptibility measurements.

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X-Ray Topographic Observation of Antiferromagnetic Domain Boundaries in Chromium

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Antiferromagnetic domains in Cr have been observed visually by the x-ray double-crystal method in the low-temperature phase which corresponds to the longitudinal spin-density–wave state. The contrast between neighboring domains is due to a slight tetragonality of the crystal lattice. The domain boundary has been found to be sharp and mobile, and its section is straight if not trapped by imperfections.

There have been several x-ray topographic investigations on antiferromagnetic domains. The so-called *T* walls were easily detected in NiO[1,2] and CoO,[3] because of a relatively large rhombohedral distortion of the crystal lattice in each case. In NiO, the *S* walls were also detected[4] because of local strain of about 10^{-4}. In the case of chromium, possible deviation from cubic symmetry in its antiferromagnetic state below the Néel temperature, 311°K, has not been detected by the x-ray–diffraction method,[5,6] at least in the temperature range surveyed. Recently this deviation was clearly shown by measurements using the strain-gauge technique[7] or the three-terminal capacitance technique.[8] Although these measurements were carried out mainly in the higher temperature region, 122-311°K, where the crystal is in a transverse spin-density–wave state (AF_1), the result obtained[8] suggested that the deviation $(a-c)/a$ is of the order of 10^{-5} in the temperature region lower than the spin-flip temperature, 122°K, where the crystal is in a state of longitudinal polarization (AF_2).

In the present study, an attempt has been made to take an x-ray topograph of antiferromagnetic domain boundaries in the AF_2 state by the method of double crystals in a parallel position using the 211 reflection of Cr, as shown in Fig. 1. Be-