Magnetic properties of electrons and holes in metallic Si : P and Si : B^T

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We present new NMR data on the P^{31} resonance in Si:P and B^{11} resonance in Si:B for samples in the impurity concentration range from 5×10^{18} to 9×10^{19} cm⁻³. The data give further information about the magnetic properties of the charge carriers in these metallic samples. These data, taken in conjunction with recent NMR data for Si^{29} by Sasaki, Ikehata, and Kobayashi in Si:P in the same concentration range place certain restrictions upon models which have been introduced to interpret magnetoresistance and spin-susceptibility data. Both $P³¹$ and $Si²⁹$ data in Si:P exhibit a temperature dependence of linewidth and Knight shift for T below 4.2 K. The results are compared to the ESR spin-susceptibility measurements of Quirt and Marko and of Ue and Maekawa. It is observed that the temperature dependence of the Knight shift and linewidth can be almost entirely accounted for by the temperature dependence of the spin susceptibility. The NMR data support a simple model in which all donor electrons or acceptor holes participate in a single, interacting system. They give no confirmation to the existence of the local magnetic moments which have been invoked to explain the negative magnetoresistance in n-type heavily doped semiconductors, but can also not rule out the existence of such moments.

I. INTRODUCTION

Heavily doped silicon and germanium have been extensively investigated in recent years. One motivation has been interest in the characteristics of the insulator-metal transition in disordered systems in which the transition is controlled by impurity doping. Mott' has given a recent synthesis of the state of interpretation of observations, in which he includes an extensive bibliography of recent work. An earlier review article by Alexander and Holcomb² gives a listing of additional papers.

In this paper, we focus upon the magnetic properties of the conduction electrons in Si:P and Si:8 for impurity concentrations from 5.4×10^{18} to 8.0×10^{19} cm⁻³. We report new NMR measurements of properties of the P^{31} spin system in Si:P and of the $B¹¹$ spin system in Si:B over the impurity concentration range noted. Knight shifts, line shapes, linewidths, and relaxation times are found to be determined by interaction of the nuclear moments with the conduction electrons. The most interesting feature of the data is a temperature dependence of linewidth and of the Knight shift K for temperatures at and below 4.2 K, for concentrations at and below 3×10^{19} cm⁻³. The experimental data complements new Si²⁹ data of Sasaki, Ikehata, and Kobayashi.³ Both sets of data extend the data and interpretations of earlier work by Sundfors and Holcomb⁴(abbreviated SH in this paper) and of Carver et $al.^5$

The temperature dependence of K is found to

parallel the temperature dependence of the electron spin susceptibility χ_s reported by Quirt and $Marko⁶$ and by Ue and Maekawa.⁷ The NMR data give no direct evidence for the existence of localized magnetic moments, which have been invoked by several authors (e.g., Toyozawa'; Yamanouchi, Mizuguchi, and Sasaki⁹; Khosla and Fischer¹⁰; Yugo¹¹) to interpret the negative magnetoresistance observed in a number of n -type heavily doped semiconductors.

Comparison of the NMR data with data with the spin susceptibility χ_s shows that changes in K and linewidth with temperature and concentration can be ascribed largely to changes in χ_s . This feature of the NMR results leads naturally to a model in which the spin susceptibility is taken to measure a single value of $\langle S_z \rangle$, the expectation value of the component of electron spin parallel to the magnetic field, appropriate to each electron in the system. In other words, for samples of Si:P and Si:B with impurity concentration n_r greater than 5×10^{18} cm⁻³, the impurity electrons are taken to be entirely delocalized and to be interacting members of a single-electron or hole system.

Detailed comparison of this model with the data shows that, while it describes the general trends of the data, it is not accurate in detail. Interpretation is complicated by lack of knowledge of the dependence of the local hyperfine coupling constant upon local and average impurity concentration.

Our interpretation of the NMR data is, in a general way, consistent with the point of view

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taken by Mott in Sec. I of Ref. 1. However, it should be emphasized that our analysis does not invoke any particular microscopic model for the nature of the interactions of the conduction electrons with one another.

In Sec. II of this paper, we develop the basic framework through which we use the NMR experiments to study the magnetic behavior of the conduction electrons. Section III gives our experimental results. In Sec. IV, we examine the implications of the NMR results, in terms of the model outlined in the previous paragraphs.

II. BACKGROUND INFORMATION PERTINENT TO NMR DATA

In earlier studies of the P^{31} NMR properties in $Si:P$ and of $B¹¹$ in $Si:B$, SH showed that, in the more heavily doped samples, the NMR properties were determined by interaction with the conduction electrons. For the case of $P³¹$, they argued that the NMR absorption line shape in these samples is consistent with the presumption that it is an inhomogeneously broadened line, with the shape determined by a distribution of values of K , the Knight shift, for the P^{31} nuclei. In this paper, we shall exploit the fact that the NMR properties are largely determined by interaction of the nuclei with the conduction electrons. In this section we shall develop the basic equations which relate the NMR properties of the nuclei to the conductionelectron properties, with particular attention to the question of how these relations may need to be modified as one goes from the case of an ordered metal crystal to the disordered atomic distribution appropriate to the heavily doped semiconductors.

The basic relations are those for the fractional resonance field shift K and the spin-lattice relaxation time T_1 . We first write K as

$$
K = \frac{8}{3}\pi \chi_e \langle |u_k(0)|^2 \rangle_{E_{\rm m}}, \tag{1}
$$

where χ_e is the electron spin susceptibility of the metal, and $\langle \, | u_{\pmb{\ast}}(0) |^2 \, \rangle_{\pmb{E}_{_{\bm{F}}}}$ is the electron-wave-fun tion probability density evaluated at the nuclea site and averaged over all electrons at the Fermi energy $E_{\boldsymbol{F}}$. The wave function is normalized so that the integral of the probability density over unit volume is unity.

Equation (1) is appropriate for the case in which the shift is determined by electrons in wave functions of s-type symmetry.

Equation (1) may also be written as

$$
K = \frac{8}{3}\pi \chi_e \Omega_0 \langle \psi_k(0) |^2 \rangle_{E_F}, \qquad (2)
$$

where Ω_0 is the volume per atom of the metal, and the new wave function ψ is normalized so that the

integral of the probability density over the volume of one atom is unity. In order to discuss the situation in a disordered meta1, we decompose the $\text{expression for }\chi_{_{\boldsymbol{\theta}}}, \text{ and write}$

$$
K = \frac{8}{3}\pi g \mu_B(\langle S_z \rangle / H_0) N \Omega_0 \langle |\psi_k(0)|^2 \rangle_{E_m}, \tag{3}
$$

where $\langle S_z \rangle$ is the mean value of the spin component S, for electrons at the Fermi energy, H_0 is the applied magnetic field, and N is the number of electrons per unit volume. g is the electron g factor, and μ_B the Bohr magneton. We make the further abbreviation

$$
\langle |\psi_{k}(0)|^{2} \rangle_{E_{F}} \equiv P_{F}
$$

In a metal such as Na, with all nuclear sites equivalent and with one free electron per atom, the product $N\Omega_0 = 1$. $\langle S_z \rangle$ is an intrinsic property of the electron system. We note that for Na, P_F is very close to the value of the probability density at the nucleus in the free atom, P_A . Experiments of Ryter¹² give the value of the ratio P_F/P_A for Na as 0.635 ± 0.030 .

If we decrease the atomic volume in a simple metal such as Na by applying pressure, the value metal such as Na by applying pressure, the value
of K rises.¹³ Since we expect $\langle S_g \rangle$ to decrease with decreasing volume, because of stronger Pauliexclusion-principle effects, this behavior must arise from increases in P_F .

Now, let us consider the situation for the "atoms" of a metal such as $Si:P$, namely the P^{31} impurities. If the conduction-electron system is still composed of fully delocalized electrons, it will remain true that $\langle S_z \rangle$ has a single value at all nuclear sites —a value characteristic of the electron system. N and Ω_0 have values which may vary from site to site, and it is no longer obvious that they always cancel one another, since the appropriate sampling volumes for averaging to obtain appropriate local values of the two quantities may not be identical. We expect P_F to vary from P^{31} site to P^{31} site.

Because of the complicated nature of the variation of the four quantities in Eq. (3), interpretation of the Knight-shift distribution observed in a disordered material is difficult. It could even be true that $\langle S_z \rangle$ does not have a unique value for all nuclei. We shall return to these complications in Sec. IV.

[We note that SH incorrectly omitted the factor Ω_0 in writing the expression similar to Eq. (3).]

Korringa¹⁴ noted that in a simple metal the spinlattice relaxation time for the nuclei wi11 commonly be determined by the off-diagonal matrix elements of the same hyperfine interaction term whose diagonal elements give K . As a result of this connection, one can write the expression

$$
\frac{4\pi k}{\hbar} \left(\frac{\gamma_n}{\gamma_e}\right)^2 K^2 T_1 T = 1, \tag{4}
$$

for those cases in which one has no electron correlation effects, and simple s-state wave functions. Equation (4) is the "Korringa relation, " and we shall refer to the left-hand side of it as the "Korringa product. "

We note that there are two situations which may lead to a deviation from Eq. (4) . (i) If there are electron correlation effects, one may expect a selective enhancement of K , and the value of the Korringa product will rise above unity. (ii) A second possible deviation from the Korringa relation, less thoroughly explored, may arise when electrons are very strongly scattered. Warren¹⁵ has discussed this effect. Strong scattering may lead to a longer dwell time for the electrons in any particular region of the lattice, and a resulting shortening of T_1 . The Warren effect decreases the value of the Korringa product-a deviation in the opposite direction to that produced by the correlation effects.

III. EXPERIMENTAL TECHNIQUES AND RESULTS

A. Samples

The Si:P samples were commercially prepared by Ventron, Inc. Phosphorous concentrations were determined by comparing room-temperature resistivity values supplied by the manufacturer and checked in our laboratory with the resistivity checked in our laboratory with the resistivity
curves of Irvin.¹⁶ Approximate values of the impurity concentration n_r may also be calculated from the room-temperature Hall coefficient. (For n_I in the vicinity of 10¹⁹ cm⁻³, discrepancies of the order of 30% may exist between the values of n_I obtained by these two techniques.) We find the argument of Quirt and Marko' in favor of using the Irvin curves to be convincing.

^a See text for discussion of accuracy of quoted concentrations for Si: P.

The Si:B samples listed in Table I are those de-The Si:B samples listed in Table I are those d
scribed by Sundfors.¹⁷ Boron concentrations for these samples were also determined by use of the Irvin curves. Table I lists the silicon samples and the appropriate impurity concentrations. For samples V-1 through V-4, n_r may vary by 10% from the value given. This error in n_I reflects the spread in resistivity values obtained from several measurements on a given sample. Such a spread is to be expected because many samples have been prepared from crystals with a substantial variation in n_r from one side to the other. For such samples, the quoted value of n_i is an average value.

B. Experimental techniques

The experiments were performed using a phasecoherent pulsed NMR spectrometer designed by coherent pulsed NMR spectrometer designed by
Clark.¹⁸ Modifications of the original design have been described by several authors.^{17, 19} The spectrometer was operated at 8.5 and 16 MHz for these experiments. For P^{31} in Si:P, measurements were made utilizing spin echoes produced by the usual 90° - τ -180° pulse sequence. Detection pf the echoes was made possible by signal averaging with a single-channel boxcar integrator. T, was measured by varying the pulse spacing τ , and T , was measured by varying the repetition interval of the rf pulse sequence. The P^{31} linewidth was determined by integration of the echo signal while sweeping the external magnetic field through resonance —^a procedure which reproduces the absorption line shape $\chi^{\prime\prime\prime}(H_0)$. This technique has been described by Narath.²⁰ The Knight shift was obtained by comparison with the $P³¹$ resonance in the reference material, GaP.

For $B¹¹$ in Si:B, data collection is more difficult because of the long spin-lattice relaxation time. It is 8 min at $1.3 \text{ }^{\circ}\text{K}$, or about a factor of 30 longer than typical values for P^{31} . Because holding-time limitations restrict the use of the boxcar integrator in this regime, a 1024-channel Fabritek signal averager was used. The $B¹¹$ linewidths were determined by measuring the lifetime of the freeinduction decay, T_2^* , and using the relation ΔH = $2/\gamma T_{2}^{*}$, where γ is the gyromagnetic ratio.

C. Experimental results

Typical P^{31} absorption lines are shown in Fig. 1 for values of n_p of 8×10^{19} and 7.5×10^{18} cm⁻³. The resonances were observed at 1.3 °K at a frequency of 16 MHz. The asymmetrical lineshape has been previously reported by SH for slightly higher P^{31} concentrations and is also characteristic

of the Si^{29} resonance observed by Sasaki et al.³

The full width at half-maximum, ΔH , of the absorption line is given by H_b-H_a , where H_b and H_a are the distances in field from the $P³¹$ reference resonance to the low- and high-field halfmaxima of the line, respectively. Figure 2 plots values of H_a and H_b for the P³¹ resonance for temperatures of 1.3 and 4.2 °K and frequencies of 8.5 and 16 MHz. Three important features of the data are indicated: temperature dependence, field dependence, and concentration dependence. H_a is insensitive to temperature, but H_b becomes temperature dependent for the low-concentration samples. The resulting temperature dependence of the linewidth is comparable to that observed by Sasaki et al., for the Si^{29} resonance in samples of similar concentration. The field dependence of the line shape is clarified by Fig. 3, which plots the normalized quantities H_a/H_0 and H_b/H_0 , where H_0 is the external resonant magnetic field. A linear dependence of H_a and H_b (and hence of ΔH) on H_0 is shown by the coincidence of data taken with different values of H_0 . Figure 2 also shows that the linewidth is increasing rapidly with decreasing n_p . Figure 4 shows that a similar behavior is found for the B^{11} linewidth in Si:B.

Ne have also determined the peak and center-ofgravity Knight shifts, K_0 and $\langle K \rangle$, for the P³¹ reso-

nance. Figure 5 shows K_0 and $\langle K \rangle$ as a function of n_p for 1.3 and 4.2 °K. The 16- and 8.5-MHz values have been combined to obtain the values plotted. Behavior similar to that of the linewidth is observed. Both K_0 and $\langle K \rangle$ increase with decreasing n_p . A temperature dependence is also seen in $\langle K \rangle$, but not so clearly in K_0 . Measurements by SH of K_0 for the B¹¹ resonance in Si:B indicated that, for $n_A = 2.1 \times 10^{19}$ cm⁻³ and $n_A = 8.5 \times 10^{19}$ cm⁻³, K_0 is identical. Again, this behavior of K_0 is consistent with the $B¹¹$ linewidth results for the same range of concentration.

Although we have not made extensive measurements of T_1 and T_2 for the Si:P samples, a few data were obtained and Table II shows these results. In contrast to the sharp concentration dependence of the linewidth and K , T_1 and T_2 appear to be approximately independent of n_p . Measurements of T_1 for B^{11} in Si:B by SH showed a cor-

FIG. 1. NMR absorption lines for P^{31} in Si:P at the two extremes of the P concentration range used in this work. Signals were recorded by integrating the spinecho signal as the external field was swept through resonance. The reference zero for Knight-shift measurements, the P^{31} resonance in crystalline GaP, is marked.

FIG. 2. Linewidth data for P^{31} NMR in Si:P, as a function of donor concentration \boldsymbol{n}_D for two temperatures and two frequencies. H_b and H_a are the magnetic field intervals from position of the GaP reference line to the field positions of low-field half-height and high-field half-height, respectively.

responding independence of n_A for the range noted in the previous paragraph.

IV. INTERPRETATION OF MAGNETIC-RESONANCE RESULTS

First, we summarize what seem to us to be the most significant features of our results for Si:P and Si:B.

A. Si:P

(i) The linewidth and shape of the P^{31} resonance in Si:P is determined by inhomogeneous broadening at all values of n_p . The echo T_2 , with a value of about 7 msec at all values of n_p and T, gives a homogeneous broadening of only O.OI G. The Knight-shift distribution model of Ref. 4 appears to correctly describe the line shape.

(ii) For impurity concentrations below 8×10^{19} cm⁻³, $\langle K \rangle$ and the linewidth increase as n_p decreases, and both become temperature dependent.

(iii) For P^{31} in Si:P, the data of Figs. 1-5 exhibit several characteristics. The line shape depends in a minor way upon concentration and temperature. The small change in shape is evident in the data of Fig. 5, in which it is seen that the ratio of K_0 to $\langle K \rangle$ varies slightly with concentration and temperature.

In seeking an organizing principle for the data, we refer to Eq. (3). The experimental line shape at each value of n_D and T is determined by the distribution of values of K at the various P^{31} sites. Let us assume that the form of Eq. (3) is a valid expression for K at each nucleus, even though there may be different local values for all quan-

FIG. 3. Data of Fig. 2 for $T = 1.3$ K, replotted with the normalized variable H_a/H_0 and H_b/H_0 .

tities, N, Ω_0 , $\langle S_z \rangle$, and P_F . If Eq. (3) provides a correct description, then any temperature dependence must reside in the electronic property, $\langle S_{n} \rangle$. (We assume that we are at temperatures sufficiently low such that there is no appreciable thermal excitation of higher-energy translational states for the electrons. Such a thermal excita-
tion could alter P_F .)
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Seeking the simplest interpretation of the NMR data, one would further assume that all conduction electrons are in sufficiently strong magnetic interaction with one another that there is a single value of $\langle S_z \rangle$ appropriate to each electron. Two consequences follow.

(i) For a sample at a given value of n_I , the line shape should be independent of field and temper ature,²¹ and the linewidth should scale linearly ature, $^{\mathsf{21}}$ and the linewidth should scale linearl with $\langle K \rangle$. Figures 3 and 6, which correlate values of $\langle K \rangle$ and $\Delta H/H_0$, show that the experimental data support this conclusion.

(ii) For a sample at a given value of n_I , the temperature dependence of $\langle K \rangle$ should mirror the temperature dependence of χ_s . For Si:P, ESR data of Quirt and Marko and of Ue and Maekawa permit a direct test of this conclusion. Comparison of the data of Fig. 5 with the data of Fig. 1 of Ref. 6(a) and of Fig. 3 of Ref. 7 shows that the temperature dependence of $\langle K \rangle$ is somewhat sharper than that for the χ _s data of Quirt and Marko and somewhat less sharp than the temperature dependence of χ_s found by Ue and Maekawa. We judge the data of Quirt and Marko to be somewhat more reliable. If it is accurate, then the simple model is roughly correct, but not precisely so.

FIG. 4. Linewidth data for P^{31} in Si:P and B¹¹ in Si:B, as a function of impurity concentration n_I . ΔH is the full width at half-maximum. For B^{11} , T_2 is measured from the free induction decay, and $\Delta H = 2/\gamma T_2$.

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The recent NMR data of Sasaki et al. on the $Si²⁹$ resonance in samples covering the same concentration range as ours show a behavior which appears to us to be in good agreement with the $P³¹$ data given here. There is a certain complication in a direct, quantitative comparison of the two sets of NMR results because of the fact that the concentration dependence of P_F is different for the two nuclear systems. The data for Si^{29} with $n_D > 3$ $\times 10^{19}$ cm⁻³ shows that the product $\Omega_0 P_F$ is approximately independent of n_D , as noted previously by SH, whereas data of Table III suggest that this 'product is more nearly proportional to $n_D^{-2/3}$ for the P^{31} system. Unfortunately, calculation of the expected dependence of this product upon n_p is not straightforward. The problem is somewhat analogous to that of calculating the pressure dependence of K in crystalline metals, a difficult probdence of K in crystalline metals, a difficult pr
lem. (See Benedek and Kushida,¹³ for example who describe the situation in the alkali metals.)

Our conclusion is that the magnetic-resonance data, both ESR and NMR, conform fairly well to the model suggested by Eq. (3) in which spin susceptibility and $\langle K \rangle$ are directly connected by a single constant for a given sample. The agreement with the model is not perfect, as evidenced by the slight temperature dependence of the line shape in a given sample.

Quirt and Marko analyzed their data on the basis of a rather different model in which the electrons were assigned to two classes—those responsible

FIG. 5. Knight shifts for P^{31} in Si:P, as a function of donor concentration n_D . $K₀$ is the shift to the peak of the line; $\langle K \rangle$ is the shift measured to the center of gravity of the absorption line. Values of the shifts at two temperatures are shown. Values of shifts are averages of data at 8.5 and 16 MHz.

Values from Ref. 5.

 b Using values of T_1 from Ref. 5 plus K_0 and line-shape data from Ref. 4 to obtain $\langle K \rangle$.

for a temperature-independent Pauli susceptibility, and those which generated a Curie-Weiss susceptibility. We see no evidence of such differentiation in the NMR data. Such a differentiation should lead to a substantial change in line shape for the more dilute samples when the temperaturedependent Curie-Weiss electrcas come to dominate broadening of the NMB line at low temperature. The NMR data lead more naturally to the conclusion that the susceptibility of the electron system arises from a single system of interacting electrons. Quirt and Marko point out that the temperature dependence of χ_s cannot be correctly described by modifying the Pauli expression to take into account the effect of nonzero temperature. Mott' observes, however, that the temperature dependence is consistent with that which results from calculations of susceptibility of a highly correlated electron gas or a "degenerate gas of spin polar ons. "

The NMB results do not appear to provide sub-

FIG. 6. Correlation of measured values of $\langle K \rangle$ at two temperatures with values of normalized linewidth $\Delta H/H_0$ measured at the same temperature. for four samples. The data are plotted in this fashion to test for direct proportionality of the two quantities.

$\begin{array}{c} n_D \\ (10^{19} \text{ cm}^{-3}) \end{array}$					Wave functions at 4.2 K	
	$10^4 \langle K \rangle$		χ_{s} (10 ⁸ cgs/vol)		P_F^{c}	
	4.2 K	1.3K	4.2 K	1.1 K	$\Omega_0 P_F$ ^b	$(10^{24} \text{ cm}^{-3})$
8.0	27.4 ± 2.4	32.6 ± 1.3	9.3	9.8	3400	0.28 ± 0.04
3.2	37.0 ± 2.5	41.7 ± 1.6	7.0	7.5	6200	0.20 ± 0.02
1.9	41.5 ± 4.7	52.2 ± 3.0	5.8	6.3	8500	0.16 ± 0.02
0.75	$(52.9 \pm 9.7)^{a}$	71.3 ± 7.0	$(6.9)^{a}$	7.2		

TABLE III. Tabulation of values of $\langle K \rangle$ in Si: P and values of P_F derived by use of ESR values of χ_s . (Values of χ_s are taken from Ref. 6. Values from Ref. 7 would lead to a sharper dependence of P_F on n_n .)

 a Value at 1.85 K.

Derived from the relationship $\langle K \rangle = \frac{8}{3} \pi (x_s)_{\text{ESR}} \Omega_0 P_F.$

^c Derived from previous column by setting $\Omega_0 = 1/n_D$.

stantial support for the "local-moment" model which has been used in interpretation of the negative magnetoresistance. Recent work by Walstedt and Walker²² on NMR line broadening by magnetic impurities suggests that one would expect lineshape changes to appear in the semiconductors if the broadening did indeed arise from such localized moments. Although there are minor changes in shape, they do not appear to be significant. On the other hand, the NMR results certainly do not rule out the local-moment model.

B. Si:B

Figure 4 shows that the $B¹¹$ NMR line in Si:B broadens over the same impurity concentration range as does the P^{31} line in Si:P. It seems natural to assume that the same mechanism is operating for both crystals. However, it should be noted that the magnetoresistance behaves rather differently in the p -type materials than in n-type.²³ Connections between spin-resonance and magnetoresistance data remain clouded. An alternate model for the explanation of the negative magnetoresistance has been proposed, 24 but there appears to be no definitive test of its validity available at this time.

C. Relaxation time and the Korringa relation

Table II gives a few values of the relaxation time T_1 for the P^{31} system. Unfortunately, the precision is low in the low-concentration samples. Within the rather large experimental uncertainty, we note that the T_1 values do not decrease at low

 n_p , as they should if the Korringa relation, Eq. (4), were correct. In other words, the value of the Korringa product rises above unity, signaling electron correlation effects on K. Sasaki et al. have concluded that their Si²⁹ data for K and T_1 show values of K enhanced by correlation effects. The Sasaki et al. data, however, are in some disagreement with the earlier SH data for K and T , in the Si^{29} system. The SH data do not support the hypothesis of electron correlation enhancement of K , since it generates a value of the Korringa product which drops away from unity, rather than increasing above unity. Since the Sasaki values of $T₁$ are longer, one may tend to favor them on the presumption that there might have been impurity contributions to T_1 in the SH samples. However, other than a general argument of that sort, there seems no certain way of selecting which of the sets of Si^{29} data is more correct.

Thus, because of a combination of low precision and some disagreement among various determinations of the Korringa product, we conclude that better measurements of $T₁$ are required before one can draw more than speculative conclusions about the visibility of electron correlation effects upon values of K .

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