interaction cannot be ignored.

A final comment concerns the undesirably large error limits of the values $\Delta V_{\text{expt}}$ listed in Table III and shown in Fig. 4. A hint of an explanation is contained in Fig. 3, where it can be seen that the deviations of the data points from the least-squares line are nonrandom. Physically, this phenomenon may be a consequence of the fact that the activated state changes its position in configurational space as a function of pressure, i.e., when the interatomic spacing is changed. In iron-nickel alloys with their competing ferromagnetic and antiferromagnetic interactions[18] this is possible. Thus, the large error limits of $\Delta V_{\text{expt}}$ may merely reflect the force fitting of the data which was prompted by the simple model used to interpret the data.

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# Domain and Wall Hyperfine Fields in Ferromagnetic Iron

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The Mössbauer effect and nuclear magnetic resonance are used to examine the temperature dependence of the domain and wall hyperfine fields in ferromagnetic iron. The difference between them is attributed to demagnetizing fields and exhibits a temperature dependence different than predicted from consideration of domain and wall spin-wave excitations. It is proposed that this temperature dependence arises from distortion of domain shapes with temperature. After correction to constant volume, the temperature dependence of the hyperfine field is compared with that of new magnetization measurements. It is shown that an intrinsic temperature dependence of the effective hyperfine coupling constant exists and may be qualitatively explained by three mechanisms: (i) phonon admixture of the *s* and *d* wave functions, (ii) Stoner-like excitations combined with a strongly energy-dependent hyperfine coupling constant, and (iii) changes in the intrinsic *s*-*d* hybridization due to the changing magnetization. It is concluded that differentiation of the three mechanisms is not possible by the present type of experiment but requires reliable theoretical estimates of the magnitudes of the various effects.

## I. INTRODUCTION

While techniques for measuring hyperfine fields have long been used to study the ferromagnetic metals, the interpretation of the dependence of hyperfine fields on thermodynamic variables and the relation of such fields to the bulk magnetization is complicated by numerous factors: (i) Most experiments are performed at constant pressure, so that the data must be corrected to constant volume before a meaningful comparison with theory may be made. (ii) The effective hyperfine coupling constant exhibits an intrinsic dependence on the thermodynamic variables. (iii) In nuclear magnetic resonance (NMR) experiments without an applied field the wall resonance signal is so greatly enhanced[1] that the results indicate properties of the walls rather than those of the bulk ferromagnet. The first and especially the second complicating factor, with a few notable exceptions,[2] have been largely ignored in the past. While attempts have been made to circumvent the third problem by examining single-domain particles,[3] the extremely small size (~100 Å) of such particles introduces surface effects and lattice expansion as further complicating factors.

In this paper we attempt to obtain a coherent picture of the hyperfine fields in a common ferromagnet, iron metal, while taking into account the above-mentioned difficulties. We have used NMR to examine the properties of the wall and Mössbauer effect (ME) to examine those of the domain. We have also made bulk magnetization measurements, corrected them for thermal expansion, and compared them to the hyperfine field measurements in order to determine the intrinsic temperature dependence of the hyperfine coupling constant. All data were taken on samples prepared from the same starting material, eliminating any possible systematic effects due to different impurity content. Parts of this work are similar to that reported earlier by Benedek and Armstrong,[2] who relied on fixed-field magnetization data from the literature.

## II. EXPERIMENTAL

The experiments were performed on high-purity polycrystalline iron obtained from K. M. Olsen of this laboratory. The total impurity concentration, including oxygen and carbon, was below 100 ppm. The NMR and ME samples were prepared by filing the iron and annealing the filings for 48 h at 600 °C in a dry hydrogen atmosphere. The magnetization measurements were made on a cylindrical sample, 0.030 in. in diameter by 0.150 in. long, which had been similarly heat treated.

The NMR measurements used a conventional marginal oscillator[4] with frequency modulation and first derivative detection. The sample was suspended in a high-temperature oil and heated by a noninductively wound oven. The temperature, measured by a copper-constantan thermocouple embedded in the sample, was known to ± 0.5 °K. The peak of the $Fe^{57}$ resonance was used to determine the hyperfine field. However, since the line was asymmetrical with a width of ~15 kHz and since a detailed study of the line shape was not performed, we feel the results have an accuracy of ± 10 kHz. While the use of fast passage would provide greatly improved accuracy,[5] this was not necessary for our purposes. The results, along with some lower-temperature data included for completeness, are shown in Table I. We should mention here that there is agreement among the various low-temperature NMR measurements[6,7] in Fe and that we are, in general, in agreement with the more extensive measurements of Budnick, Bruner, Blume, and Boyd,[8] except at the highest temperatures.

The ME spectrometer was a conventional constant acceleration spectrometer using a $Co^{57}$ in palladium source. Iron filings were sandwiched between Mylar sheets to form the absorber. The source-to-detector distance was kept constant for all the experiments. Low-temperature data were taken with the sample cooled by liquid He, $H_2$, or $N_2$; high-temperature data, with a vacuum furnace containing a small amount of argon for improved heat transfer. The data were analyzed by fitting four independent Lorentzian lines to the inner four lines of the spectrum. From these, two independent values for the ground-state splitting were obtained. These were generally consistent to 3 parts in $10^4$, which is about the uncertainty of the line positions indicated by the least-squares procedure.

TABLE I. ME and NMR measurements of the hyperfine field in Fe at constant pressure.

| Temperature (°K) | Hyperfine field (MHz) ME | NMR |
|---|---|---|
| 1.5 | ... | 46.65[a] |
| 4.2 | 46.71 | 46.65[a] |
| 14.0 | ... | 46.64[a] |
| 20.3 | 46.75 | 46.64[a] |
| 50 | ... | 46.59[a] |
| 77 | 46.62 | 46.54 |
| 100 | ... | 46.48[a] |
| 151 | 46.41 | ... |
| 195 | ... | 46.10[b] |
| 273 | ... | 45.64 |
| 298 | 45.50 | 45.46 |
| 317 | ... | 45.32 |
| 336 | ... | 45.16 |
| 352 | ... | 45.02 |
| 372 | ... | 44.84 |
| 374 | 44.93 | ... |
| 399 | ... | 44.59 |
| 418 | ... | 44.39 |
| 424 | 44.37 | ... |
| 443 | ... | 44.10 |
| 463 | ... | 43.91 |
| 473 | 43.81 | ... |
| 475 | ... | 43.72 |
| 493 | ... | 43.52 |
| 517 | ... | 43.14 |
| 522 | 43.14 | ... |
| 535 | ... | 42.88 |
| 555 | ... | 42.58 |

[a] Reference 6. [b] Reference 7.

TABLE II. Fe magnetization at constant pressure.

| Temperature (°K) | $(10^2)\Delta M/M$ |
|---|---|
| 77 | $0.15 \pm 0.10$ |
| 100 | 0.34 |
| 125 | 0.39 |
| 150 | 0.61 |
| 175 | 0.75 |
| 200 | 1.01 |
| 225 | 1.23 |
| 250 | 1.46 |
| 273 | 1.84 |
| 297 | 2.10 |
| 323 | 2.37 |
| 348 | 2.61 |
| 373 | 3.11 |
| 398 | 3.50 |

Using the ground-state splitting of an iron foil[9] at $(24.1 \pm 0.5)$ °C for velocity calibration and the wavelength of the ME $\gamma$ ray,[10] we are then able to express the ground-state splittings in frequency units. These results are shown in Table I.

The magnetization measurements were obtained with a PAR model FM-1 vibrating sample magnetometer. The temperature was stabilized using a feedback system and measured to $\pm 0.5$ °K. Determination of the spontaneous magnetization at each temperature posed somewhat of a problem. It is a well-known fact that the empirical relationship $M_H = M_S(T)(1 - a/H_i - b/H_i^2) + cH_i$, where $H_i$ is the internal field, adequately describes the field dependence of the magnetization for not too small a field. The last term is just the intrinsic susceptibility of the material and is small enough to be neglected. The $1/H_i^2$ term has been related to the anisotropy and magnetostriction of the material,[11] while the origin of the $1/H_i$ term remains somewhat vague. Thus probably the constant $a$ and most definitely $b$ are temperature dependent. For this reason magnetization measurements at a fixed field as a function of temperature, as are commonly reported, undoubtedly include other temperature dependences than that intrinsic to the magnetization. Our measurements were therefore analyzed by examining the data at each temperature, both as a function of $1/H_i$ and as a function of $1/H_i^2$, and extrapolating the linear portions to infinite field by a least-squares fit. The two sets of extrapolated magnetization measurements were then examined on a $T^{3/2}$ plot and fitted onto the accurate low-temperature data of Argyle, Charap, and Pugh.[12] This enabled us to obtain accurate values for the spontaneous magnetization. Using these values the deviations from saturation as a function of temperature were calculated. As the deviation from saturation for the $1/H_i$ fit and the $1/H_i^2$ fit agreed within experimental error, an average of the two values at each temperature was taken. The results are shown in Table II.

## III. DISCUSSION

Since all our experiments are done at constant pressure, while the temperature dependences of the measured quantities may be meaningfully compared only at constant volume, it is necessary to convert the data to a constant-volume basis. This may be accomplished using the following thermodynamic relationship:

$$\left(\frac{1}{F}\frac{\partial F}{\partial T}\right)_V = \left(\frac{1}{F}\frac{\partial F}{\partial T}\right)_P - \left(\frac{1}{F}\frac{\partial F}{\partial P}\right)_T \times \left(\frac{1}{V}\frac{\partial V}{\partial P}\right)_T^{-1} \left(\frac{1}{V}\frac{\partial V}{\partial T}\right)_P , \tag{1}$$

where $F$ is the quantity in question, $[(1/F)\partial F/\partial P]_T$ is its pressure dependence, $[(1/V)\partial V/\partial P]_T$ is the compressibility, and $[(1/V)\partial V/\partial T]_P$ is the thermal expansion. At this point we must ask whether it is justifiable to use the bulk macroscopic measurements of compressibility and thermal expansion when considering microscopic measurements such as the hyperfine field. In a ferromagnet this is probably a reasonable procedure when we are considering domains, which account for a major fraction of the sample volume. However, in domain walls, compressibility and expansivity may be radically different from the bulk values if the magnetic energy plays a significant role. Measurements of compressibility and expansivity within domain walls are not available at present. For Fe metal, which is cubic, the anisotropy is small and thus the walls, whose thickness goes as $(J/K)^{1/2}$, the ratio of exchange to anisotropy, are very thick. Estimates of the thickness are in the range of several hundred lattice spacings.[13] Hence any small section of a wall looks very much like the domain, and it seems reasonable to expect their compressibility and thermal expansion to be similar. This argument is supported by the agreement between the pressure dependence of the hyperfine field as measured in the walls by NMR,[2] and in the domains by ME.[14] In this experiment we have therefore used the bulk values.

### A. Comparison of Domain and Wall Hyperfine Fields

We will consider first the difference between the hyperfine fields in the domain and the wall. As we have pointed out above that corrections to constant volume are the same for both quantities, we are justified in examining their differences at constant pressure. If one considers the NMR data to define a smooth curve then the difference between the ME data and this curve is shown in Fig. 1.

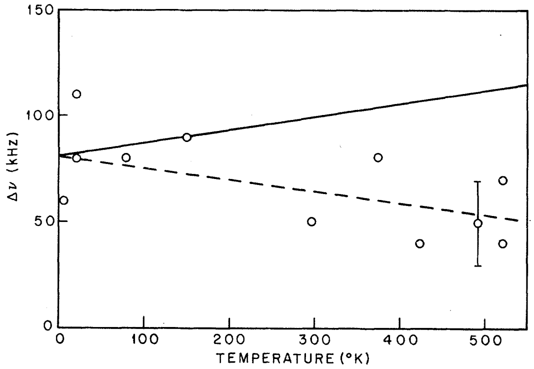

FIG. 1. The difference between the domain and wall hyperfine splittings in frequency units plotted as a function of temperature. The solid line is the expected temperature dependence due to wall excitations and the dashed line is a least-squares fit of a straight line to the data. The error bar shown is characteristic of all the data.

While there is sizable scatter, the domain hyperfine field is larger than that of the wall. One possible explanation of this is that nuclei in walls do not see demagnetizing fields while those in a domain do. It is expected that for cubic materials such as Fe the domains would form configurations which include closure domains, and therefore no domain demagnetizing fields would be present. However, we are examining polycrystalline samples in which it is known[13] that "free magnetic poles" may exist on grain boundaries. Such magnetic charge would produce small domain demagnetizing fields. These demagnetizing fields would not be uniform throughout the domains and would lead to a distribution of fields at the nuclear sites. Some evidence for this effect can be found in the width of the outer lines of the spectra of iron foils, which are generally greater than can be accounted for by finite absorption effects. The measured difference between wall and domain hfs fields corresponds to a change of the Mössbauer splitting of 0.0008 cm/sec, i.e., 4% of the linewidth. It could, therefore, easily result from asymmetrical broadening (this effect may set a limit to the precision which can be obtained in the use of metallic iron as a velocity calibration standard).

Keeping the above reservations about the interpretation of the ME data in mind, let us now consider what temperature-dependent effects may be expected in the difference between the domain and wall hyperfine fields. First, there is a small temperature dependence due to the fact that the demagnetizing field is proportional to the magnetization. This is a small effect and unobservable with our present accuracy. Second, we would also expect the magnetization in the wall to decrease faster than in the domain owing to the existence of wall excitations, as predicted by Winter.[15] This dependence as calculated in the long-wavelength approximation[15] is shown as the solid line in Fig. 1. The dashed line is a least-squares fit of a straight line to the data. It appears that the calculated dependence does not adequately describe the data. Whether this discrepancy is due to problems with the theory or to other temperature-dependent effects is difficult to say. However, consider the following additional source of temperature dependence which may be of importance. The demagnetizing fields depend on the domain shapes. Since the domain configurations result from an attempt to minimize the magnetic energy, it is possible that the domains could change shape and orientation as a function of temperature. This would not only alter the main value of the demagnetizing field but would be expected to change the distribution of demagnetizing fields. This mechanism may account for the discrepancy between the predicted and observed temperature dependence of the domain-wall hyperfine field difference.

### B. Comparison of Magnetizing and Hyperfine Field Measurements

It is of interest to investigate whether the temperature dependence of the hyperfine field accurately tracks that of the magnetization, as it does in many insulating magnetic materials. In order to do this, both quantities must be corrected to constant volume. Since the magnetic ordering temperature in Fe is greater than the Debye temperature, thermal expansion effects are important. Integrating Eq. (1) with respect to temperature we obtain

$$F_V = F_P \exp\left[-\int_0^T \left(\frac{\partial \ln F}{\partial P}\right)_T \left(\frac{\partial \ln V}{\partial P}\right)_T^{-1} \left(\frac{\partial \ln V}{\partial T}\right)_P dT\right]. \tag{2}$$

It has been found that the pressure dependence of both the hyperfine field and the magnetization are relatively temperature independent.[3,16] Similarly, the compressibility is almost temperature independent,[17] and therefore we may rewrite Eq. (2) as

$$F_V = F_P \exp\left[-\left(\frac{\partial \ln F}{\partial P}\right) \left(\frac{\partial \ln V}{\partial P}\right)^{-1} 3\left(\frac{\Delta l(T)}{l_0}\right)\right], \tag{3}$$

where $\Delta l(T)/l_0$ is the linear thermal expansion.[18] Using published values for the correction factor constants, we find, in qualitative agreement with previous results,[2] that even at constant volume the temperature dependences of the hyperfine field and the magnetization differ appreciably. However, the magnitude of the difference is approximately a factor of 2 smaller than that observed by Benedek and Armstrong.[2] The discrepancy is due to their use of magnetization data taken at fixed field.

It should be noted that here and henceforth we use the wall hyperfine field for comparison with the magnetization. While this avoids errors that would be introduced by the temperature dependence of the additional field observed in the domain, it does introduce a small systematic error due to the difference between domain and wall spin-wave spectra. However, Winter's estimates[15] indicate that this is a factor of 2–3 smaller than our indicated experimental errors.

The possible origins of the difference are many, and depend principally on the origin of the hyperfine field. For simplicity and in order to continue our discussion we will assume that the hyperfine field consists of two contributions: (i) a core polarization component due to localized *d* electrons and (ii) a contribution from bandlike electrons. However, one must always remember that this assumption may be a gross oversimplification. We then write

$$H_{\rm hf}(T)=A_{\rm local}(T)\,\sigma_{\rm local}(T)+A_{\rm itinerant}(T)\,\sigma_{\rm itinerant}(T) \qquad (4a)$$

and

$$\sigma_{\rm tot}(T)=\sigma_{\rm local}(T)+\sigma_{\rm itinerant}(T), \qquad (4b)$$

where the $A$'s and $\sigma$'s are the appropriate hyperfine coupling constants and magnetizations, respectively. One can see that if the two contributions to the magnetization have different temperature dependences, the temperature dependence of the hyperfine field will differ from that of the magnetization, even if the $A$'s are temperature independent, except in the special case when $A_{\rm local}=A_{\rm itinerant}$. Or one may argue that it is not reasonable for the magnetization to consist of two components which have different temperature dependences. For this situation we have

$$H_{\rm hf}(T)=\left[A_{\rm local}(T)\,\sigma_{\rm local}(o)+A_{\rm itinerant}(T)\,\sigma_{\rm itinerant}(o)\right]\frac{\sigma(T)}{\sigma(o)} \qquad (5)$$

and the difference between $H_{\rm hf}(T)$ and $\sigma(T)$ is a weighted measure of the temperature dependences of the various components of the hyperfine coupling constant.

In order to analyze our data we make the assumption that all components of the magnetization have the same temperature dependence. If a consistent interpretation of the data is then possible, we can feel confident within some limits that (i) the assumption has a basis in fact or (ii) one component dominates all others. The fractional change in $A\equiv H_{\rm hf}/\sigma$, the effective hyperfine coupling constant, is plotted in Fig. 2. Since calculations show[19] that the major contribution to the hyperfine field comes from polarization of the core electrons by the 3*d* electrons, we will concentrate on temperature-dependent effects involving the 3*d* electrons. By examining the possible mechanisms one at a time and comparing the temperature dependence of $A$ expected from each mechanism with experiment we hope to distinguish between the various mechanisms. A more realistic picture would involve contributions from all possible mechanisms and components of $A$. However, such an approach, with the magnitudes of the individual components being unknown, would have too many variables to provide a meaningful comparison with experiment.

Benedek and Armstrong[2] have proposed a mechanism to explain such a temperature dependent $A$ in Fe metal: thermal excitation of *d* electrons to states higher in the *d* band combined with a strongly energy-dependent hyperfine coupling constant resulting in a $T^2$ dependence for $A$. We see in Fig. 2 that a $T^2$ dependence due to these Stoner-like excitations fits the data quite well. However, since precise low-temperature magnetization measurements[12] have shown no evidence for single-particle excitations and the required energy dependence of $A$ is unreasonably large, we can conclude that this is not the dominant mechanism causing the temperature dependence of $A$. A different mechanism was proposed by Simanek and Orbach[20] to explain the temperature dependent $A$ of $S$-state ions in insulators. Here phonons admix a small amount of higher-lying wave functions with *s* character into the 3*d* wave functions. Since core polarization produces a hyperfine field opposite in sign to the contact interaction for the same spin direction, this reduces the total hyperfine field. It has been pointed

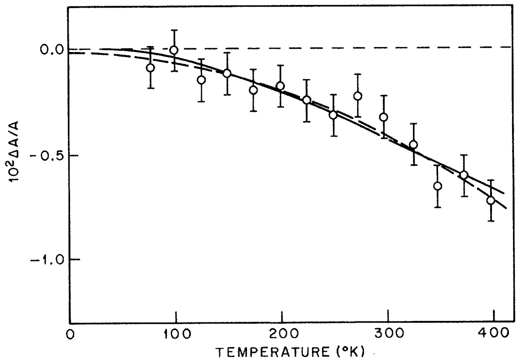

FIG. 2. The fractional change of the effective hyperfine coupling constant as a function of temperature. The solid line is a fit to the data for the *s-d* phonon admixture mechanism and the dashed line, a $T^2$ fit due to the Stoner-like excitation mechanism.

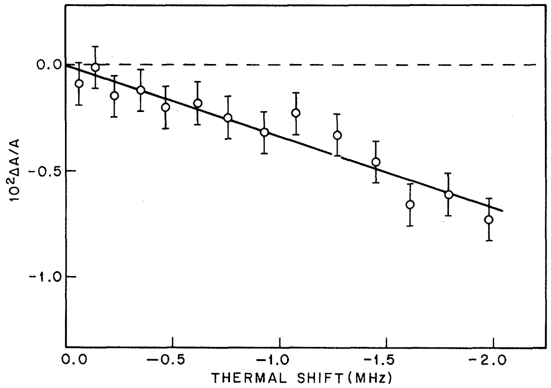

FIG. 3. The fractional change in the effective hyperfine coupling constant versus the ME thermal shift. The straight line is a least-squares fit to the data.

out that this mechanism should also be operative in the transition metals.[21] However, here the details of the effect are much more complicated since consideration must be given to shielding by the conduction electrons and the fact that the *s* and *d* bands overlap. These factors complicate the calculation of the magnitude of the effect but do not invalidate the basic idea of phonons distorting the lattice and thereby inducing changes in the amount of *s*-*d* admixture.

The net change in the hyperfine field is proportional to the mean-square strain $\langle\epsilon^2\rangle$ and, in the model of Simanek and Orbach, to the total lattice heat. Another quantity which follows this dependence is the thermal shift in the Mössbauer effect,[22] which arises through the second-order relativistic Doppler effect from the thermal motion of the atoms in the solid. In Fig. 3 we have plotted the change in $A$ versus our measurements of the thermal shift in Fe metal corrected to constant volume. The measured thermal shift is consistent with a Debye temperature of 436 °K. The magnitude of the phonon-induced *s*-*d* admixture is smaller than that observed for Mn in MgO.[23] Therefore the reasonable fit to the data by a straight line indicates that phonon *s*-*d* admixture also provides an adequate description of the temperature dependence of $A$. By replotting this fit on Fig. 2 we see that one cannot distinguish between the two mechanisms on the basis of the accuracy of the two fits.

The situation in transition metals is, however, inherently more complicated, as hybridization of the *s* and *d* bands exists to some extent even in the absence of lattice vibrations. Since the amount of this *s*-*d* mixing is dependent upon the electron momentum, it should be different for spin-up and spin-down electrons in the ferromagnetic state, and this difference should vanish in the paramagnetic state. Thus we might expect the effective hyperfine coupling constant $A$ to exhibit a temperature dependence proportional to the magnetization. In Fig. 4 we display the temperature dependence of $A$ as a function of the deviation of the magnetization. The apparent linear relationship again indicates that one can describe the intrinsic temperature dependence of $A$ by such a mechanism. If we transferred this fit to Fig. 2, the curve would fall between those of the phonon-admixture and Stoner-type excitation mechanisms. Therefore a choice between the three mechanisms cannot be made on this basis.

## IV. CONCLUSIONS

We have seen that the domain hyperfine field as measured by the ME and the wall hyperfine field as measured by NMR are different and that the magnitude of this difference may be ascribable to a demagnetizing field. The observed temperature dependence of this difference disagrees with that predicted for spin-wave-like excitations in the walls and may indicate a temperature dependence to the demagnetizing field caused by changing of domain configurations with temperature.

An examination of the magnetization and the hyperfine field shows that the major components which make up the total magnetization appear to have similar if not identical temperature dependences, as indicated by reasonable fits to the data. Given this premise, the effective hyperfine coupling constant $A$ has an intrinsic temperature dependence. Three mechanisms have been shown capable of fitting the data: (i) Thermal excitation of electrons higher into the *d* band, coupled with a strong energy dependence to the core polarization, leads to a $T^2$ dependence for the hyperfine field. While this adequately describes the data, the lack of evidence for these single-particle excitations in magnetization measurements and the requirement of a strong energy dependence to the core polarization make the

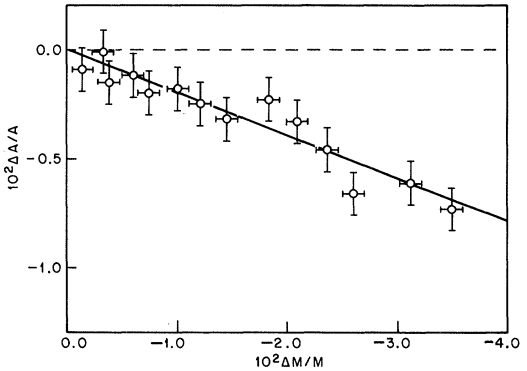

FIG. 4. The fractional change in the effective hyperfine coupling constant versus the fractional change in the magnetization. The straight line is a least-squares fit to the data.

mechanism unlikely. (ii) Phonon admixture of *s* character into the 3*d* wave functions leads to a temperature dependence proportional to the lattice heat. Such an effect has been shown to exist in insulators, and the observed temperature dependence is of a corresponding magnitude here in Fe metal. However, the effects of conduction electrons on such a mechanism have not been explored. It might be pointed out that one would also expect phonon admixture of different orbital states for noncubic metals such as cobalt, which would lead to a temperature dependence of the orbital component of the hyperfine field. (iii) The momentum dependence of the *s-d* mixing which already exists in Fe metal may result in a temperature dependence proportional to the deviation of the magnetization. While such a mechanism also fits the data, we cannot estimate whether the observed magnitude is reasonable without knowing in detail the origin of the hyperfine field and the nature of the band structure.

It is clear from Fig. 2 that to differentiate between the above three mechanisms on the basis of temperature dependence alone would require an improvement of an order of magnitude in the accuracy of the experimental results. It appears that reliable theoretical estimates of the magnitudes of the various effects are currently the only way to identify the origin of the intrinsic temperature dependence of the effective hyperfine coupling constant.

*Note added in proof.* A recent precision measurement of the ground-state splitting of metallic iron, see C. E. Violet and D. N. Pipkorn [J. Appl. Phys. 42, 4339 (1971)], differs significantly from that quoted in Ref. 9. The new value brings the wall and domain hyperfine fields into coincidence within the precision of measurements.

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