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Investigation of dilute magnetic impurities via the Mössbauer effect: Ag^{57}Fe and Ag^{57}Co

J. R. Thompson and J. O. Thomson

Department of Physics, The University of Tennessee, Knoxville, Tennessee 37916
*and Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830**

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We have studied the dilute Kondo alloys Ag^{57}Fe and Ag^{57}Co via the Mössbauer effect at temperatures between 0.022 and 4.2 K and in applied magnetic fields from 0 to 60 kG. For Fe in Ag, this allows study of both the thermal and magnetic breakup of the nonmagnetic ground state. Where they overlap, our results agree with those of Kitchens, Steyert, and Taylor. As the temperature is lowered, a strong reduction from free spin behavior of the local moment is observed. At our lowest temperatures the dependence of the Fe-hyperfine field on applied field is compared with several theoretical and semiempirical expressions, where each describes the data moderately well, none with marked superiority. We also report the first measurements of the hyperfine field of ^{57}Co in Ag, obtained via the intensity asymmetry of the spectra. The field can be described in terms of a large positive Knight shift $K = (28 \pm 8)\%$.

I. INTRODUCTION

In the past few years a great deal of experimental effort using diverse techniques has gone into the study of the dilute magnetic impurity in a "nonmagnetic" host metal.[1-3] Substantial theoretical efforts have followed Kondo's work,[4] which showed that treating the s-d exchange interaction in the second Born approximation gives a divergence in the impurity scattering cross section for conduction electrons. This leads to an increasing electrical resistivity with decreasing temperature which has been observed in many dilute magnetic alloys. At high temperatures, both theory and experiment indicate a temperature-dependent Curie-Weiss susceptibility $\chi = C/(T + T_c)$. Below a characteristic temperature T_c, the susceptibility becomes Pauli-like as the wave function for the impurity–conduction-electron system approaches what is apparently a ground-state singlet.[1,2] This ground-state wave function can of course be destroyed by application of a sufficiently strong magnetic field H_c, given by $H_c \sim k_B T_c/\mu$, where μ is the high-temperature moment. An alternative spin-fluctuation model[5] has also been presented which starts from the low-temperature nonmagnetic impurity state, and gives results which seem to be experimentally indistinguishable from the above theories. Depending upon the theoretical point of view taken, T_c may be identified with the Kondo temperature or a spin-fluctuation temperature, where both theories recognize T_c as marking a change of regime rather than a sharp transition temperature. As yet, no theory has been able to account for the behavior of such a system over the full range of temperature and field from ~0 to well above T_c and H_c, respectively, although the theoretical situation is improving as new techniques are being brought to bear.[6]

Perhaps the most fundamental physical quantity in the study of a dilute magnetic alloy is the local magnetic moment of the impurity atom. This local moment may be inferred, for example, from studies of the bulk susceptibility, although the host magnetization, conduction-electron polarization, impurity interaction effects, and presence of incidental impurities in the alloy may affect the interpretation of bulk measurements. A very useful measure of the local magnetic moment is provided by the magnetic hyperfine coupling to the impurity nucleus. Such a measurement is specific to the impurity species under investigation and, by using techniques such as the Mössbauer effect or nuclear orientation, can be performed with extremely low impurity concentrations so as to reduce impurity interaction effects.

We present here results of magnetic hyperfine studies for ^{57}Co and ^{57}Fe in Ag, obtained using a source of ^{57}Co in Ag metal. For the Fe impurity, we have made Mössbauer measurements over a range of both temperature and magnetic field sufficient to destroy the low-temperature nonmagnetic state. For the Co impurity, the low-temperature nuclear polarization was determined from the intensity asymmetry of the Fe Mössbauer spectra.

In a preliminary communication[7] we reported measurement of the very-low-temperature impurity magnetization for ^{57}Fe and ^{57}Co in Ag. Similar measurements for ^{57}Fe in Ag for $T > 1$ K have been presented[8] and more recently measurements from 30 mK to 60 K were reported for this system.[9] Because of the very low solubility[10] of both Fe and Co in Ag, other techniques which typically require higher concentrations of impurity have not been applied.

If the electronic spin relaxation time is substantially shorter than the nuclear precession time, then the hyperfine interaction for the dilute impurity can be described in terms of an effective, measured magnetic field H_m. This field is parallel (or antiparallel) to the externally applied field H_a, here chosen to define the z axis, and acts on both the spin-$\frac{1}{2}$ ground and spin-$\frac{3}{2}$ excited nuclear states. The nuclear Zeeman levels are then described by the Hamiltonian $\hat{H}$

$$\hat{H} = -g_N\, \mu_N\, H_m\, \hat{I}_z\,, \qquad (1)$$

where g_N is the nuclear gyromagnetic ratio, μ_N is the nuclear magneton, and $\hat{I}_z$ is the z component of nuclear-spin operator. Then in a dilute sample where the bulk magnetization is negligible, one may determine the hyperfine field as $\vec{H}_{hf} = \vec{H}_m - \vec{H}_a$.

H_{hf} is dominantly composed of three contributions: a negative core polarization field, a (probably) positive field due to conduction-electron polarization, and a positive orbital field proportional to $\langle \hat{L}_z \rangle$.[11] The first two contributions arise from interactions with the impurity spin S and are proportional to $\langle \hat{S} \rangle$. Here $\langle \hat{S}_z \rangle$ and $\langle \hat{L}_z \rangle$ are the expectation values of the z-components of the spin and orbital angular momentum operators. The signs given to the contributions indicate the direction of the field with respect to the direction of the associated magnetic moment. Other small contributions may arise from a Van Vleck susceptibility, the conduction-electron susceptibility and magnetic dipole shielding effects. If we neglect these latter three contributions and assume that we remain in the spin-orbit ground state,[11] then the local susceptibility χ_l is found from H_{hf} as $\chi_l H_a = M = M_{sat} H_{hf}/H_{sat}$, where H_{sat} and M_{sat} are the saturation values of H_{hf} and the impurity moment M, respectively.

If as in our experiments the detected ^{57}Fe γ rays travel parallel to the internal magnetic field then the intensities for the six allowed transitions are in the ratios 3 : 0 : 1 : 1 : 0 : 3, in order of increasing energy, assuming equal populations of the excited substates. At sufficiently large values of H_a/T, however, the parent ^{57}Co nucleus is oriented such that the excited ^{57}Fe substates are populated unequally: consequently, an intensity asymmetry appears in the Mössbauer spectrum. From the magnitude and sign of the asymmetry the value of H_m^{Co}/T is readily deduced since the ground-state magnetic moment for ^{57}Co is reported as 4.72 μ_N.[12] In particular, if the lowest-energy Mössbauer γ ray with absorption at positive relative velocity is the most intense, then the ^{57}Co and ^{57}Fe fields have opposite signs.

II. EXPERIMENTAL PROCEDURES

The Ag source was prepared by evaporating to dryness ~0.5 mCi of carrier-free ^{57}CoCl$_2$ activity and ~10 μCi of similar ^{54}Mn activity on a foil of nominally 99.9999%-purity Ag metal.[13] The activity was reduced to the metal by heating it in H$_2$, after which the foil was melted, rolled to appropriate thickness, annealed in H$_2$ near 920 °C, and finally etched in HCl. The thickness of the foil was 0.0025 cm by ~2 cm diam. An autoradiograph showed a reasonably uniform distribution of activity over the surface. The deliberately introduced magnetic impurity level was ~1 ppm, consisting of the ^{57}Co and ^{54}Mn activities and their decay products ^{57}Fe and ^{54}Cr. The total level of incidental magnetic impurities was found after the experiment to be $\lesssim$60 ppm, a portion of which is attributed to surface contaminants in the solder.

The measurements were made in a ^{3}He-^{4}He dilution refrigerator manufactured by S. H. E. Corp. The sample was indium soldered onto a sintered copper cylinder located within the mixing chamber of the refrigerator. A high uniformity superconducting magnet, calibrated through NMR measurements and operated in a persistent current mode, surrounded the refrigerator providing highly stable axial fields up to 60 kG.

The 14.4-keV γ radiation from the source passed through a series of beryllium windows before reaching a moving absorber of Na$_4$Fe(CN)$_6$ · 3H$_2$O enriched in ^{57}Fe. It was located 15 cm below the source, where the field was 7% of its central value. The absorber was attached to the top of an electromechanical transducer suspended below the Dewar of the cryostat. It was operated in a triangular velocity mode synchronously with the addressed channels of a dual-input zero-dead-time multiscalar. A second Mössbauer spectrum was acquired simultaneously to provide calibration of the velocity scale. We used an auxiliary source of ^{57}Co in either Fe or Cu, which was attached to the bottom end of the transducer; the fixed absorber was Armco iron. The γ counts from both sources were registered in Kr-methane proportional counters and processed by standard techniques prior to counting by the dual multiscalar.

The spectra so obtained were computer fit to a sum of eight Lorentzian lines which correspond to the convolution of four circularly polarized source lines with four circularly polarized absorber lines. The source line positions were determined from Eq. (1) in terms of the variable $H_m^{Fe} = H_m$. The absorber line positions were also determined from Eq. (1) using for H_m the value of the fringing field at the absorber of 0.07 H_a. Other variables in the computer code in addition to H_m^{Fe} include an isomer shift, a linewidth, a total intensity parameter, and H_m^{Co}/T, which determines the intensity asymmetry as discussed above. These parameters could also be held fixed if desired in the computer fits. This program was developed by John Burton and modified with his assistance for this application.

For thermometry, the nuclear orientation of ^{60}Co in hexagonal Co metal was used to calibrate a carbon resistance thermometer in high magnetic

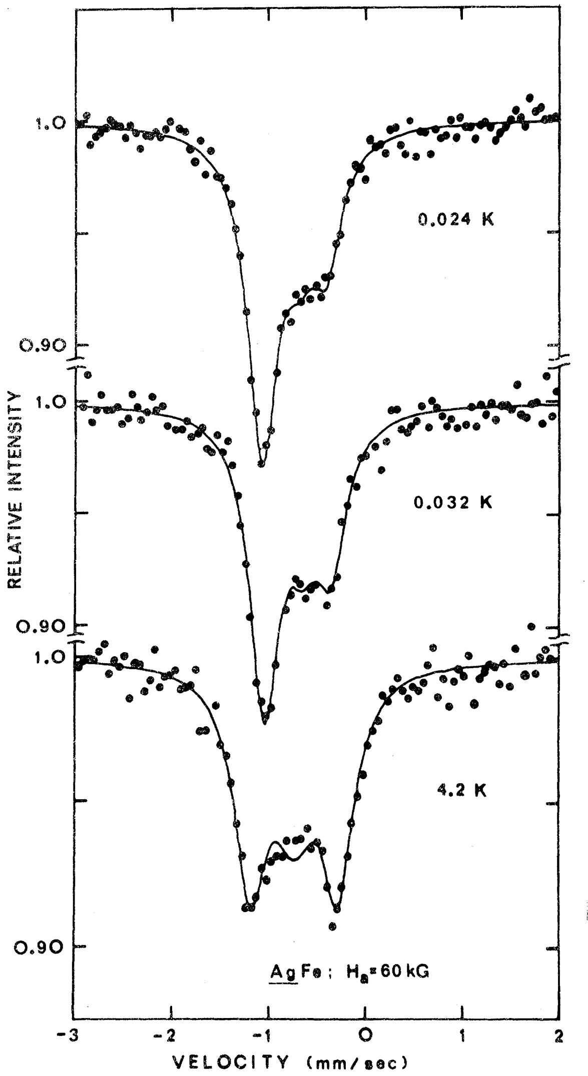

FIG. 1. Typical ^{57}Fe Mössbauer spectra for Fe in Ag obtained at temperatures of 0.024, 0.032, and 4.2 K, in applied field of 60 kG. Note the changing asymmetry in intensity in the outer peaks caused by the polarization of the ^{57}Co parent nuclear states.

fields at low temperature. In low fields we also used the Curie-law susceptibility of cerium magnesium nitrate (CMN) measured by a superconducting quantum interference device (SQUID) magnetometer. Our estimated errors in the measurement of reciprocal temperature ($1/T$) do not exceed 2 K^{-1} in the ^{60}Co calibration range below 50 mK, i.e., where $T^{-1} > 20$ K^{-1}. In addition to the Kapitza boundary resistance, the presence of magnetic fields of several tens of kilogauss magnitude considerably complicates the problem of very-low-temperature thermometry and accounts for the most of the quoted uncertainty. No precise calibration is available to us at intermediate temperatures in large fields because of the large and complex magnetoresistance[14] in our carbon resistor, cut from a Speer Grade 1001A resistor.

III. RESULTS

Several Mössbauer spectra are shown in Fig. 1. It is evident that they are characterized by the smallness of the splitting, with an observable (and measurable) intensity asymmetry only when the applied field is large and the temperature is low. In most of the spectra, the applied field was insufficient to split clearly the lines. In these cases, very careful attention was given to insuring that the linewidth and positions used did not affect the results for H_{hf}^{Fe} and H_{hf}^{Co}/T. For the isomer shift, we obtained a value of − 0.75 mm/sec for the cold *Ag*Fe source measured with respect to the room-temperature absorber. The shift is in agreement with the value obtained by Kitchens, Steyert, and Taylor.[8]

A. *Ag*Fe

We first describe the *Ag*Fe results. In Fig. 2, the triangles show the absolute measured Fe field H_m^{Fe} determined by computer fits for those runs taken between 20 and 25 mK (i.e., the low-temperature limit where $T \sim T_c/100$), plotted as a function

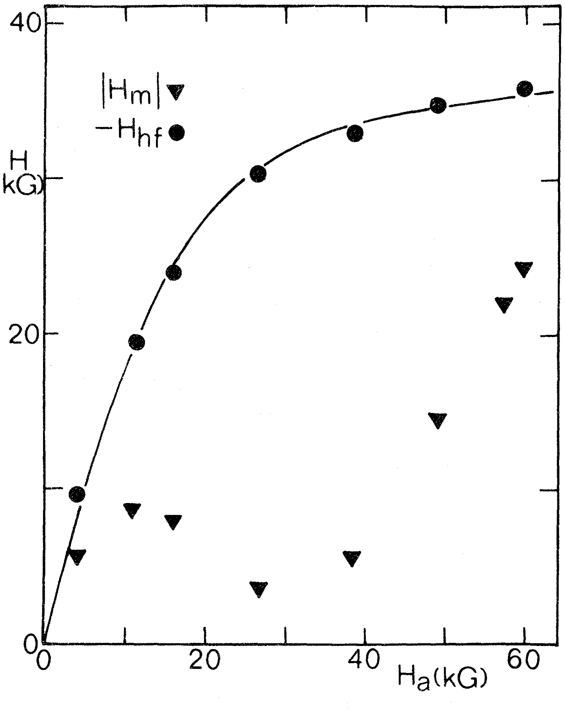

FIG. 2. Plot of the ^{57}Fe field vs applied field H_a for $T \approx 0.022$ K. The triangles (▼) represent the magnitude of the measured field H_m^{Fe}. Circles (●) represent the negative of the deduced hyperfine field, $-H_{hf}^{Fe}$, for ^{57}Fe in Ag.

TABLE I. Results of the fits to the Ag^{57}Fe data taken below 50 mK.

Function form		H_{sat} (kG)	χ^2 per degree of freedom	$\mu = gJ$ (μ_β)[d]
$\frac{H_{\text{hf}}}{H_{\text{sat}}} = \left\| 1 + \left(\frac{kT_c}{\mu H_a}\right)^m \right\|^{-1/m}$	$m = 2$[a]	-36.9	4.7	4.0
	$m = 3/2$	-38.7	1.6	4.4
	$m = 1$	-45.3	2.4	7.2
$\frac{H_{\text{hf}}}{H_{\text{sat}}} = 1 - \frac{2}{\pi} \coth^{-1}\left(\frac{\mu H_a}{k_B T_c}\right)$	b	-40.8	1.8	6.0
$\frac{H_{\text{hf}}}{H_{\text{sat}}} = B_J\left(\frac{\mu H_a}{k_B(T+T_c)}\right)$	$J = 2$[c]	-36.1	4.4	4.1
	$J = 3$[c]	-36.6	4.0	4.6

[a] Reference 16.

[b] Reference 19.

[c] Reference 18.

[d] For $T_c = 2.4$ K.

of the applied field H_a. These fits were performed using a fixed width of 0.34 mm/sec as determined from zero-field spectra taken at 4.2 and 300 K. We have assumed that the electronic relaxation is sufficiently fast that a hyperfine field model is appropriate to describe the data. The small values of $|H_m^{\text{Fe}}|$ at 27 and 39 kG suggest that the measured field changes sign between these two values of H_a with $H_m^{\text{Fe}} > 0$ above ~35 kG. Based on this assumption we have determined the hyperfine fields at the iron nucleus $H_{\text{hf}}^{\text{Fe}} = H_m^{\text{Fe}} - H_a$, and these values are shown as circles on Fig. 2. The statistical uncertainty in the fields obtained in the computer fits are 0.3–0.5 kG and are smaller than the size of the plotted symbol. In addition, there may be small systematic errors arising, for instance, from uncertainties in width, calibration, or fringing field.

Steiner *et al.*[9] in Mössbauer measurements of ^{57}Fe in Cu and Au find evidence for a positive temperature-independent contribution to the hyperfine field. For ^{57}Fe in Ag, however, they estimate the contribution to be very small. We cannot detect such a term in our measurements, as they do not extend to the high temperatures where such a term becomes important.

To assess the effect of the chosen linewidth on our data, we have also fit all of the spectra to narrow lines which, of course, artificially increases the absolute value of the measured fields for those spectra which are not resolved. This results in an artificial decrease in the values of $H_{\text{hf}}^{\text{Fe}}$ for $H_a < 35$ kG, and an increase for $H_a > 35$ kG causing the data in Fig. 2 to appear discontinuous. From the smoothness of the data through this field region as seen in Fig. 2, it is evident that no serious error arises from the use of 0.34 mm/sec width, thus substantiating the correctness of using the zero-field width for fitting our low-temperature data. The width used is also consistant with our measurements at 4.2 K and $H_a = 60$ kG, where the spectra were resolved.

Theoretical treatments of the low-temperature limit for the magnetization of a spin-$\frac{1}{2}$ impurity using the *s-d* Hamiltonian have been given by Nam and Woo,[15] Ishii,[16] and Bloomfield *et al.*[17] Semi-emperical forms have been suggested by Kitchens and Taylor[18] and by Gallop.[19] We have computer fit our data on $H_{\text{hf}}^{\text{Fe}}$ vs H_a to several of these forms, each with two adjustable constants (except for the Brillouin function type of Kitchens and Taylor[18] where a value of J can also be chosen), and find they all describe the low-temperature data acceptably. The solid curve shown in Fig. 2.shows Ishii's expression when fit to the data. In Table I, we give parameters, including χ^2 per degree of freedom, which result from fitting several of these functions. Only statistical errors in H_{hf}, typically 0.3–0.5 kG, were accounted for in the fits. For the Ishii and Kitchens-Taylor fits, the larger values of χ^2 are associated with insufficient curvature of the function at low H_a. It is inappropriate to draw too much significance from these deviations since all of the functions are either semiempirical or involve approximations, particularly the use of spin $\frac{1}{2}$. Furthermore, no systematic errors have been included in determining the goodness of fit. We have also fit the data to the empirical function

$$H_{\text{hf}} = H_{\text{sat}} \left| 1 + (kT_c/\mu H_a)^m \right|^{-1/m},$$

and find that the best fit occurs for $m \sim \frac{3}{2}$, rather than for $m = 2$, which corresponds to the theoretical result of Ishii.[16] It is conceivable that the failure of the data to fit the former functions at low temperature and fields may arise from a slow relaxation at low fields which would act to broaden and shift the lines.

In Fig. 3, we show our *Ag*Fe data for all temperatures plotted versus $\log(H_a/T)$. These data are in essential agreement with those of Kitchens *et al.*[8] in the region above 1 K where they overlap. Although their values for H_{hf} appear to be ~1 kG larger in magnitude where comparison is readily

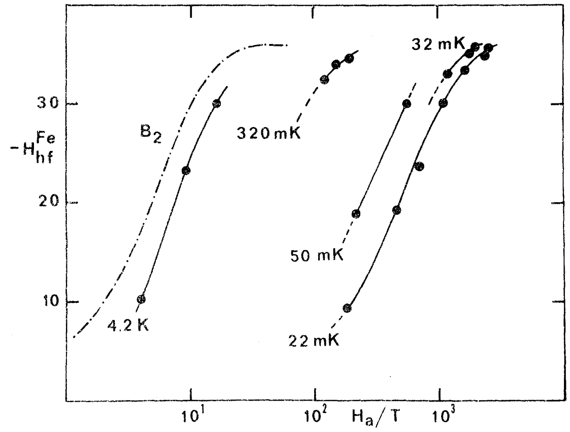

FIG. 3. Plot of the negative of the Fe-hyperfine field $-H_{hf}^{Fe}$ vs the applied field divided by the temperature H_a/T, in kG/K. Solid curves are isotherms at the temperatures indicated. The broken line labeled B_2 shows a Brillouin function for $J=2$ and $g=2$. Note the strong suppression of the hyperfine field as the temperature is lowered.

made, such a discrepancy may readily be accounted for by small errors associated with velocity or field calibration, fringing field, etc., and we feel it is not significant. We also show a $J=2$ Brillouin function with g value of 2. This curve saturates somewhat less rapidly that the $J=1$, $g=\frac{7}{2}$ Brillouin function suggested by Hirst[10] for Fe in noble metals above T_c. It is evident that the Fe moment is very strongly quenched at lower temperatures, as already noted by Kitchens *et al.*[8]

We have also fitted all of our data including the high-temperature results using the empirical function of Kitchens and Taylor. Allowing for no systematic error, we find $H_{sat} = -36.1$ kG and $T_c = 2.44$ K with $g=2.03$. The value of $J=2$ gives $\mu = gJ\mu_B = 4.1\ \mu_B$, all in agreement with the earlier results.[18]

At small values of H_a/T, the ratio H_a/H_{hf}^{Fe} is inversely proportional to the impurity susceptibility. If we plot this ratio as a function of T, it follows a Curie-Weiss law with intercept at $T = -1.6 \pm 0.3$ K.

B. *Ag*Co

As mentioned above, the intensity asymmetry of the Mössbauer spectrum is fit directly in terms of a parameter H_m^{Co}/T, where the sign of H_m^{Co} relative to H_m^{Fe} also comes from the fit. From the systematics of H_{hf}^{Fe}, we know the sign of H_m^{Fe} and therefore can determine both sign and magnitude for H_m^{Co}/T for those spectra where an appreciable asymmetry is observable. In Fig. 4, we have plotted the experimentally determined dependent variable (H_m^{Co}/T) vs H_a/T, which combines properly the independent variables. Over the temperature range from 20 to 80 mK and for $H_a > 40$ kG, where there is a measurable asymmetry, these factors are directly proportional. Hence H_m^{Co} is independent of temperature and describable by a large positive Knight shift which is temperature and field independent:

$$H_m^{Co} = H_a(1+K) \quad \text{with } K = (28 \pm 8)\%.$$

A large positive Knight shift of 29.2% has also been observed for Co in Au by nuclear orientation techniques[20] and NMR measurements[21] in less dilute *Au*Co alloys. Nuclear relaxation studies for Co in Au have shown that a substantial exchange enhancement occurs in this system, where apparently the positive orbital field exceeds the negative core polarization field arising from the d electron spin. In the absence of further information for *Ag*Co we suggest that this system is very similar to the more extensively studied *Au*Co system.

Although measurements were made on the nuclear orientation of ^{54}Mn in the Ag sample by means of the 864-keV γ-ray anisotropy, the data were poor because of background γ rays resulting from Compton scattering of γ rays from the ^{60}Co thermometer source and from small amounts of ^{58}CO and ^{56}CO impurities included with the ^{57}CO activity. Within our errors, our results agreed with previous workers[22] for Ag^{54}Mn.

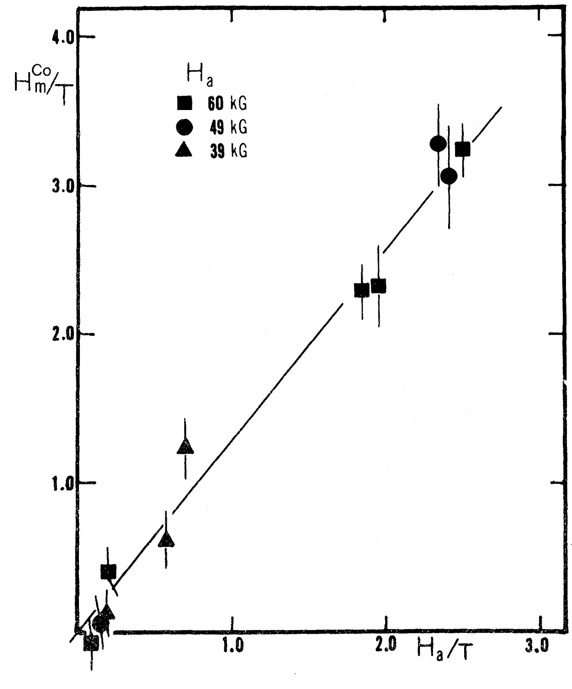

FIG. 4. Plot of the deduced cobalt nuclear magnetic field divided by temperature H_m^{Co}/T vs applied field divided by temperature H_a/T. Units are kG/mK. Data are described by a Knight shift $K = (28 \pm 8)\%$.

IV. DISCUSSION

Qualitatively, we attribute the small magnitude of the Fe-hyperfine field to orbital effects which offset most of the spin-induced negative field. Assuming that the smaller Fe ion enters the Ag lattice substitutionally, the predominantly cubic environment does not fully quench the orbital momentum. Freeman and Watson, as discussed in Narath's review,[23] have shown that large positive orbital fields obtain for ions near the end of the 3*d* series. If $\langle \hat{L}_z \rangle$ is a fraction of a Bohr magneton, this would suffice to account for the small observed field. The positive Co-hyperfine field is consistent with this, for the orbital contribution is substantially larger here, while the Co spin is smaller. The detailed behavior is undoubtedly complicated by spin-orbit effects, the Kondo condensation, etc. It is possible, too, that if the spin-orbit coupling in the Fe atom is sufficiently weak, then the spin and orbital contributions to H_{hf}^{Fe} do not have the same dependence on H_a and T. It is unfortunate that the solubility problem prevents application of other techniques such as EPR, NMR, or neutron scattering. The general behavior, however, is accounted for in this way.

A number of studies have shown that interaction effects can play a role in the behavior of dilute alloys. In these measurements we believe that interaction effects are not significant. (i) The characteristic temperature $T_c \sim 2$ K, while the solubility[10] of the Fe and Co impurities is ~10 ppm. From both a theoretical[24] and an empirical[25] basis, interaction and coupling effects should be negligible for concentrations less than about 30–60 ppm. (ii) The Co and Fe magnetizations, inferred from the hfs coupling, have different field and temperature behavior so that at least static coupling of these species is not evident.

There remains the possibility that electronic spin relaxation may be sufficiently slow so as to affect the hyperfine field representation used to interpret our results. If this were the case, line shifts and line broadening effects would be present, which might not be evident because of the small splittings observed. We offer the following as evidence that such effects are not present: (i) For those spectra where a line width is available, i.e., 4.2 °K, $H_a = 0$ and $H_a = 60$ kG and 22 mK, $H_a = 60$ kG, the same width fits the data well. (ii) Window *et al.*[26] suggest that a large value of $|\rho J_{exc}|$, the parameter which enters the calculation for the Kondo temperature as well as the Korringa relaxation rate, will cause rapid relaxation in high T_c alloys. An order-of-magnitude calculation confirms that the relaxation rate is, in fact, rapid compared with the nuclear Larmor frequency, which is small since $|H_m^{Fe}|$ is ≤ 30 kG. Since below 49 kG our spectra are unresolved, our hyperfine field data for Fe below 49 kG come, in practice, from linewidth measurements. The behavior of the data suggests that there is no additional line broadening. A possible exception to this is the low-temperature 4- and 10.7-kG data, where the data points lie somewhat above the various functions, as mentioned above. We conclude, however, that it is unlikely that these results are affected by slow relaxation.

V. SUMMARY

In conclusion, we have measured for isolated ^{57}Fe and ^{57}Co impurities in Ag the hyperfine magnetic field which is proportional to the net impurity magnetization. The ^{57}Fe field H_{hf}^{Fe} shows that the magnetism is restored only for $T \sim T_c = 2.4$ K or for $H_a \sim H_c \sim 20$ kG. The field is negative, saturating at a value of $H_{sat} = -37$ kG. The low value of H_{sat} probably arises from incomplete quenching of the orbital magnetism which gives a positive contribution to H_{hf}. Several theoretical and semiempirical functions can be fit to the data, describing it moderately well.

While our results for H_{hf}^{Co} extend over only a small range of temperature, our data are consistent with the cobalt also being nonmagnetic with T_c well above our measurement temperature. The positive hyperfine field of Co, which is of the form of a Knight shift, implies that the orbital field of Co in Ag is even greater than that of Fe in Ag.

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