Local magnetization of dilute Fe in Tc, Ru, and Ir from Mössbauer measurements

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The magnetic character of the localized spin-fluctuation systems *Tc*Fe, *Ru*Fe, and *Ir*Fe has been investigated by Mössbauer experiments between 1.4 and 290 K in external magnetic fields up to 50 kOe. Saturation values of the hyperfine fields in 50 kOe are -7.3 ± 1 kOe for *Tc*Fe and -9.3 ± 1 kOe for *Ir*Fe, which are much smaller than those of usual Kondo systems. For Tc and Ir hosts, the saturation values are proportional to the external field, and this property closely resembles Kondo systems like *Cu*Fe and *Rh*Fe. For Ru host, the observed small and temperature-independent hyperfine field ($|H_{hf}| < 2$ kOe) is consistent with the extremely high spin-fluctuation temperature.

I. INTRODUCTION

In the years after the paper of Clogston *et al.*,[1] who made the first systematic study on the magnetic character of dilute Fe impurities in 4*d* transition-metal alloys, considerable efforts have been devoted, both experimentally and theoretically, to understanding the magnetic property of dilute alloys with 3*d* transition-metal impurities.[2]

The usefulness of the Mössbauer effect in this field was demonstrated by Kitchens *et al.*[3] They showed that the localized magnetic moment obtained from the hyperfine-field measurements of ^{57}Fe in several metal hosts agrees with that derived from the magnetic-susceptibility measurements in Ref. 1.

In recent years Mössbauer-effect studies on Kondo systems of noble-metal–Fe alloys (*Cu*Fe, *Ag*Fe, and *Au*Fe) have provided valuable information about the magnetic behavior of these systems above and below the Kondo temperature.[4,5] However, only little is known experimentally of the magnetic character of Fe impurities in the hosts like Tc, Ru, and Ir, where Fe is in the transition region from the nonmagnetic to magnetic state. In fact, among these, the Mössbauer study so far published is, to the authors' knowledge, limited to the early work with *Ir*Fe by Taylor and Steyert.[6] Besides, the theoretical situation for these hosts is less clear than for noble metals, because of the complicated band structure and the small valence difference between Fe and these hosts.

From macroscopic measurements, e.g., susceptibility, electrical resistivity, and superconducting transition temperature, the following have been revealed on the magnetic property of dilute Fe in Tc, Ru, and Ir. Clogston *et al.*[1] showed from the susceptibility measurement that the magnetic character of Fe impurities changes from magnetic to nonmagnetic when the host changes from Mo (the number of outer electrons $N=6$) to Re ($5d, N=7$), which was used instead of 4*d* Tc ($N=7$), and the change from nonmagnetic to magnetic occurs when the host changes from Ru ($N=8$) to Rh ($N=9$). In the 5*d* series, it was shown that Fe impurities start to have the localized moment character when the 5*d* band is fuller than that of Ir ($N=9$).[7]

Recently, it has been revealed that the characteristic temperature dependence of electrical resistivity of *Ru*Fe (Ref. 8) and *Ir*Fe (Ref. 9) is due to the scattering of conduction electrons by localized spin fluctuations (LSF). The additional resistivity $\Delta\rho(T)$ due to Fe impurities has no minimum, but increases as T^2 and then as T when temperature T increases. From the analysis of $\Delta\rho(T)$ the spin-fluctuation temperature T_{sf} has been derived to be more than 700 K for *Ru*Fe (Ref. 8) and 225 K for *Ir*Fe.[9]

Since Tc, Ru, and Ir metals are superconductors, the effect of Fe impurities on the superconducting properties of the hosts gives additional information about the magnetic character of Fe impurities. For example, measurements of the depression of the superconducting transition temperature of Tc (Ref. 10), Ru (Refs. 8 and 11), and Ir (Ref. 12) by Fe impurities have corroborated that these alloys are LSF systems.

Taking into account the above-mentioned situation, it is worthwhile to investigate the *Tc*Fe, *Ru*Fe, and *Ir*Fe alloys using the microscopic Mössbauer probe, with which one expects to get more information on the magnetic character of LSF systems and on the transition of Fe impurities between magnetic and nonmagnetic states.

The present work is an extension of our previous works on *Tc*Fe alloys, in which we reported the superconducting property[10] and the hyperfine field[13] of *Tc*Fe. Here we present the results of the Mössbauer experiment (hyperfine fields as a function of external magnetic field and of temperature), with ^{57}Fe in Tc, Ru, and Ir. Our results are compared with the

Mössbauer results for typical Kondo systems like *Cu*Fe and *Rh*Fe. Discussion on the magnetic character of LSF systems is also given combining the data of the hyperfine field and the depression of the superconducting transition temperature.

II. EXPERIMENTAL

A. Sample preparation

In order to eliminate interactions between impurities and to observe only the single impurity behavior in any host, samples with extremely dilute impurity concentration (in a low-ppm region) are needed. With these samples, the Mössbauer measurement is feasible if one uses them as the source rather than as the absorber. Preparations of three kinds of sources (*Tc*Fe, *Ru*Fe, and *Ir*Fe) were made as follows.

1. *Tc*Fe

As the first step, Tc was electrodeposited on a Cu plate. The thickness of Tc was estimated to be about 1 μm. Then carrier-free ^{57}Co (in the chemical form of $CoCl_2$, 0.12 μg/mCi) and Tc (in NH_4TcO_4) were simultaneously electrodeposited on the Tc base. The total thickness of the sample was about 10 μm. This sample was annealed (or reduced) in an atmosphere of highly purified, flowing hydrogen. The production of metallic Tc was examined by means of x-ray analysis and was confirmed having the characteristic hcp structure. In order to examine the uniformity of the sample produced by the above method, the superconducting transition temperature was measured by the ac susceptibility method, which is sensitive to lattice imperfections.[14] Details of Tc sample preparations were previously reported in our relevant works.[14,15]

2. *Ru*Fe

Since it is very difficult to cold roll Ru metal into a foil form, a pressed flat disk of 200-mesh powder was used as the Ru host. After dropping the proper amount of liquid ^{57}Co on it, the Ru sample was heat treated in the hydrogen atmosphere and then in a high vacuum.

3. *Ir*Fe

As the Ir host, an Ir foil of 50 μm was used. Similar to the Ru sample, the proper amount of liquid ^{57}Co was dropped on it and heat treatment was made in the hydrogen atmosphere. Specific information on the preparation of all samples is listed in Table I.

TABLE I. Summary of the data for sample preparation.

Host metal	Shape	Thickness (μm)	Plated ^{57}Co (μCi)	Diffusion treatment
Tc (hcp)	Electrodeposited foil	10	35	900 °C in H_2 for 46 h
Ru (hcp)	Pressed disk	500	400	1100 °C in H_2 for 6 h 1050 °C in high vacuum for 10 h
Ir (fcc)	Foil	50	100	1100 °C in H_2 for 25 h

B. Mössbauer measurements

The Mössbauer experiment was performed in the temperature range of 1.4–290 K and in the external magnetic field H_{ext} up to 50 kOe. The spectra were obtained in the standard transmission geometry with a fixed source and a moved absorber. The sample was placed at the center of a superconducting magnet and was attached to a copper holder by Apiezon-*N* grease. The external field was perpendicular to the sample surface and was parallel to the γ-ray direction. Field-strength calibrations were made using a GaAs Hall element, within uncertainty of ± 0.5 kOe. Temperatures (1.4–4.2 K) measured by vapor pressure of ^{4}He were stable to better than 2% during measurements of about 10 h. Above 4.2 K, a thermocouple of Au-0.07 at.% Fe vs Ag-0.37 at.% Au was used, which is insensitive to a magnetic field.

A single line absorber of potassium ferrocyanide containing 0.5 or 1.0 mg/cm^2 of ^{57}Fe was always kept at room temperature. A fringing field which is less than 5% of the field at the coil center acts on the absorber and slightly broadens the line width. The calibration of the velocity was made by the known magnetic splitting of α-Fe.

The effective magnetic field H_{eff} acting on the ^{57}Fe nucleus contained in the sample is given by the sum of the external field H_{ext} and the induced hyperfine field H_{hf}; i.e., $H_{eff} = H_{ext} + H_{hf}$, where H_{eff} is determined from the hyperfine spectra. Since H_{hf} is assumed to be proportional to the electronic polarization localized on the impurity site, the field and temperature dependence of H_{hf} reflects the dependence of the local magnetization on fields and temperatures.

It is well known that in hcp transition metals there is an electronic field gradient resulting in appearance of a quadrupole splitting (QS). In our previous paper,[13] the QS of ^{57}Fe impurity in Tc (hcp) was determined as -0.13 ± 0.02 mm/sec. For the Ru (hcp) host, by a similar analysis of the present spectrum, we determined the absolute value of QS as

0.14 ± 0.02 mm/sec, which is in excellent agreement with that by Wortmann and Williamson.[16]

Based on the small values of QS for both Tc and Ru samples, we made an analysis of the hyperfine field by assuming the Lorentzian line shape, the ideal intensity ratio of 3:0:1:1:0:3, and the equal linewidth.

For the Ir (fcc) sample, which has no quadrupole interaction, the spectra at 290 and 86 K were well fitted by the above functional form. However, below 4.2 K, the spectra showed an anomalous strengthening of the inner part of spectra (see below). Nevertheless, the value of $H_{\rm eff}$ was determined within an error of ± 1 kOe from the splitting of the outer pair of lines, which were well resolved and unaffected by the anomalous growth at the central part of the spectrum.

III. RESULTS AND DISCUSSION

In Figs. 1 and 2, the hyperfine field $H_{\rm hf}$ at ^{57}Fe nucleus in Tc, Ir, and Ru is plotted as a function of $H_{\rm ext}/T$. The solid and broken lines in the figures are drawn only for seeing easily and do not represent any theoretical prediction.

To begin with, we discuss the results of *Tc*Fe and *Ir*Fe. In general, $H_{\rm hf}$ measured in the Mössbauer experiment involves the temperature-independent term (in addition to the temperature-dependent term) due to the field-induced Van Vleck paramagnetism of higher-lying electronic states. Hence $H_{\rm hf}$ is expressed as

$$H_{\rm hf} = H_{\rm loc}(H_{\rm ext}, T) + \beta H_{\rm ext} \ , \qquad (1)$$

where $H_{\rm loc}$ denotes the contribution of local *d*-spin

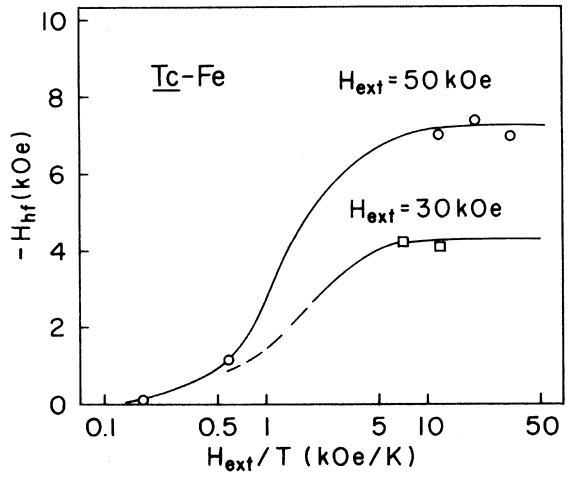

FIG. 1. Behavior of the ^{57}Fe hyperfine field in Tc as a function of $H_{\rm ext}/T$ at external fields of 30 and 50 kOe. The standard error is ± 1 kOe. Solid lines are drawn for seeing easily.

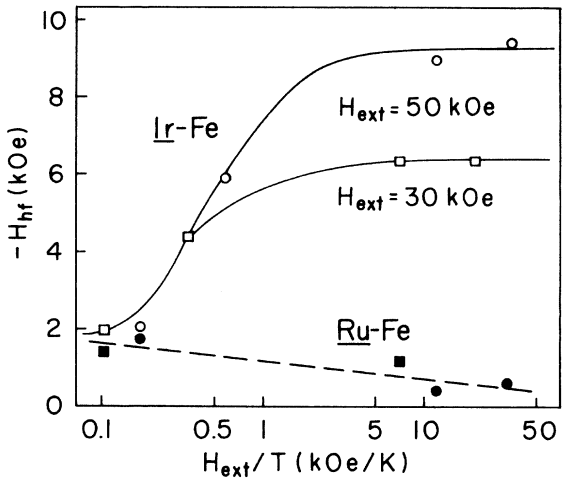

FIG. 2. Behavior of the ^{57}Fe hyperfine field in Ir and Ru as a function of $H_{\rm ext}/T$. Experimental data are taken at $H_{\rm ext}$ = 50 kOe (○:Ir, ●:Ru) and $H_{\rm ext}$ = 30 kOe (□:Ir, ■:Ru). The standard error is ± 1 kOe.

moment. The parameter β in Eq. (1) is determined by plotting the observed values of $H_{\rm hf}/H_{\rm ext}$ as a function of $1/T$, and then by extrapolating the curve to $1/T \to 0$. In the present case, for *Tc*Fe, β is zero within the experimental error, and for *Ir*Fe, β is estimated to be -0.02 ± 0.01. Because of the smallness of β found here, $H_{\rm hf}$ can be considered to be proportional to the 3*d* magnetization of Fe impurity site.

As seen in the figures, the saturation values of $H_{\rm hf}$ in $H_{\rm ext}$ = 50 kOe are -7.3 ± 1 kOe for *Tc*Fe and -9.3 ± 1 kOe for *Ir*Fe. The latter result is in agreement with the previous work,[6] where the Van Vleck term was estimated as $\beta = -0.064$, being less important to the following discussion. It is remarkable that the saturation values of $H_{\rm hf}$ of *Tc*Fe and *Ir*Fe are much smaller than those found for usual Kondo systems under a similar experimental condition. The curves of $H_{\rm hf}$ observed here are not a unique function of $H_{\rm ext}/T$, but the saturation values of $H_{\rm hf}$ are almost proportional to the magnitude of $H_{\rm ext}$. As to *Tc*Fe, there are no data comparable with ours, but for *Ir*Fe, the present result is consistent with the bulk susceptibility measurement of dilute Fe impurities in Ir by Knapp,[17] who has indicated that the magnetization does not saturate, but is practically linear with $H_{\rm ext}$ between 0.37–293 K under $H_{\rm ext}$ up to 11 kOe.

The field dependence of $H_{\rm hf}$ in the present experiment closely resembles the behavior in such Kondo systems as *Cu*Fe (Refs. 18 and 19) and *Rh*Fe (Ref. 20) under the condition of $\mu H_{\rm ext} < k_B T_K$, where μ is the magnitude of the local moment and T_K is the Kondo temperature. In these Kondo systems, the proportionality between the saturated value of $H_{\rm hf}$ and $H_{\rm ext}$ is interpreted as the evidence for the growth

of the magnetic moment due to the gradual breaking up of the spin-compensated Kondo state by the external field. Estimations of the saturation hyperfine field H_{sat} of these systems are made by assuming that the data at temperatures much higher than T_K are represented by the Brillouin function.

In the present *Tc*Fe and *Ir*Fe cases, however, a similar analysis is not applicable by the following reason: Based on the assumption that T_{sf} of LSF systems is equivalent to T_K of Kondo systems, as suggested by Rivier and Zuckermann,[21] the experimental temperature should be much higher than T_{sf} for estimations of H_{sat}. But the maximum temperature in our experiment is 290 K, and besides H_{hf} is so small that the comparison with the Brillouin function cannot be made.

Unfortunately, at the present stage, no other appropriate prediction for the behavior of $H_{hf}(H_{ext},T)$ based on the LSF model exists. Therefore, using a simple stochastic model, we attempt a qualitative explanation of the small negative H_{hf} observed in *Tc*Fe and *Ir*Fe systems. For simplicity, we assume that the magnitude of the temporary spin at the Fe impurity is $\frac{1}{2}$ and that it decays with a characteristic lifetime of $\tau_{sf}=h/k_B T_{sf}$ (5×10^{-13} sec for $T_{sf}=100$ K) at temperature below T_{sf}. Under an external field H_{ext}, due to the electronic Zeeman splitting, the energy level of the down-spin state becomes lower than the up-spin state by $g\mu_B H_{ext}$. Hence the probability that the temporary spin stays in the down state $[P(\downarrow)]$ is larger than that of the up state $[P(\uparrow)]$.

It is well understood that H_{hf} measured by the Mössbauer effect is a time-averaged value during the Larmor precession period τ_L. For example, at $H_{eff}=50$ kOe, τ_L corresponds to 2.5×10^{-7} sec, much larger than τ_{sf}. Thus H_{hf} can be written as

$$H_{hf}=-\langle P(\downarrow)|H\downarrow|-P(\uparrow)|H\uparrow|\rangle \ , \qquad (2)$$

where $H\downarrow$ and $H\uparrow$ represent the hyperfine field for the down-spin state and the up-spin state, respectively. The brackets mean a time average during τ_L.

It is not possible to express $P(\downarrow)$ and $P(\uparrow)$ in a simple form, because they do not obey the Boltzmann distribution. The reason for this is that at a low temperature well below T_{sf} and in a high external field, the thermal relaxation time becomes larger than τ_{sf}, and consequently a temporary spin does not have time to come into thermal equilibrium before vanishing. By this reasoning, one may expect that the ratio $P(\uparrow)/P(\downarrow)$ is enhanced over the value in the thermal equilibrium. In addition, since the temporary spin decays with a lifetime τ_{sf}, the probability for the spin absence is not zero; i.e., $P(\downarrow)+P(\uparrow)<1$. This is in contrast with the usual relaxation phenomenon of permanent spins.

From the above discussion, one can reasonably expect the possibility that the difference between $P(\downarrow)$ and $P(\uparrow)$ is much smaller than unity; i.e., $P(\downarrow)-P(\uparrow)\ll 1$. Thus the small negative value of H_{hf} is qualitatively understood. Furthermore, if $|H\downarrow|$ and $|H\uparrow|$ are presumed to be almost equal, the proportionality between H_{hf} and H_{ext} is reduced to the proportionality between $[P(\downarrow)-P(\uparrow)]$ and H_{ext}. Taking into account the fact that the spin does not obey the Boltzmann distribution, this proportionality is not unlikely.

In Fig. 2 are shown the results obtained for the *Ru*Fe sample. All of the values of H_{hf} are so small ($|H_{hf}|<2$ kOe) and lack definite dependence on T and H_{ext}. Negative H_{hf} can be interpreted as the result of the fact that the contribution of the negative $3d$ magnetization exceeds the positive Van Vleck contribution. In particular, the lack of a strong temperature dependence of H_{hf} below room temperature permits us to say that the spin-fluctuation temperature is much in excess of 290 K. This is in general agreement with the result inferred by Kao and Williams[8] ($T_{sf}>700$ K) from an analysis of the temperature dependence of the electrical resistivity.

Now we turn out to the Mössbauer spectra of the *Ir*Fe sample. In Fig. 3, we show the anomalous spectra observed at low temperature (4.2 and 1.4 K) under 50 kOe. These spectra have an additional amplitude in the inner part compared with the spectra at 290 and 86 K. After various attempts, it was found that these spectra were well fitted by a computer analysis with the ratio of $3{:}0{:}\alpha{:}\alpha{:}0{:}3$ and with the equal linewidth. Thus the value of α was determined as 1.4 and 1.2 at 4.2 and 1.4 K, respectively. The calculated linewidth at 4.2 and 1.4 K was larger than that at 290 K by 12% and 17%, respectively. However, under $H_{ext}=0$, there is no appreciable broadening of linewidth (compared with that at 290 K), indicating that no magnetic ordering occurs down to 1.4 K. Similar anomalous spectra of *Mo*Fe (Refs. 3, 22, and 23) and *Rh*Fe (Ref. 24) have been reported, but in the previous paper with *Ir*Fe by Taylor and Steyert,[6] there is no description about the anomaly.

There are two possible explanations of this enhanced amplitude in the inner part of the spectra. A simple explanation is a distribution of the hyperfine field due to inhomogeneities in the sample. In other words, the spectrum is a superposition of several peaks corresponding to different magnitudes of H_{hf}, which is caused by an interaction between impurities or by clustering of the impurities. Another explanation is, as proposed by several authors on *Mo*Fe (Refs. 22 and 23) and *Rh*Fe (Ref. 24), the presence of relaxation effects which generally occur when the electronic relaxation time becomes comparable with the Larmor precession time of ^{57}Fe nucleus in the effective hyperfine field.

In order to inquire into the cause of the anomaly, another Ir sample containing 0.35 at.% Co impurities was prepared by means of the simultaneous electro-

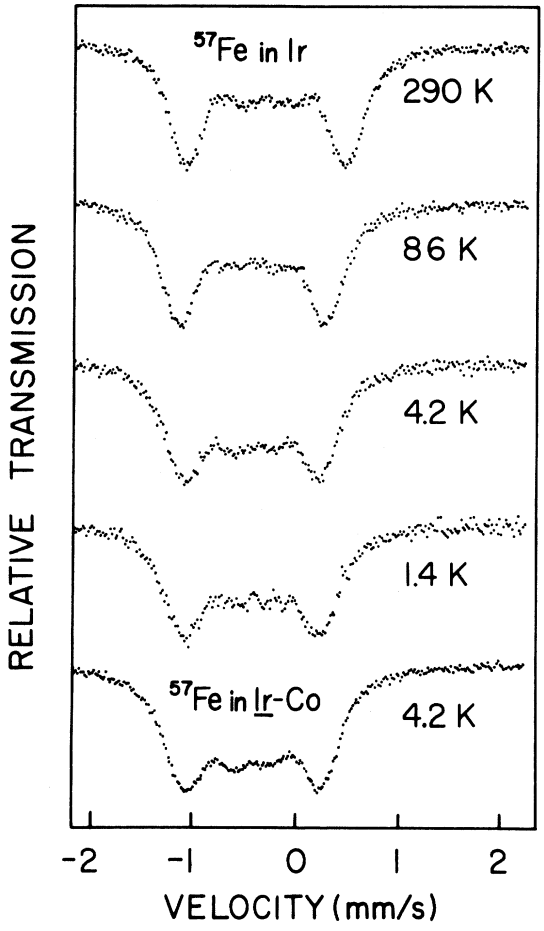

FIG. 3. Mössbauer spectra of ^{57}Fe in Ir in an external field of 50 kOe at various temperatures. The lowest spectrum is obtained with a source of ^{57}Fe in *Ir*Co (see text). The direction of γ rays is parallel to the external field.

deposition of 1-mCi ^{57}Co with nonactive Co. The diffusion treatment was carried out by the same procedure as the "pure" Ir sample. The local concentration of Co near the surface is expected to be more than 0.5 at.%. As shown in the lowest part of Fig. 3, the spectrum at 4.2 K and 50 kOe of this sample does not show any essential difference from that of the sample containing only carrier-free ^{57}Co. This result means that the anomaly in the *Ir*Fe spectrum is not caused by clustering of Co atoms in the sample, but the relaxation effect seems to be a more plausible explanation. For treating theoretically the relaxation phenomena, it is necessary to know the initial population of the electronic system. However, for LSF systems, $P(\downarrow)$ and $P(\uparrow)$ are not represented by the Boltzmann distribution, as discussed before. This makes it very difficult to obtain the spectrum by a model including the relaxation effect. An advanced theoretical work is needed to get a more definite conclusion on the anomalous Mössbauer spectrum observed for *Ir*Fe.

It is well known that the superconducting property is one of the most sensitive probes to the magnetic character of impurities. In particular, the magnetic character is remarkably reflected in the depression of the superconducting transition temperature T_0 by the impurity concerned. Recently, we have found[10] that the linear decrease in $\ln T_0$ of dilute *Tc*Fe alloys with respect to Fe concentration is consistent with the theoretical prediction for the LSF system.[25] In this respect it is interesting to compare the H_{hf} obtained by Mössbauer measurements with the depression of the transition temperature of the $4d$ Fe and $5d$ Fe systems in question. In Table II are listed the data on the initial depression of T_0 and H_{sat} (the saturation value of H_{hf}) of *Nb*Fe, *Mo*Fe, *Tc*Fe, *Ru*Fe, and *Ir*Fe systems. Since H_{sat} depends on H_{ext}, the values found under $H_{ext}=50$–60 kOe are adopted in the table.

TABLE II. Initial depression of superconducting transition temperature and saturation hyperfine field of $4d$ Fe and *Ir*Fe alloys. N is the number of outer electrons and n is the concentration of Fe impurities.

Host metal	Nb	Mo	Tc	Ru	Ir
N	5	6	7	8	9($5d$)
$-(dT_0/dn)_{n\to 0}$ (K/at.%)	0.22[a]	$\gtrsim 60$[b]	2.9[c]	0.28[d]	2.06[e]
H_{sat} (kOe)	+1[f]	−110[g]	−7.3	−1	−9.3

[a]See Ref. 26.
[b]See Ref. 27.
[c]See Ref. 10.
[d]See Ref. 11.
[e]See Ref. 12.
[f]See Ref. 3.
[g]See Ref. 22.

The magnetic character of Fe impurities in Nb ($N=5$, bcc) is simple nonmagnetic. The initial depression of T_0 and H_{sat} are both the smallest among five systems listed in the table. In contrast, Fe impurities in Mo ($N=6$, bcc) have a well-defined magnetic moment, and indeed the Kondo anomaly has been observed below 1 K.[22] Both the initial depression of T_0 and H_{sat} are the largest among those given in the table. In the Ru host ($N=8$, hcp), both values are comparable to those of the Nb host. But, as discussed before, this nonmagnetic behavior of Fe impurities in Ru does not mean that the *Ru*Fe system is a simple nonmagnetic system, but is caused by the extremely high T_{sf}. Judging from the initial depression of T_0 and H_{sat}, the magnetic character of Fe impurities in Tc and Ir lies in the intermediate region between magnetic and nonmagnetic, and they closely resemble each other. This similarity suggests that the spin-fluctuation temperature of *Tc*Fe has the same order of magnitude as *Ir*Fe.

IV. SUMMARY

The hyperfine field H_{hf} of LSF systems *Tc*Fe, *Ru*Fe, and *Ir*Fe has been measured in the temperature region of 1.4–290 K, in the external magnetic field H_{ext} up to 50 kOe. The saturation values of H_{hf} under $H_{ext}=50$ kOe are -7.3 ± 1 kOe for *Tc*Fe and -9.3 ± 1 kOe for *Ir*Fe. These values are much smaller than those of the usual Kondo systems. Combining the results of $H_{hf}(H_{ext},T)$ and of the depression of the superconducting transition temperature, a close similarity between the magnetic properties of Fe impurities in Tc and Ir is revealed. In addition, for both Tc and Ir hosts, the proportionality between the saturated value of H_{hf} and H_{ext} was observed in the present experimental region. This property closely resembles that of Kondo systems like *Cu*Fe and *Rh*Fe under the condition of $\mu H_{ext} < k_B T_K$. This resemblance suggests, as pointed out by Béal-Monod and Mills,[28] that there is no essential difference between LSF systems and Kondo systems below T_{sf} (or T_K).

For the Ru host, the observed small hyperfine field ($|H_{hf}|<2$ kOe) in the temperature region of 1.56–290 K supports the previous result, $T_{sf}>700$ K,[8] derived from resistivity measurements.

More detailed and systematic Mössbauer measurements on LSF systems are in progress under higher external magnetic fields up to 80 kOe and in a wider temperature region, 0.4–400 K.

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