Magnetic State of Cu in Ferromagnetic Ni†

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Nuclear magnetic relaxation of Cu^{63} in dilute *Ni*-Cu alloys has been measured in the temperature range 2.1–300 °K. The observed spin echo comes from nuclei in the wings of the domain walls in zero applied field and from nuclei in the saturated bulk in large external field. The longitudinal relaxation rate was found to be proportional to the temperature in both cases. We find $T_1T = 0.1$ sec °K and 1.02 ± 0.10 sec °K for zero and high external field, respectively. The difference between these rates is attributed to a mechanism involving the domain walls. Comparison of the high-field rate is made with Moriya's calculation for pure transition metal ferromagnets. It is concluded that there is little or no contribution from the *d* band to the relaxation of an isolated Cu in Ni. This strongly suggests that a Cu atom in Ni has no local moment.

I. INTRODUCTION

Nuclear magnetic resonance in the internal field of a ferromagnet provides a powerful tool for investigating the interactions in the ferromagnetic state.[1] The fluctuations in this field can be studied by observing the relaxation rate of the nuclear spin. Nuclear magnetic relaxation has been measured for the pure 3*d* ferromagnetic metals[2,3] and for some 3*d* transition impurities in these metals.[4-6] Moriya[7] has calculated the expected relaxation rates for Fe, Ni, and Co and has achieved remarkably good agreement with experiment (see Sec. V). Relaxation measurements[5] on Mn^{55} in Fe, undertaken to help clear up this complicated situation, uncovered new complexities. *Ni*-Cu was chosen for the present investigation in an attempt to extend the range of agreement with theory to this rather simple alloy system whose magnetization, for instance, is in agreement with the rigid band model up to large Cu concentrations. That Ni and Cu have similar properties is indicated by the fact that they form a complete series of solid solutions and have very similar band structures. The difference is that Ni has 0.54 holes in the $3d\uparrow$ band, while the 3*d* band of Cu is full.

The results of the present relaxation study are consistent with a picture of *Ni*-Cu, in which an isolated Cu has all, or nearly all, of its *d* states filled. This model will be developed in Sec. V.

II. EXPERIMENT

A. Samples

The experiments on pure Ni were done with Johnson-Matthey 99.999% Ni sponge as received. Of the *Ni*-Cu samples, the 2.5- and 5-at. % Cu samples were obtained in rod form from the Materials Research Corporation. The 1 and 2% alloys were melted from powders in a 4% H_2, 96% He reducing atmosphere. They were then cooled as quickly as the furnace would allow and raised to a temperature about 30 °C below the melting point for a day for homogenization. All four alloy samples were ground against 120-mesh alumina abrasive paper and strain annealed in a vacuum of about 10^{-4} Torr at 600 °C for a half-hour.

B. Apparatus

A block diagram of the apparatus is shown in Fig. 1(a). The oscillator is an Arenberg PG650 pulsed oscillator, which nominally delivers 100 W of rf power. The narrow-band preamplifier and wide-band amplifier are also made by Arenberg. The network for coupling to the sample is illustrated in Fig. 1(b). The diodes and tuned lines direct the rf power into the sample during transmission and into the receiver during reception. To achieve maximum rf power in the sample, it is matched to the 50-Ω characteristic impedance of the line. This is done by cancelling out the reactive impedance with a capacitor, thereby enhancing the input and output voltages by the Q of the circuit. The Q at 52 MHz is about 12. The inductance is chosen to satisfy $R = \omega L/Q \simeq 50\ \Omega$. With the sample resonant circuit matched to the characteristic impedance of the coaxial line leading down into the Dewar, the length of this line is immaterial.

Since the Arenberg oscillator is pulsed, it was not possible to use phase coherent detection. Instead, the precision attenuator and 40-dB amplifier shown in Fig. 1(a) were used to measure the signal amplitude accurately. The attenuator is adjusted to keep the rf power to the detector constant to within the 1-dB steps of the attenuator. Within this range, the response of the detector (found to be nearly square law) can be used to make a small correction to the attenuator reading. A boxcar integrator was used to improve the signal-to-noise ratio. Because the relaxation times are

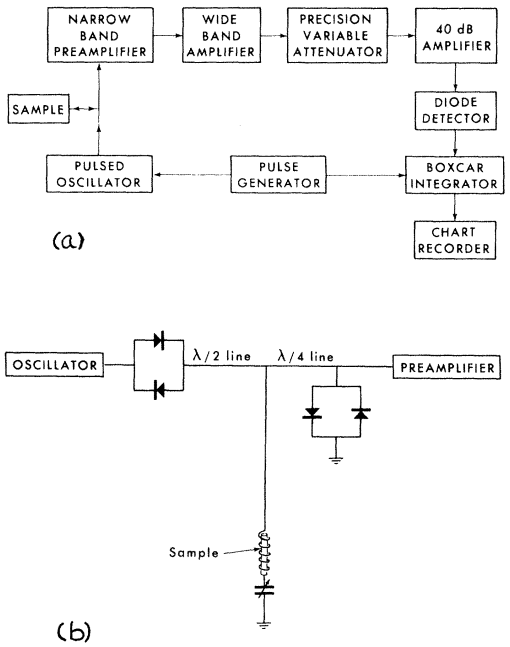

FIG. 1. (a) Block diagram of the apparatus. (b) Schematic illustration of the sample matching network.

of order 10^5 times the echo width, a digital pulse delay unit based on a 1-MHz crystal oscillator was used to accurately position the boxcar gate on the echo.

III. MEASUREMENT OF LONGITUDINAL RELAXATION IN A FERROMAGNET

A. Domain Wall and Bulk Resonance

In a multidomain ferromagnetic metal, the nuclear-resonance signal is dominated by nuclei in the domain walls.[1] This is due to shielding of the domain bulk from the applied rf field by the motion of the domain walls. In addition, the wall enhancement factor is much larger than that of the bulk.

The present study was directed toward measuring the longitudinal relaxation rate of nuclei in the domain bulk. Nuclei in the domain walls relax rapidly by processes peculiar to the wall.[8] Domain relaxation is caused by fluctuations of the interactions in the bulk and is thus of more fundamental interest. It is also the domain relaxation rate which is to be compared with Moriya's calculations, which pertain to bulk mechanisms.

The enhancement factor in a domain wall arises from the hyperfine field following the wall magnetization which is driven by the applied rf field. It is thus proportional to $d\theta/dz$,[1,9] where $\theta(z)$ is the direction of magnetization going through the wall, and so decreases going from the center of the wall to the wings. If one uses large rf fields with long pulse times, the spins in the center of the walls are driven through many rotations, which tends to cancel out the resulting echo; spins with enhancement factors such that $\theta\,\eta\gamma_n H_1\tau \approx \frac{1}{2}\pi$, where η is the enchancement factor, γ_n is the nuclear gyromagnetic ratio, H_1 is the circular component of the rf field, and τ is the pulse length, are under optimal conditions. Thus, the larger the rf field, the larger is the contribution to the signal from spins with lower enhancement, i.e., farther out of the wall. Nevertheless, the center wall signal predominates for small times. This signal relaxes fast by wall processes, which decrease in effectiveness as one goes out of the domain wall.[8] This leads to a decreasing relaxation rate as the fast relaxing signal dies away. One would expect that far enough out in the wall, the relaxation is dominated by bulk processes, leading to a constant relaxation rate and exponential decay. This behavior has been observed in the present study and is shown in Fig. 2(a). Weger[2,3] observed this behavior in Fe, Co, and Ni and considered the high-power long-time relaxation to give the "bulk" value.

One can drive out the walls by applying an external dc magnetic field. Because of crystalline imperfections which act to impede wall motion, it is possible for walls to be present in fields considerably above the demagnetizing field. One could ignore these pinned walls for the rf field parallel to the saturating field. However, the doman enhancement is very small in this configuration. No signal was observed from Cu in Ni in a dc field of several kG in the parallel configuration. In perpendicular field, it is possible that the pinned walls would be free to respond to the rf field. However, it seems unlikely that these pinned walls would be very mobile. Therefore, they do not experience much enhancement and are not able to shield the rest of the particle from the rf field. In a perpendicular field of 6 kG with an rf level, $H_1 \approx 14$G, the Cu^{63} echo in a *Ni*-2 at. % Cu sample was found to be a maximum for both rf pulses about 2 μsec long. The domain enhancement factor is $H_n/H_0 = 47/6 \simeq 8$, where H_n is the hyperfine field of an isolated Cu atom in Ni and H_0 is the applied field. These numbers yield $\theta = \eta\gamma_n H_1\tau \approx \frac{1}{2}\pi$, which indicates that the nuclei in the magnetically saturated bulk are responsible for the observed echo. Longitudinal relaxation measurement in the magnetically saturated bulk is illustrated in Fig. 2(b). The relaxation is quite exponential as a result of the unique domain bulk relaxation rate. The fast initial relaxation is due to incomplete saturation[10] because of the low domain enhancement factor. The excitation diffuses into the unsaturated part of

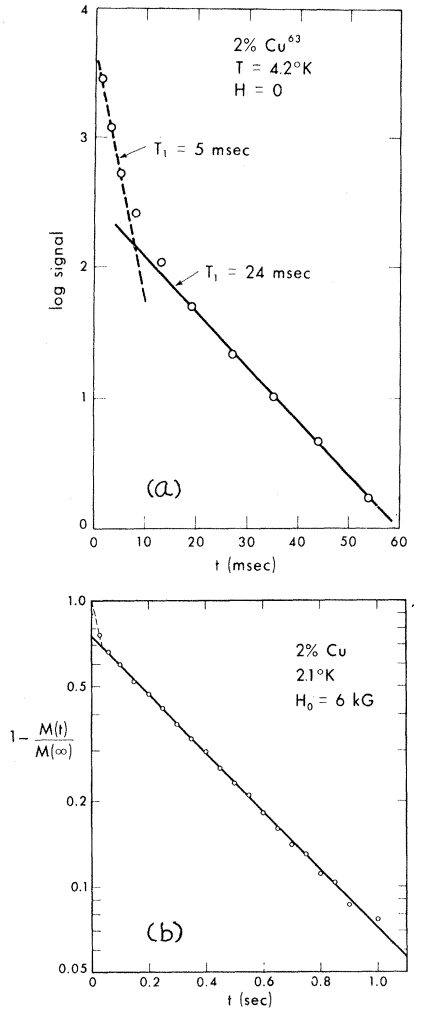

FIG. 2. (a) Log of the zero-field stimulated echo signal of Cu^{63} versus time. (b) High-field longitudinal relaxation of Cu^{63} measured by the saturation method. The relaxation is quite exponential, while at short times, effects of incomplete saturation are seen.

the line causing a rapid initial recovery of z magnetization.

Further evidence that the resonance comes from nuclei in the walls in zero applied field and in the bulk in large applied fields comes from the field dependence of the resonant frequency (Fig. 3).[11] As Portis and Gossard found for Co,[1] the curve is flat up to the coercive field, for a Ni sphere equal to $4\pi M/3 \simeq 2$ kG, while the signal intensity decreases rapidly due to driving out of the walls. At higher fields, the signal comes from saturated domains. Alignment of these domains is done at the cost of demagnetizing energy and anisotropy energy, depending on the orientation of the particle with respect to the field. For small deviations from the easy [111] direction, $H_a = 4K_1/3M \simeq 2$ kG. The particles are mostly magnetized for $H_0 \gtrsim H_a + 4\pi M/3 \simeq 4$ kG. As seen in Fig. 3, for both Ni in pure Ni and Cu in *Ni*-Cu, the frequency is linear with field above 4 kG (with the correct γ_n), indicating that the resonant nuclei reside in the bulk and feel the full applied field.

B. Method - Saturation versus Stimulated Echo

The longitudinal relaxation time T_1 can be measured in two ways. In the saturation method, the spin echo[12] is used to monitor the recovery of the z magnetization, following its saturation by a "comb" of rf pulses, typically numbering 20 of width five times the echo producing pulses. The

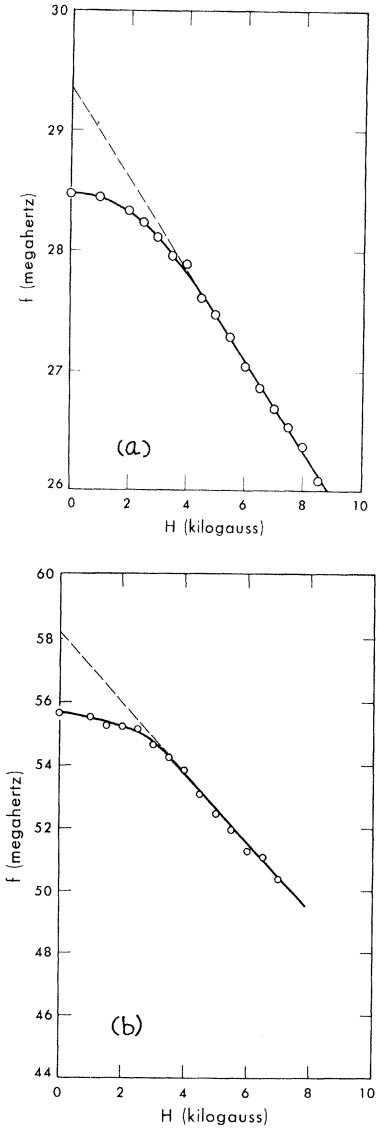

FIG. 3. Resonant frequency versus external field at 4.2 °K for (a) Ni^{61} in Ni sponge, (b) Cu^{63} in 2 at. % *Ni*-Cu.

other method uses the stimulated echo,[12] in which a pair of rf pulses, separated by τ, sets up a modulation of the z magnetization, which is allowed to decay for a time t. The extent of this modulation remaining is indicated by the amplitude of the stimulated echo observed a time τ after another rf pulse. The relaxation rate of the stimulated echo, for small τ, is given by[9]

$$1/T_{se} = 1/T_1 + 4\pi D_\nu \tau^2,$$

where D_ν is the constant for diffusion in frequency. In these experiments, the pulse separation was made small enough so that the diffusion term was less than 1% of the T_1 term. Thus the measured relaxation time can be considered the stimulated echo value of T_1.

T_1 of Ni^{61} in pure Ni was measured by these two methods as a function of external field, as shown in Fig. 4. In zero field, the results are in fair agreement with the saturation method indicating somewhat slower relaxation. On applying a static field large enough to saturate the domains, the stimulated echo rate stays essentially constant, while that by saturation shows a significant decrease. Since different relaxation mechanisms add to the relaxation rate, the smaller rate measured by saturation must be considered an upper limit on the domain bulk rate. In the event of incomplete saturation, as might occur for the low enhancement nuclei in the saturated bulks, there will be a fast initial relaxation rate due to diffusion in frequency of the excited nuclei to the unexcited one.[10] However, since this can only increase the observed rate, the saturation method still provides an upper bound on the intrinsic domain rate. Therefore, the saturation method is presumed preferable for measuring T_1, particularly in an applied field.

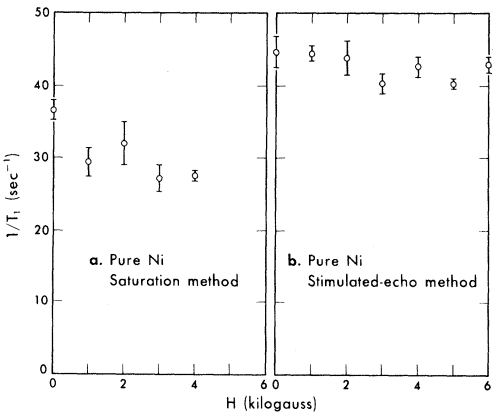

FIG. 4. Comparison of the field dependence of the longitudinal relaxation rate of Ni^{61} measured by (a) the saturation, and (b) the stimulated echo technique.

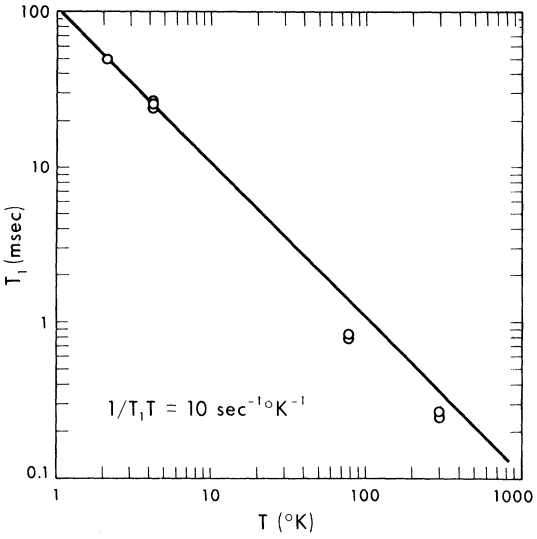

FIG. 5. Zero external field longitudinal relaxation time of the Cu^{63} in 2 at. % *Ni*-Cu versus temperature. The 45° line represents $T_1T = \text{const}$.

Similar behavior of the Ni^{61} relaxation in the 2at. % *Ni*-Cu alloy was observed by the two methods. The stimulated echo rate is larger and roughly constant as a function of field. In external fields of 3 kG or more, values of the relaxation measured by the saturation method agree with those in pure Ni, i.e., $T_1 \simeq 35$ msec at 4.2 °K.

IV. EXPERIMENTAL RESULTS

The longitudinal relaxation data previously reported[13] for Cu^{63} in a 2at. % *Ni*-Cu sample was measured in zero external field using the stimulated echo technique at 2.1, 4.2, 77, and 300 °K. The near equality of the zero-field relaxation rates found by the two methods for Ni^{61} indicates that these stimulated echo data can be taken as the zero-field "bulk" values. The data are in fair agreement with a relation $T_1T = \text{const}$ as shown in Fig. 5. The signal-to-noise ratio is better at low temperatures, so the 45° line is heavily weighted to these data, giving a value $(T_1T)^{-1} = 10$ sec^{-1} °K^{-1}.

Longitudinal relaxation of Cu^{63} is applied fields of several kG and more has been measured in four alloys: 1, 2, 2.5, and 5at. % Cu at 2.1, 4.2, and 77°K. The domain signal in a field is much weaker that the zero-field wall signal, so the relaxation was measured at 77 °K only in the 5at. % alloy, where the signal-to-noise ratio was large enough to get reasonable accuracy. In applied fields of greater than 4 kG, as previously concluded from the field dependence of the resonant frequency and the observed enhancement factor, the signal arises from bulk nuclei. In this regime, there is a small

decrease in the relaxation rate with increasing field. Although this dependence appears to be real, it is almost masked by the experimental error. This slight field dependence will be ignored, and a mean value in the 6–8-kG region will be taken as the high-field bulk relaxation rate. Similarly, constant high-field relaxation rates have been reported by Salamon[6] and Jaccarino *et al.*[14] The high-field rates, divided by the temperature, are shown in Fig. 6, as a function of Cu concentration. The error bars represent the combined uncertainty due to scatter of the data about exponential relaxation and variation in the 6–8-kG range. The data obey the relation $T_1T = \text{const}$ for each concentration within the indicated error. It is not understood why the 2% sample shows a higher rate. With no clear dependence on concentration, the measured relaxation rate is concluded to be independent of concentration up to 5at.% Cu. The mean value of this rate and rms deviation are determined by weighting the data inversely to the estimated error. The high-field relaxation rate of dilute Cu in Ni is $(T_1T)^{-1} = 0.98 \pm 0.10$ sec^{-1} °K^{-1}.

V. INTERPRETATION

Moriya[7] has calculated the expected longitudinal relaxation rate in the ferromagnetic transition metals, Fe, Co, and Ni. He concludes that the primary contribution to the relaxation is from thermal fluctuations of the orbital field at the nucleus produced by the d-band electrons, which are treated in the tight-binding approximation. Korringa relaxation by the 4s conduction electrons via the Fermi contact interaction is sometimes appreciable, while other mechanisms, such as dipolar and spin-wave related mechanisms, were found to be negligible. Since both orbital and Korringa rates, as direct processes, are proportional to the absolute temperature as well as the square of nuclear coupling γ_n, it is convenient to consider the relaxation rate in the form $(\gamma_n^2 T_1 T)^{-1} \times 10^7$, which shall be designated R. This expression, which is proportional to the square of the thermally fluctuating field at the nucleus, facilitates comparison of the relaxation rates in different materials.

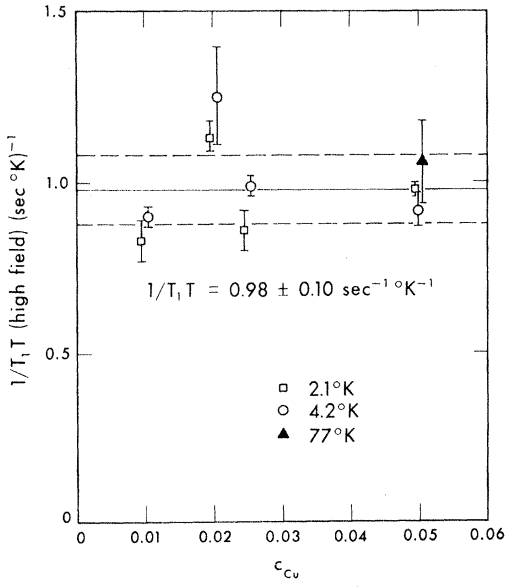

FIG. 6. Mean high-field $(T_1T)^{-1}$ of Cu^{63} in *Ni*-Cu versus Cu concentration and temperature. The dashed lines represent the rms deviation about the solid mean, with the data weighted inversely to the estimated error.

After account is taken for the mistakes in the original paper, involving the densities of states, Moriya calculated for the orbital relaxation in Ni: $R^{\text{Ni}}_{d\ \text{orb}} = 8$.[7] The value of the s-contact relaxation in Ni estimated from the known values for Cu and again corrected for the mistake in the density of states in $R^{\text{Ni}}_{s\ \text{contact}} = 0.2$. The experimental values for Ni are[15] $R^{\text{Ni}}_{H=0} = 15$, $R^{\text{Ni}}_{\text{high-}H} = 10$. Thus, in Ni, the s-contact mechanism is negligible in comparison to the d-orbital relaxation. The high-field relaxation rate which, it has been argued, is the intrinsic domain rate appropriate to the calculation is in agreement with the calculated d-orbital rate within the accuracy of the estimate, indicated by Moriya, of about 50%. Thus, the high-field relaxation in Ni can be considered satisfactorily understood.

The experimental rates for dilute Cu in Ni, given in Sec. IV, are $R^{\text{Cu}}_{H=0} = 2.0$, $R^{\text{Cu}}_{\text{high-}H} = 0.19 \pm 0.02$. The excess relaxation rate for zero field over that at high field is attributed to a mechanism related to the domain walls, whose thermal motion relaxes the nuclei in the wings of the walls, where the high-power long-time signal originates in zero field. For Cu in Ni, this wall rate, taken as the difference between the zero- and high-field rates, is 1.8 while for pure Ni it is 5. Since R is proportional to the square of the fluctuating field at the nucleus and motion of the wall is involved, it is reasonable to assume this field to be proportional to the total field at the nucleus H_n. These hyperfine fields are 75 kG for Ni and 47 kG for Cu in Ni.[16] Thus, one would expect $R^{\text{Cu}}_{\text{wall}}/R^{\text{Ni}}_{\text{wall}} = (H^{\text{Cu}}_n/H^{\text{Ni}}_n)^2 = 0.39$. Experimentally, the ratio of excess relaxation rates attributed to the wall is 0.36. This surprisingly good agreement suggests that this excess relaxation is due to rigid thermal motion of the walls.

How do the d-orbital and s-contact mechanisms contribute to the high-field Cu in Ni rate? With a naive interpretation of the rigid-band model, one might estimate the relaxation rate of Cu in Ni by inserting the pure Ni density of states at a higher ϵ_F, corresponding to the alloy's higher elec-

tron concentration, into the expressions for the d-orbital and s-contact contributions to the relaxation. Since $N_d(\epsilon)$ varies rapidly in energy, the possibility that the small rate for Cu in Ni reflects a dip in $N_d(\epsilon_F)$ must be considered. The concentration independence of the low rate argues against this, as one would have to assume that $N_d(\epsilon_F)$ drops abruptly with 1 at.% Cu and then remains nearly constant up to 5at.% Cu. Furthermore, the Ni relaxation rate was measured in high field in the 2at.% Cu alloy and was found to be close to the pure Ni high-field value. This indicates a considerable d density at the Ni sites in the alloy.

This result is readily understandable if one considers the screening of an isolated Cu impurity in Ni. The Cu, because of its greater Coulomb potential, will attract an extra electron into its neighborhood. Because of the high density of states in the d band of Ni, the screening will be done approximately 90% by the s band. However, since the Ni atoms have only 0.54 d holes, the Cu can accommodate only 0.54 of the 0.9 d screening electrons before its d states are filled up. The remaining 0.36 screening d electrons are forced out to the Ni neighbors, thereby reducing their moments. It can be assumed the Ni neighbors whose resonant frequency is drastically shifted by this diminution of moment are not observed in the main Ni resonance. Similarly, the density of states, and hence relaxation rate, of the Ni nuclei left in the resonance line will not be much affected by the presence of the Cu, as is observed.

In this screening picture, there is no possibility of d-orbital relaxation, involving fluctuations of the orbital field at the nucleus, as the d states of the Cu atom are continually filled. Thus, only s-contact relaxation from the conduction electrons is allowed. The expected s-contact relaxation rate can be estimated from the observed relaxation rate in pure Cu, which can be accounted for in terms of this mechanism.[17] Rotary saturation measurements give[18] $T_1T = 1.27 \pm 0.10$ sec °K, or $R_{\text{expt}}^{\text{Cu in Cu}} = 0.16 \pm 0.01$.

This is close to the present $R_{\text{high-}H}^{\text{Cu in Ni}} = 0.19 \pm 0.02$. However, one would expect the s-contact contribution to be smaller in *Ni*-Cu because of the smaller number of s electrons. From the previous screening considerations, one can take the number of s electrons affecting the Cu as 0.54 s electrons of pure Ni plus 0.1 s electrons in the screening. One may then crudely assume the relaxation produced by this 0.64 s electron scales as a free-electron gas, i.e., $(T_1)^{-1} \propto [N_S(\epsilon_F)]^2 \propto n^{2/3}$. Further, assuming $\langle |\psi(0)| \rangle_{FS}$ to be the same as in pure Cu, the s-contact relaxation rate for Cu in Ni is predicted to be 74% of the pure Cu rate. Experimentally, it is (119 ± 20)%, leaving $R_{\text{excess}} \simeq 0.07$ to be explained.

This excess could be explained by assuming that more of the screening is done by the s band, although this seems unlikely as it would involve higher energy states. It also seems unwarranted because the calculation of the s contribution involves the local density of s states at the Cu impurity, which requires more careful handling of the screening problem. The other obvious possibility is that there is some d-orbital relaxation due to incomplete filling of the d states of the Cu. As previously mentioned, the observed relaxation is an upper limit, so R_{excess} is a factor-of-2 estimate of an upper bound on the d contribution. Comparing $R_{\text{excess}} \simeq 0.07$ to the d-orbital contribution in Ni, $R_{d\ \text{orb}}$, and considering the large density of d states in the alloy which would strongly scatter with any d holes on the Cu impurity, it is clear that there is little if any Cu moment.

The same conclusion has been drawn from nuclear relaxation studies of some analogous paramagnetic systems, *Pd*-Cu,[19] *Pt*-Cu,[20] and *Pd*-Ag,[21] using a model similarly based on screening considerations and the large d-band density of states. Itoh *et al.* have found [19,20] T_1T of Cu^{63} constant over all concentrations and temperatures investigated in Pd and Pt alloys, respectively. They do not indicate how T_1 was measured or what rf power level was used, so it is possible that they experienced the difficulties found by Narath[10] in pure Pd, which can lead to spuriously fast relaxation. The only Cu relaxation data given by Itoh *et al.*,[19] $T_1 = 220 \pm 10$ msec at 1.5 °K for *Pd*-Cu with $0.01 \leq c_{Cu} \leq 0.95$, implies a Cu relaxation rate nearly four times that[18] of pure Cu over the whole concentration range in spite of changes in the density of states.

Pd-Ag is the closest analog of *Ni*-Cu, and Narath[21] has measured T_1 of Ag^{109} over the whole range of concentration for temperatures 1–4 °K, taking care to achieve saturation. The data are nicely explained by the previously mentioned considerations. T_1T of Ag^{109} is nearly constant up to 50 at.% Ag, in which the added electrons are going mainly into filling the d band. This indicates that the d electrons are ineffective in relaxing the Ag nuclei. Above 50 at.% Ag, as the s density of states increases rapidly after the d band is filled, T_1T decreases and goes smoothly to the pure Ag value. The conclusion that there is little d contribution ot the Ag relaxation in dilute *Pd*-Ag is supported by the large ratio $R^{\text{Pd in Pd}}/R^{\text{Ag in Pd}} \simeq 40$. The analogous expression $R_{\text{high-}H}^{\text{Ni}}/R_{\text{high-}H}^{\text{Cu in Ni}}$ is found, from the present work, to be about 50, which indicates a close similarity of the two alloys. The quantitative difference is that $(\gamma_n^2 T_1 T)^{-1}$ of dilute Ag in Pd is less than that of pure Ag scaled by the assumed number of s electrons, whereas for Cu in Ni, a

small excess relaxation rate was found, as discussed previously.

VI. CONCLUSION

Nuclear magnetic relaxation has been measured for dilute Cu^{63} in Ni; for nuclei in the wings of the domain walls in zero external field, and those in the saturated bulk in high external field. The excess longitudinal relaxation in the former case as compared to the latter is probably due to fluctuations of the hyperfine field resulting from thermal motion of the domain walls. The domain longitudinal relaxation rate can nearly be accounted for by s-contact relaxation by the 4s bands. This indicates that there is little or no density of 3d states at an isolated Cu impurity in Ni, which implies that there is little or no d moment on the Cu.

Streever[4] has measured longitudinal relaxation in zero external field of dilute Ni impurities in Fe and Co and dilute Co in Ni. In each of these cases, he finds the relaxation rate $(\gamma_n^2 T_1 T)^{-1}$ of the impurity to be less than that of the host. This is also found in the present experiment. However, the explanation in terms of the absence of d density of states at the impurity would not be expected to hold for the case of Co in Ni. Although Streever's measurements were made in zero external field and thus may include wall-relaxation mechanisms, his value $R_{H=0}^{\text{Co in Ni}} = 0.85$ is considerably less than the calculated d-orbital contribution $R_{d\,\text{orb}}^{\text{Ni}} = 8$. If such a low domain relaxation rate were comfirmed by measurements in a large external field, it would be necessary to find a more general explanation than the present screening argument for the reduced spin-lattice relaxation of an impurity in a ferromagnetic alloy.

Nevertheless, the decrease of the Cu^{63} longitudinal relaxation rate in high field to a value so close to the expected Korringa s electron rate suggests an absence of d-band fluctuations. It would be interesting to measure the relaxation rates of Ag and Au impurities in Ni to see if the results agree with the predictions of a simple screening picture. However, in addition to the experimental difficulties due to the small nuclear moments of Ag and Au, the interpretation would be complicated by considerations of atomic size.

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