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Temperature Dependence of the Hall Effect in Dilute Alloys of the Group-1B Metals, I. Case of the Intermediate-Field Condition

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The temperature dependence of the Hall effect in the range 6-80 K has been determined in monocrystalline samples having \tilde{H} along principal crystallographic directions. Samples of Au and of Cu containing Fe, Mn, or Ni (4-24 at. ppm) were studied in experiments encompassing the intermediate-field condition. These data for monocrystals are compared with those obtained in earlier work for Cu and Cu containing Zn. The comparison supports the validity of the earlier qualitative interpretation of the temperature dependence, and also shows how a more detailed understanding of the behavior observed in polycrystals can be obtained from the observed changes in behavior of the various contributing cyclotron orbits as solute is added.

I. INTRODUCTION

The Hall effect in alloys is generally studied in polycrystalline samples. The aim of this paper is to show how a study of the temperature dependence of the Hall effect in monocrystalline samples improves the understanding of the behavior shown in the corresponding polycrystalline cases. We have previously described¹ a similar study of monocrystals of Cu and Cu containing Zn, and in the present work we extend this to Au and to Cu containing Ni, Fe, or Mn. Just as the earlier results remain apparently the only ones available where H (the applied field) is along principal crystallographic directions, so we know of no others obtained for monocrystals (oriented or otherwise) comparable to those described below.

The reasoning behind this work is as follows. First, since there are certain unexplained details in the behavior previously observed in a Cu monocrystal,¹ it is desirable to confirm that the gross features observed there are indeed typical of a group-1B metal. A comparative study in another noble metal would obviously be helpful, and we have chosen Au. These results are presented and discussed in Sec. III. Second, it is known² that in the class of alloys consisting of a group-1B metal containing a transition-metal solute there appears at low temperatures an anomalous Hall effect which, although evidently associated with the magnetism of the solute, is not well understood.^{2,3} We felt that a comparative study of the effects of magnetic and nonmagnetic solutes upon the electronic motion in the various bands of contributing orbits might illuminate this problem. Consequently we have extended the work on monocrystals of Cu containing Zn to include the additions cited above, and these results are presented and discussed in Sec. IV.

It will be helpful to have an over-all view of the characteristic behavior we are trying to elucidate. This is given schematically in Fig. 1, which shows the typical temperature dependence observed for the Hall effect at a fixed field in dilute polycrystalline alloys of the class under discussion. A is the behavior observed for the pure solvent, while $B \rightarrow$ H represent alloys of increasing concentration (typically up to ~ 2000 at. ppm) but restricted such that we can assume that no appreciable effects due to either solute-solute interactions or solvent-bandstructure changes are manifested. (The temperature range covered in Fig. 1 is typically ~ 4–100 K.) For the most dilute samples the same gross features are observed for both magnetic and nonmagnetic solutes (A \rightarrow E of Fig. 1): The magnitude of the Hall effect in the lowen temperature presion is

tures are observed for both magnetic and nonmagnetic solutes $(A \rightarrow E \text{ of Fig. 1})$: The magnitude of the Hall effect in the lower-temperature region is generally reduced by the addition of solute until (for the nonmagnetic solute at least) a relatively concentration-independent temperature dependence is observed. In the moderate fields we envisage for Fig. 1 (up to about 30 kOe), the Hall effect is field independent in this condition. (An example of this can be seen in the Ag-based alloys discussed by Alderson and Hurd.⁴) In the case of the magnetic solute, further additions produce a reversal in the behavior and lead to an increase in the magnitude of the Hall effect at the lower temperatures $(F \rightarrow H \text{ of Fig. 1})$. This contribution is field dependent; it is the spin contribution⁵ and has an anomalous component referred to above.

In this paper we shall be concerned only with the range of concentrations represented by $A \rightarrow E$ which, as will be shown, is the intermediate-field condition and the range in which extrapolation from the monocrystalline case is meaningful. We have also studied several alloy systems under the conditions $F \rightarrow H$ of the right-hand side of Fig. 1, but these conditions are entirely low field (see Sec. III) where the monocrystalline results, which relate to the particular dynamics of electrons in the different bands of contributing orbits, are irrelevant.



FIG. 1. Schematic representation of the gross features of the temperature dependence of the Hall effect (Hall resistivity ρ_{Hall} or coefficient *R*) in polycrystalline alloys of the class under discussion. A is the pure solvent, and $B \rightarrow \text{H}$ alloys of increasing concentration. The temperature range envisaged is ~ 6-80 K and the fixed field strength is assumed to be moderate, say <~ 35 kOe.

Since the data of $F \rightarrow H$ refer to the special problem of skew electron scattering,³ it is convenient to 'present them in a separate publication.⁵

II. EXPERIMENTAL

The specimens were prepared by the Bridgman method exactly as described previously for Cu and Cu containing Zn.¹ The starting materials were Cu and Au of 99.9999% purity (supplied by American Smelting and Refining Co. and Consolidated Mining and Smelting Co., respectively) and Ni, Mn, and Fe of 99.99% or equivalent purity (supplied, respectively, by Materials Research Corp., King Products, and the Battelle Memorial Institute). Some typical chemical analyses of the starting noble metals have been given elsewhere^{6,7} while the residual-resistance-ratio (RRR) values ($R_{273 \text{ K}}$ / $R_{4,2K}$) of the samples measured after preparation are shown in the accompanying figures, together with the estimates (in atomic ppm) of the solute concentration in the alloys provided by the Analysis Section of NRCC from off-cuts of each end of the sample.

The samples were spark-cut from prepared monocrystalline sheets (typically 0.08 cm thick) using a template having the dimensions specified previously.⁸ In all cases the sample's orientation put the primary current (10 A) along [110] while \vec{H} (15.17 kOe) could be positioned empirically along the chosen directions indicated in the stereographic triangle of Figs. 2 and 3. The instrumentation and techniques used to determine the Hall voltage are identical to those described previously.^{7,8}

III. MONOCRYSTALLINE Cu AND Au

Figure 2 compares the temperature dependence of the Hall resistivity (ρ_{Hall}) observed in the range 6-80 K for equivalent orientations of \vec{H} (15. 17 kOe normal to the plane of the sample) in Au and Cu. (The data for the latter are taken from Ref. 1.) It is seen that the same general behavior is observed in both cases. Since the two metals have Fermi surfaces of equivalent topology, our qualitative interpretation¹ of the gross features in Cu can be applied equally to Au. It is unnecessary to repeat all the details of that interpretation here; we shall simply draw attention in the following to those points of difference or similarity between the data of Fig. 2 which deserve special mention.

To see the general behavior, however, it is useful to recall that the experiments of Fig. 2 encompass the intermediate-field condition, i.e., that condition which does not approximate closely to either the low- or high-field limits. Physically, some of the electrons at any given temperature are "high field" and describe cyclotron orbits for which $\omega \tau > 1$ (in the usual terminology), while others, because of the anisotropy of the phonon scat-



FIG. 2. Temperature dependence of the Hall resistivity determined for the indicated orientations of a fixed field (15.17 kOe). The primary current is along [110] in all cases. The data for Cu and the polycrystalline samples, included here for comparison, are taken from Refs. 1 and 8. The symbols indicating specific orientations refer to the unit stereographic triangle of Fig. 1 and are maintained throughout Figs. 2–5. RRR is the residual resistance ratio.

tering over the Fermi surface, remain "low-field" $(\omega \tau < 1)$ and cover an insignificant part of a cyclotron orbit during their lifetime. There is no theory developed for this region so that at best we can hope only for a qualitative interpretation of the data.

For temperatures above about 50K the phonon scattering reduces $\langle \tau \rangle_{av}$ (where the average is over all contributing orbits for an orientation) to the point where all orbits are apparently in the lowfield condition; ρ_{Hall} is consequently isotropic with a magnitude determined by an integral over the Fermi surface involving the electron's mass, velocity, and (anisotropic) τ at each point.⁴ As the temperature is reduced, the onset of the high-field behavior is first manifested for certain groups of orbits in planes normal to [100] and [110] (as at B in Fig. 2), while further reduction in temperature subsequently produces the same effect for the other orientations studied (as at A). Continuing reduction of temperature leads ultimately to the impuritydominated region, which extends below about 10 K in Cu and from a somewhat lower temperature in Au, and this is the closest approximation to the high-field condition which the experimental circumstances permit. In the high-field limit $\rho_{\rm Hall}$ is determined solely by the topology of the metal's Fermi surface.

It is important to note that a small-angle scattering process (such as by a low-energy phonon, for example), which only takes the electron from one point to another on the same orbit, does not destroy the orbit's high-field characteristic for the Hall effect. (This contrasts with the more stringent requirements of a quantum effect—such as the dHvA effect—where the loss of quantum phase coherence around the orbit means that any scattering process automatically terminates the electron's "high-field" lifetime.) This fact is presumably important in accounting for the observation from Fig. 2 that high-field effects occur to relatively elevated temperatures, viz., to at least 35 and 50 K in Au and Cu, respectively.

Although the same general behavior is observed for Cu and Au in Fig. 2, there are quantitative differences which require attention. First, whereas A and B correspond, respectively, to about 37 and 45 K in Cu, they occur at about 24 and 35 K in Au. (It should be emphasized that these temperatures are a function of the material and not of the sample. In the presence of such overwhelming phonon scattering a small variation in purity between samples has an insignificant effect upon the temperature of the high-field-low-field transition. This can be seen from Fig. 4, where the temperature corresponding to B is evidently independent of even quite appreciable additions of solute to Cu.) In other words, the data of Fig. 2 show the expected result that at any given temperature above the impuritydominated range the electron scattering by phonons in Au is relatively more effective than in Cu, as would be expected from the relative stiffnesses of the two lattices reflected in their differing Debye temperature (Θ_D).

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In an attempt to see through these lattice effects and to observe the manifestations in ρ_{Hall} of the metal's intrinsic electronic properties, we show in Fig. 3 the data of Fig. 2 replotted on a reduced temperature scale T/Θ_p (Θ_p values taken from Meaden).⁹ Ideally, this should bring into coincidence equivalent phonon contributions, and this assumption is supported by the effective superposition of the data for \overline{H} along [112] (solid triangle symbol). This orientation is unique among those studied in that the temperature dependence of the Hall effect is determined simply by the conductivity of longitudinal open orbits in the sample (see Fig. 6 of Ref. 1). These orbits contain electrons which are unable to respond to the Lorentz force and they provide a short circuit of the Hall effect which would be complete in the high-field limit. In the intermediate-field condition, however, the effectiveness of this short-circuit depends upon the electron's mean free path (i.e., upon the amount of phonon scattering in this case) and consequently, for metals such as Cu and Au having equivalent Fermi-surface topologies, the observed superposition of ρ_{Hall} would be expected in any plot which superimposes equivalent phonon contributions.

Turning to the behavior observed for orientations having negligible open orbit contributions, Fig. 3 shows that the onset of the high-field effects for \overline{H} along [100], [110], and [111] occurs in each case at a reduced temperature which is appreciably higher in Au than in Cu (this being a reversal of the situation depicted in Fig. 2). Since Fig. 3 is thought to eliminate differences arising from phonon effects, we must look to the electronic properties for an explanation. This is presumably found in the fact, known from dHvA studies, ¹⁰ that the cyclotron masses obtained for extremal orbits are in all cases greater in Cu than in Au. If we can assume that these extremal cases are representative of their associated band of orbits contributing to the Hall effect, ¹ the cyclotron frequency of contributing orbits will therefore be higher in Au than in Cu in the same experimental circumstances.



FIG. 3. Data of Fig. 2 replotted on a reduced temperature scale T/Θ_D , where Θ_D is the Debye temperature indicated.

Consequently, phonon contributions being equal in Fig. 3, the greater tendency towards high-field behavior (where $\langle \omega \tau \rangle_{av} > 1$) at a given reduced temperature observed in Au is consistent with the known electronic properties of the metals.

Finally, we turn to the open triangle and open square orientations of \vec{H} which both show an unexpected behavior in the earlier work.¹ (The same symbols used for the orientations of \vec{H} in Ref. 1 are maintained throughout the figures of the present paper.) For the open triangle orientation Fig. 2 shows evidence of a contribution from holelike orbits below about 20 K in Au, just as was observed for Cu.¹ (Figure 4 confirms that this contribution is lost to the low-field condition as solute is added—just as in the case of additions of Zn.¹) Taken together, these results strongly support the earlier result that this orientation does not support simply electron orbits but has another, holelike contribution.

A detail relating to the open square orientation is also worth noting. This was previously thought to have no contributions from effective open or

easily quenched hole orbits,¹ and reference to Fig. 4 shows that the alloy data support this view: It is again the orientation which shows relatively the least change in the Hall effect upon the addition of solute, and its monotonic temperature dependence is apparently the prototype to which those shown by all except the [110] and [100] orientations tend as the concentration is increased. But a detail from Fig. 2 upsets this interpretation for Au at least since, unlike Cu, the temperature dependence observed there is not monotonic. It is impossible to say at present whether this illustrates some fault in the interpretation given for Cu or is simply a real difference in behavior between the two metals arising from differences between their Fermi surfaces.

IV. MONOCRYSTALLINE Cu-BASED ALLOYS

Figure 4 shows a comparison of the temperature dependences of ρ_{Hall} observed in the range ~ 6-80 K for equivalent orientations of \vec{H} (15.17 kOe) in

Cu containing the indicated concentrations of Ni, Mn, or Fe. With the exception of Cu + 21-ppm Fe (and the Cu+176-ppm Zn of Ref. 1), the total scattering induced by the solute is generally negligible compared with the phonon scattering for temperatures above about 30 K in the examples studied. Consequently, above this temperature the results of Fig. 4 are essentially identical with those of pure Cu (Fig. 2) and are thus not of great interest. It is worth noting, however, that the temperature corresponding to B of Fig. 2 is the same (~ 50 K) in these data, whatever the solute (the CuZn data of Ref. 1 can also be included in this statement). This indicates that even though there are significant mass differences between the solutes, the lattice dynamics are not sufficiently affected at these concentrations to show an appreciable change in Θ_p from the pure Cu.

A second notable feature is that the qualitative behavior observed in Fig. 4 is identical to that seen previously upon addition of Zn, and is super-



FIG. 4. Data obtained as in Fig. 2 except that here the samples contain the indicated solutes in the concentrations shown in at. ppm.

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ficially independent of whether or not the solute is magnetic: For temperatures below about 50 K the temperature dependences of ρ_{Hall} observed for the various orientations fall into two classes. The first gives a monotonic increase in $|\rho_{\text{Hall}}|$ as the temperature is reduced, and is typical of \vec{H} along [100] or [110], while the second has a local extremum at ~ 50 K and gives a subsequent decrease in $|\rho_{\text{Hall}}|$ as the temperature is reduced. As the total amount of scattering is increased at a given temperature (i.e., as the RRR decreases), so this segregation of the temperature dependences into two classes is seen to become more pronounced (compare the CuNi and CuMn data) while simultaneously the two types of dependence move closer to a superposition. (See, for example, the Cu + Zn data in Fig. 3 of Ref. 1.) This superposition is the concentration-independent temperature dependence represented by $E \rightarrow H$ in the left-hand side of Fig. 1. It occurs, of course, when the scattering at any temperature is great enough that no orbit can make a detectable high-field contribution in the given experimental circumstances.

We can now see the relevance of Fig. 4 to the interpretation of the temperature dependence of ρ_{Hall} in corresponding polycrystalline samples. This dependence will clearly be some average of all behaviors such as shown in Fig. 4, but we have already commented¹ that the weight of certain orientations of H in this average is apparently very small. For example, with \overline{H} along [111] we find an enormous value of $|\rho_{Hall}|$ when the band of sixcornered rosette orbits is in the high-field condition (Fig. 2), but no appreciable change in the polycrystalline result occurs when this band is removed to the low-field condition by a very small addition of solute. From similar arguments applied to other orientations of \overline{H} , we have deduced¹ that of the singular topological features associated with the open Fermi surface of Cu only orbits of the four-cornered rosette and dog's bone type contribute significantly to the high-field Hall effect in a (presumably) random polycrystal of Cu. It is these singular features which give the monotonic temperature dependence of ρ_{Hall} of the first class referred to above. In addition to the contribution from these singular features there is, of course, a contribution from the usual, closed electron cyclotron orbits, and it is these "nonsingular" features which give the second class of behavior having the local extremum referred to above.

The origin of the behavior we set out to illustrate (curves $A \rightarrow E$ of Fig. 1) can now be understood qualitatively as follows. (This argument follows exactly that given previously for the Cu + Zn data.¹) Curve A evidently arises from an average of contributions such as in Fig. 2. Upon addition of a very small amount of solute there is a drastic change in the low-temperature contributions arising from the more vulnerable orientations of H (such as when it is along [111] or [211]) but no appreciable change in the value of ρ_{Hall} for the corresponding polycrystal because of the very low statistical weight of these orientations.¹ (Such a situation is illustrated by the CuNi data of Fig. 4; only the behaviors for \vec{H} along [111] and [211] are greatly changed from those observed in pure Cu.) A further addition of solute emphasizes the segregation into the two classes of behavior which we defined above (illustrated by the CuMn data in Fig. 4) but with an important detail: Whereas that class showing a local extremum tends towards the relatively fixed behavior of the open square orientation, the monotonically varying class is quantitatively more affected by the addition. (Compare the change of these two classes across the series of alloys $CuNi \rightarrow Cu + 4$ -ppm Fe $\rightarrow CuMn \rightarrow Cu + 21$ -ppm Fe of Fig. 4; $|\rho_{Ha11}|$ at low temperatures is continuously reduced for the monotonically varying class whereas in the other class it remains relatively fixed.) This reduction in $|\rho_{\text{Hall}}|$ arises from the increasing loss of contributing hole orbits to the low-field condition as solute is added, ¹ and it is reflected directly in the polycrystalline results; it is the origin of the behavior shown by the curves A - E of Fig. 1. As we pointed out above, curve E represents the case when sufficient solute is added that no high-field contributions can be made to ρ_{Hall} in the given experimental circumstances, and in terms of monocrystalline results it represents the superposition of the two classes of temperature dependence described above.

In the preceding we have not considered the fact that some of the solutes are magnetic while others are not. It has not been necessary to do this with reference to Fig. 4 because the qualitative interpretation we have given is gross enough to be concerned only with the total amount of scattering introduced by a given concentration of solute. Fine details, such as the effects of any variation between solutes in the anisotropy of their scattering over the Fermi surface, cannot be observed in such as Fig. 4 unless equivalent intermediate-field conditions exist in the different experiments. Figure 5 shows a case in which we have attempted to do this. It compares the temperature dependence (at \overline{H} = 15.17 kOe) obtained for Cu + 4-ppm Fe (of Fig. 4) and Cu + 172-ppm Zn (of Ref. 1). The two cases have almost equivalent amounts of solute scattering (the RRR values are, respectively 240 and 263), and since the above evidence is that solute mass differences have no appreciable effect upon the phonon scattering at this dilution, we can reasonably assume that equivalent $\langle \omega \tau \rangle_{av}$ values apply to the two alloys at any given temperature. We can therefore attribute any observed gross differences to the

intrinsic anisotropy of the solute scattering. Such gross differences are observed only between the behaviors for \mathbf{H} along [110] and [100]: At any given temperature below about 45 K the *Cu*Fe sample shows appreciably less high-field contribution from electron orbits than does the *Cu*Zn. [To see this, compare Eq. (3) and the associated discussion in Sec. 4 of Ref. 1.] In other words, Fig. 5 indicates that electron scattering from belly states is greater with Fe than with an equivalent amount of Zn added in identical conditions.

It is known¹¹ that the regions of the Fermi surface having a high *d*-symmetry content are localized about the $\langle 110 \rangle$ directions, being extended somewhat more towards [100] than [111], and it can be seen that the bands of electron orbits which contribute to ρ_{Hall} for both the [100] and [110] orientations traverse these regions (Fig. 5 of Ref. 1). Consequently, the gross differences observed in Fig. 5 are consistent with the known anisotropies of scattering by Zn and Fe; the relatively high scattering for belly states shown by the Fe reflects the stronger *d* component in its scattering potential.

V. SUMMARY AND CONCLUSIONS

The usefulness of the new results presented above for a monocrystal of Au is mostly indirect: They clearly support the earlier data obtained¹ for Cu, and consolidate the qualitative interpretation of the observed temperature dependence of ρ_{Hall} suggested there, but they lead to no new insights in this area. The results for the monocrystalline alloys enable one to obtain a somewhat clearer understanding of the origins of the temperature dependence of ρ_{Hall} in polycrystalline alloys in that they indicate quite clearly the principal types of contributing cyclotron orbits and their typical behavior when solute is added. In one particular case we have been able to show what is probably evidence of the effect of different solute scattering anisotropies-something we tried unsuccessfully to do earlier¹² from the field dependence of the Hall effect observed at 4.2 K in some of the alloys of Fig. 4.

This paper is concerned exclusively with very dilute alloys in which the experimental circumstances produce the intermediate-field condition. For slightly more concentrated alloys, where the lowfield condition is maintained throughout the experiment, the anomalous behavior indicated by curves $F \rightarrow H$ of Fig. 1 is known to occur when the solute is magnetic, and we present corresponding results obtained in this region in the following publication.⁵

We have assumed throughout that the alloy is sufficiently dilute to avoid appreciable effects from solute-solute interactions or changes in band structure. What the nature of these effects would be is not evident *a priori*, although there are results which suggest that in some alloy systems of the



FIG. 5. Comparison of the temperature dependence of the Hall resistivity observed as in Fig. 2 for two alloys with essentially equivalent amounts of solute scattering. The gross differences are attributed to the intrinsic anisotropy of the solute's scattering. CuFe data taken from Fig. 4; CuZn data from Ref. 1.

class we are discussing (AgLi and CuAu, for example^{13,14}) they give competitive contributions to the Hall effect. This conclusion derives from the concentration dependence of the effect which is found to be nonmonotonic and may even show fine structure in the very dilute range. AgLi is such a case, ¹³ and we can qualitatively understand from the foregoing the origin of this fine structure. The addition of 0.74 at.% Li is found to produce a reduction in the magnitude of the Hall effect, while further addition (1.7 at. %) gives a resurgence followed subsequently by a diminution (at 3.1 at. %) which continues apparently to the α -phase limit. From the foregoing, we suggest that the initial reduction produced by the 0.74 at. % Li corresponds to the coverage of the range A - E of Fig. 1, being the transition to the low-field condition at lower temperatures. The subsequent behavior as solute concentration is increased would be attributed to the unspecified (but competitive) contributions arising from solute-solute interactions and the band-structure changes. It is this competition which leads to the local extremum at about 2.0 at. %Li in the concentration dependence of the Hall effect.

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Temperature Dependence of the Hall Effect in Dilute Alloys of the Group-1B Metals. II. Case with a Magnetic Solute

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The temperature dependence of the Hall effect in the range 6-80 K has been determined in polycrystalline alloys of the CuFe, AgMn, CuMn, and AuFe systems. The solute concentrations range between 4 and 500 at. ppm. A qualitative interpretation is given of the gross features observed in these samples, and the argument is then widened to the general problem of the extraction of the spin component in dilute alloys of this class. The work is an extension of the equivalent study of monocrystalline samples reported by us in the preceding paper.

I. INTRODUCTION

In the preceding paper¹ we discussed the temperature dependence of the Hall effect observed in very dilute alloys of the class consisting of a polyvalent solute dissolved in a group-1B metal. That paper was restricted to alloys of such dilution that the experiment encompassed the intermediate-field condition in which the results from monocrystalline alloys we reported were relevant to the interpretation of those from corresponding polycrystals. As we indicated (Fig. 1 of Ref. 1), in the intermediatefield condition there is an essentially similar behavior observed for the temperature dependence of the Hall effect in very dilute polycrystalline alloys having either a magnetic or a nonmagnetic solute. But characteristic differences do arise when the solute concentration is increased so that the experiment encompasses entirely the dynamical low-field condition²; the principal feature is the appearance

of a spin component in the Hall effect when the solute is magnetic, part of which is anomalous in terms of our present understanding^{3,4} in that it probably does not arise from the Lorentz force.

It is the purpose of the present paper to show new results for the temperature dependence of the Hall effect when the anomalous component is present, and in this respect the present work is the extension of the preceding¹ into the entirely (dynamical) low-field condition. Not only is the physical origin of this anomalous component controversial, ^{3,4} but so also is its correct extraction from the total measured effect. These new data bear upon the latter problem, but it is not our aim here to make a quantitative study of the spin component (our data are not sufficiently extensive in either range of concentration or applied field strength to permit this) but rather to illuminate by inference drawn from the data the typical behavior of the Hall effect observed in the above class of alloys having a magnetic solute,