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AC susceptibility of PdNi through the ferromagnetic percolation threshold

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Abstract. Detailed measurements of the field and temperature dependent AC susceptibility of a number of **Pd**Ni alloys containing between 2 and 5 at.% Ni are presented. In ferromagnetic samples attempts are made to analyse these data for Curie temperatures and critical exponent values. It is demonstrated, however, that spin–orbit induced anisotropy associated with a Ni orbital moment precludes reliable estimates for exponents, although Curie temperatures can be determined. Despite such difficulties these data are shown to be consistent not only with a previous suggestion that a spin-glass ground state would appear at low Ni concentration if complications due to the Kondo effect were not present, but also with assigning a low spin value ($S = \frac{1}{2}$) to the Ni site.

1. Introduction

Available experimental data indicate unequivocally that amongst the 3d transition metals Ni is unique in the sense that when dissolved in a non-magnetic host it invariably exhibits non-magnetic behaviour in the dilute limit (see, for example, Fischer 1982). The theoretical interpretation of the latter is, however, still contentious. The differing model approaches are best illustrated in the case of dilute **Pd**Ni in which the Ni impurities have been variously described as being (i) non-magnetic due to a failure to satisfy the static Hartree–Fock criterion for magnetism, but where strong local exchange enhancement effects enable dynamic localised spin fluctuations to take place (Lederer and Mills 1968, Kaiser and Doniach 1970, Blandin 1973), (ii) non-magnetic at low temperature due to interconfigurational fluctuations between nearly degenerate d^9 (magnetic) and d^{10} (non-magnetic) many-electron configurations at the Ni site (Hirst 1970; Williams 1976), or (iii) the result of Kondo compensation of a single energetically stable, magnetic many-electron Ni configuration, below a moderately high Kondo temperature $T_K \simeq 55$ K (Loram and Mirza 1985).

Away from the dilute limit, interaction effects in **Pd**Ni are known to result in the occurrence of long ranged ferromagnetism above a critical composition $c_0 \simeq 2.3 \pm 0.05$ at.% Ni (Murani *et al* 1974). However, the interpretation of this result is again subject to some debate. Specifically, while there is general agreement that local environmental effects play some role in this system, the extent of these effects is not agreed upon at present. Inhomogeneous models assert that only that fraction of Ni atoms forming nearest neighbour pairs (Aldred *et al* 1970)—or possibly triplets (Chouteau *et al* 1974, Chouteau 1976, Sain and Kouvel 1978, Cheung *et al* 1981)—are magnetic, and

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hence that above the critical composition c_0 ferromagnetism evolves via interactions between such magnetic clusters. By contrast, a recent re-analysis of susceptibility and magnetisation data on **Pd**Ni (Loram and Mirza 1985) concluded that this system was magnetically homogeneous, and ferromagnetic order results from the interaction of such isolated (through fluctuating) Ni moments. The principal physical argument presented against the inhomogeneous approach was the absence of spin-glass ordering at compositions below c_0.

In addition to the numerous magnetic investigations referred to above, the **Pd**Ni system has also been studied using a variety of specific heat, resistivity, magnetoresistance and thermopower measurements (for example, Fischer 1982). However, despite this extensive experimental interest (and its attendant theoretical activity), no detailed AC susceptibility has been reported; this latter technique has been widely adopted during the last decade to investigate the ground state occurring in metallic alloys, in which the often subtle spin configurations are minimally disrupted by the small ($<10^{-4}$ T) exciting fields associated with this measurement. Here we report the results of such a study for Ni compositions spanning the ferromagnetic percolation threshold.

2. Experimental details

The samples studied were prepared by arc melting using 99.999% pure Pd wire and 99.998% pure Ni rod (both supplied by Johnson–Matthey (London)) as starting materials. Initially a master alloy of Pd + 5 at.% Ni was prepared, and subsequently fifteen specimens containing between 2 and 5 at.% Ni were made by successive dilution from this master sample. At each step specimens were homogenised, etched and annealed as described previously (Hall *et al* 1984). Measurements were made on samples with typical dimensions $1.7 \times 0.2 \times 0.015\ \text{cm}^3$ cut from strips produced by cold-rolling (between protective Melinex sheets), and on which the corners had been rounded; following etching such samples were annealed for 24 hours in vacuo at 1000°C prior to measuring.

The AC susceptibility of each sample was measured using a previously described phase-locked susceptometer (Ho *et al* 1981a, b) operating at 2.4 kHz with a driving field of 5 μT. The superimposed collinear static biasing field varied between 0 and 0.1 T. The temperature range covered was 1.4 to 77 K, temperatures below about 10 K were measured with a calibrated Ge thermometer (Cryocal Inc. CR2500 H), while at higher temperatures a Au + 0.03 at.% Fe versus chromel P thermocouple was used; each sensor was in good thermal contact with the sample.

3. Results and discussion

3.1. General features of χ_{AC}

The data reported below are similar qualitatively to those acquired previously for Pd containing comparable amounts of Co (Maartense and Williams 1976). In the vicinity of the ordering temperature T_c, the measured zero-field susceptibility $\chi_{AC}(H_a = 0, T)$ increases rapidly with decreasing temperature, as shown in figures 1–3 which reproduce a representative selection of available results. $\chi_{AC}(0, T)$ peaks at a temperature

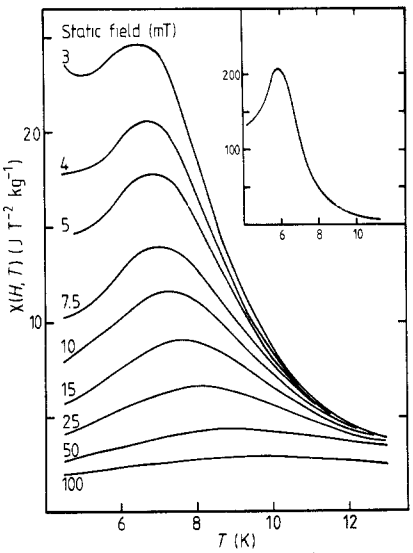

Figure 1. The AC susceptibility (in $J\,T^{-2}\,kg^{-1}$) measured in zero static applied field (inset) and in various other static fields (shown in mT against each curve), plotted against temperature T (in K) for the Pd + 2.7 at.% Ni sample.

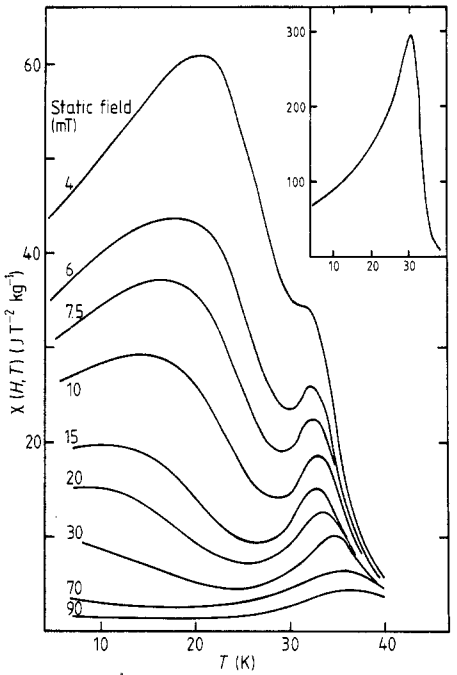

Figure 2. As figure 1, but for the Pd + 3.5 at.% Ni specimen; the behaviour of the principal maximum (the Hopkinson peak) and the true critical peak is made apparent here. The inset again shows the behaviour of the measured zero-field susceptibility.

somewhat below T_c—the Hopkinson peak (Chikazumi 1964)—with a maximum susceptibility of typically (2–4) $\times 10^{+2}\,J\,T^{-2}\,kg^{-1}$. These latter values represent only about 1% of the limit set by demagnetising constraints† (the demagnetising factors N were estimated numerically as described previously (Saran and Williams 1987)). Furthermore, they are up to a factor of 50 times smaller than those observed in *soft* ferromagnetic alloys based on the same host, i.e. **Pd**Mn (Ho *et al* 1981a, b). They are, however, comparable with the maximum susceptibility observed in systems like (**Pd**Ni)Mn where anisotropy/coercivity effects are known to play a role (Hall *et al* 1984, Kunkel *et al* 1988a). Indeed the conventional explanation for the rapid decrease evident in $\chi_{AC}(0, T)$ with decreasing temperature below the Hopkinson peak is in terms of a strong temperature dependence for anisotropy/coercivity mechanisms (Morrish 1980). Furthermore while anisotropy can arise from a variety of sources, the most likely origin for it in the present system is spin–orbit coupling; the analyses of magnetoresistance (Senoussi *et al* 1977) and Hall effect measurements (Hamzic *et al* 1978) on **Pd**Ni confirm the presence of an orbital component in the total moment at

† The measured susceptibility χ_m is related to the true susceptibility χ_t by: $\chi_t = \chi_m/(1 - N\chi_m)$, this relationship being the result of a correction for the internal field (induction) H_i rather than the applied field (induction) H_a, with $H_i = H_a - NM$ (in the usual notation). This restricts the measured susceptibility to values $\leqslant N^{-1}$.

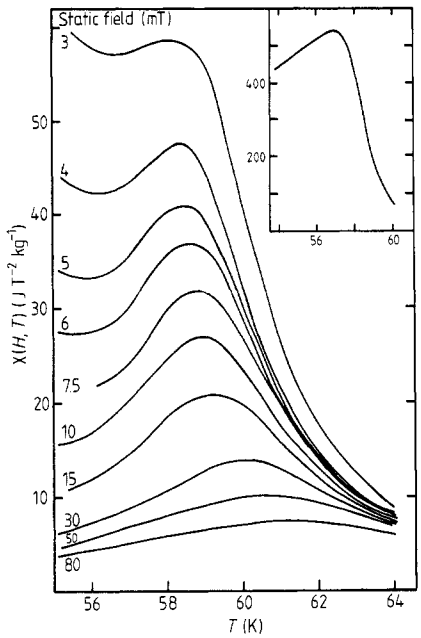

Figure 3. As figure 1, but for the Pd + 4.5 at.% Ni alloy; as in the main part of figure 1, the behaviour of the critical peak is detailed.

the Ni site, and this has been used recently to estimate accurately the critical composition for ferromagnetism c_0 via the appearance of a non-zero spontaneous resistive anisotropy (SRA) (Kunkel *et al* 1987, 1989). Further influence of this spin–orbit induced anisotropy is discussed below.

3.2. Field-Dependence of χ_{AC}

The effects of externally applied fields (H_a) are also summarised in figures 1–3; figure 2 shows that the Hopkinson peak is suppressed in amplitude and moves downwards in temperature as H_a increases, thus facilitating the observation of smaller secondary peaks in the vicinity of T_c. The behaviour of these latter peaks is consistent with that suggested by the static scaling law (Stanley 1971)

$$\chi(h, t) = t^{-\gamma} F(h/t^{\gamma+\beta}) \tag{1}$$

where h and t are the conventional linear scaling fields. The scaling equation of state predicts the occurrence of a crossover line above T_c (when $H_a \neq 0$), the temperature T_m of which is given by (Ho *et al* 1981a, b, Kunkel *et al* 1988b)

$$(T_m - T_c)/T_c = t_m \propto h^{(\gamma+\beta)^{-1}}. \tag{2}$$

Along this line the field dependent susceptibility exhibits a maximum, the amplitude of which depends on the field according to

$$\chi(h, t_m) \propto h^{(1/\delta)-1}. \tag{3}$$

The experimentally observed peaks do indeed move upwards in temperature and their height diminishes as H_a increases. The power-law relationships given by equations (2) and (3) have formed the basis of detailed analyses of the magnetic response of a number of systems, enabling the critical exponents γ, β and δ to be estimated (Ho *et*

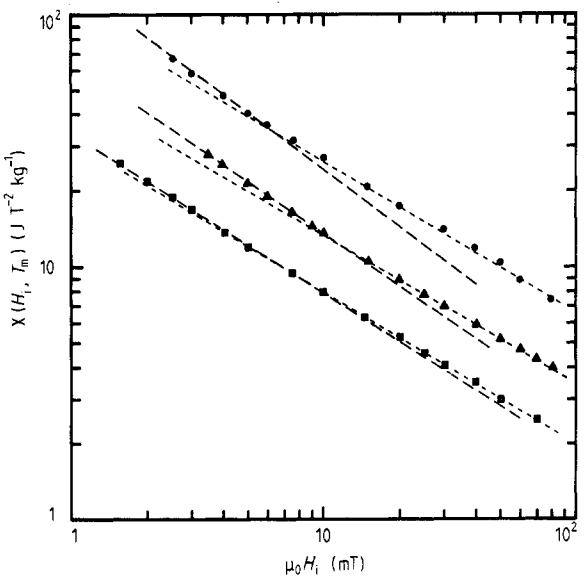

Figure 4. The critical peak amplitude (in J T^{-2} kg^{-1}) corrected for background and demagnetising effects plotted against the internal field $\mu_0 H_i$ (in mT) on a double-logarithmic scale. The curves shown, arranged in order of increasing Ni concentration, were obtained from samples containing 2.4, 2.9, and 4.5. at.% Ni respectively. The lines are discussed in the text.

al 1981a, b, Gaunt *et al* 1981, Saran and Williams 1987, Kunkel and Williams 1988a, b). However, one pre-requisite for fitting experimental data to these equations is that the measured magnetic response be dominated by that component arising from critical fluctuations (i.e. that component summarised by equation (1)). In practice the latter occurs in materials with a relatively low net moment in which the regular contribution to the response (due to coherent rotation and/or domain wall motion, for example) approaches saturation in low applied fields. This leads directly to the rapid suppression of the Hopkinson peak in amplitude in addition to its downward shift in temperature. Such a situation does not occur in the present system; as can be seen from figures 1–3, applied fields ($\mu_0 H_a$) in excess of 2 mT are required before the emerging critical peaks can be resolved (more than an order of magnitude larger than those required to produce similar effects in soft ferromagnets such as **Pd**Mn (Ho *et al* 1981a) and (**Pd**Fe)Mn (Kunkel and Williams 1988b)). Furthermore, even in applied fields up to 10 mT, the temperature derivative of the susceptibility reverses quite rapidly with decreasing temperature just below these critical peaks, causing them to appear rather broad. Thus while the presence of a Ni orbital moment is essential to explain the non-vanishing spontaneous resistive anisotropy in **Pd**Ni (Senoussi *et al* 1977, Kunkel *et al* 1987, 1989), its occurrence inhibits the interpretation of magnetic data.

Quantitative support for this latter statement is provided by attempts to analyse susceptibility data in terms of the power-law predictions of equations (2) and (3).

In figure 4 plots of the peak susceptibility $\chi(h, t_m)$ (corrected for background and demagnetising effects) against the internal field H_i (estimated from $H_i = H_a - NM$) on a double-logarithmic scale are presented. For all the ferromagnetic alloys studied ($c = 2.4$ to 5 at.% Ni), a power-law behaviour with a *unique* index (from which the critical exponent δ can be found) is *not* observed. These double-logarithmic plots exhibit curvature and the straight lines drawn on them at high and low field enable the corresponding *effective* exponents δ^* to be found; such estimates are listed in table 1. Two aspects of their variation warrant comments: (i) the result that, in general, δ^*

Table 1. Summary of parameters deduced from AC susceptibility data. (Samples containing less than 2.4 at.% Ni do not order above 1.5 K.)

Composition (at.% Ni)	T_c (K)	δ^*_{LF}	δ^*_{HF}	T_m versus $\sqrt{\mu_0 H_i}$ plot (K $T^{-1/2}$)
2.4	1.95 ± 0.1	2.7	2.5	8.5 ± 0.2
2.5	3.85 ± 0.1	2.7	2.2	10 ± 0.3
2.6	4.05 ± 0.1	2.8	2.4	10.5 ± 0.3
2.7	6.0 ± 0.2	3.0	2.5	13 ± 0.4
2.9	11.0 ± 0.3	3.2	2.4	16 ± 0.5
3.0	12.5 ± 0.5	3.0	2.5	17 ± 0.5
3.5	31.0 ± 0.5	3.3	2.2	18 ± 0.5
4.0	44.3 ± 0.5	4.0	2.2	19 ± 0.5
4.5	57.5 ± 1.0	4.1	2.5	15 ± 0.5
5.0	62.7 ± 1.0	4.1	2.4	10 ± 0.3

appears to decrease with increasing field, and (ii) the variation with composition in the numerical values of these effective exponents.

(i) Effective δ^*-values which decrease with increasing field have been reported previously in **Pd**Mn alloys of intermediate concentration (Ho *et al* 1981b). Such a result is consistent with that predicted for the ferromagnetic phase of a Sherrington–Kirkpatrick-like model (Sherrington and Kirkpatrick 1975, Kaneyoshi 1975, Southern 1976) with an Ising spin–spin interaction Hamiltonian of the form

$$\mathcal{H} = -\sum_{i<j} J_{ij} S_i S_j - H \sum S_i \tag{4}$$

in which the exchange coupling strengths J_{ij} fluctuate according to a Gaussian distribution. As the ratio $\eta = \bar{J}_0/\bar{J}$ of the first to second moments of this distribution declines towards 1, the effective exponent δ^* exhibits a more marked reduction as the applied field H increases, although its asymptotic value remains unchanged (Roshko and Williams 1984). In addition, model calculations yield Arrott plots that display increasing curvature at low field as $\eta \rightarrow 1$ (Yeung *et al* 1986), as has also been observed previously for **Pd**Ni as the composition c is reduced towards c_0 (Murani *et al* 1974). The data displayed in figure 4 can thus be interpreted (within a localised model) as indicating a distribution of effective exchange interactions between Ni sites which narrows from a ratio $\eta \geqslant 1$ close to the critical composition c_0 to larger η values as the composition increases.

(ii) This conclusion is confirmed by the concentration dependence of the numerical values found for δ^*. For fields $\mu_0 H_i \geqslant 10$ mT, figure 4 indicates $\delta^*_{HF} = 2.2$–2.5 for all samples examined. At lower field†, however, the effective exponent δ^*_{LF} increases with Ni concentration from 2.7 at 2.4 at.% to a value of 4.1 at 5 at.%, the latter being close to that found in elemental Ni (Kouvel and Fisher 1964). It is important to

† These numerical values are quoted subject to the complications that might be associated with a non-vanishing regular contribution to the estimated critical peak amplitude from the Hopkinson maximum. The trend noted, however, will hold provided the balance between the true critical contribution and the supposed regular component does not change with composition. This appears reasonable since the lowest field needed to resolve the critical peak does not vary significantly with Ni concentration.

reiterate that these effective exponent estimates do not extend below $\mu_0 H_i \leqslant 2$ mT, so they may not be true asymptotic exponents. Nevertheless the above results suggest that the asymptotic δ-values *may* be close to those found in a variety of ferromagnets ($\delta \simeq 4.2$). The failure to observe an increase in δ^*_{LF} as the Ni concentration c is lowered toward $c_0 = 2.25$ at.% is consistent with the conclusions reached above, that the ratio $\eta \rightarrow 1$ as $c \rightarrow c_0$; as $\eta \rightarrow 1$ one approaches the limit of stability of the ferromagnetic ground state, when the range of validity of the scaling law description of $\chi(h, t)$ shrinks in the (h, t) plane (Roshko and Williams 1984). These data clearly do not show any indication of an increase in the asymptotic δ-value predicted to result from a reduction in effective topological dimensionality as c_0 is approached (Stauffer 1979, Coniglio 1981, Kaul 1985); they appear dominated by the behaviour of the bond distribution (i.e. η).

As a corollary, as c is lowered below c_0 and η falls below 1, the model approaches predict a spin-glass ground state (Sherrington and Kirkpatrick 1975), provided of course that the Kondo effect does not interfere (Loram and Mirza 1985).

Finally, despite the complications introduced by spin–orbit coupling into the quantitative analysis of the variation with field of the critical peak amplitude, discussed above, there nevertheless exists an interesting comparison between such peak amplitudes associated with different magnetic ions in Pd. Specifically, for internal fields $\mu_0 H_i$ near 10 mT the ratio of $\chi(H_i, T_m)$ for Pd containing comparable amounts ($\simeq$3 at.%) of Gd (Saran and Williams 1987), Mn (Ho *et al* 1981a) and Ni is approximately 5 : 3 : 1. This ratio is remarkably close to that predicted by model calculations (Kunkel *et al* 1988b) if spin values of $\frac{7}{2}$, $\frac{5}{2}$ and $\frac{1}{2}$, respectively, are assigned to these impurity sites. Such an assignment, though indirect, provides further support for arguments attributing a d^9 configuration to the Ni site in this host (Loram and Mirza 1985, Kunkel *et al* 1987, 1989).

Next we examine the field dependence of the critical peak temperatures, as described by equation (2). In soft ferromagnetic systems it has been possible to implement double-logarithmic plots of $t_m = (T_m - T_c)/T_c$ against field H_i and extract reliable estimates for T_c, γ and β (Kunkel and Williams 1988a, b). Here, the Ni orbital moment, through its effect on the behaviour of the Hopkinson peak, precludes such an analysis not only by obscuring the critical peak at low field (thus limiting substantially the field range over which double-log plots can be made) but also—through peak broadening effects—by introducing increased uncertainty in the peak temperature T_m (this is particularly noticeable on a log scale as $T_m \rightarrow T_c$ and $t_m \rightarrow 0$). An approximate analysis of the field dependence of the critical peak temperature and an estimate for T_c can be made using a similar approach to that adopted previously for (**Pd**Ni)Mn (Hall *et al* 1984). If the exponents γ and β are assumed to be close to the theoretical values predicted by the isotropic three-dimensional Heisenberg model (LeGuillou and Zinn-Justin 1980), equation (2) can be rewritten approximately as

$$T_m - T_c \simeq \sqrt{H_i}. \tag{5}$$

Figure 5 demonstrates that this approximate relationship is tolerably well obeyed in **Pd**Ni, and while such a procedure does not allow the exponents γ and β to be evaluated with any precision, it nevertheless enables estimates for T_c to be made; these are listed in table 1.

There appears to be an interesting correlation between the concentration dependence of the slopes in figure 5 and those exhibited by the high field magnetoresistance

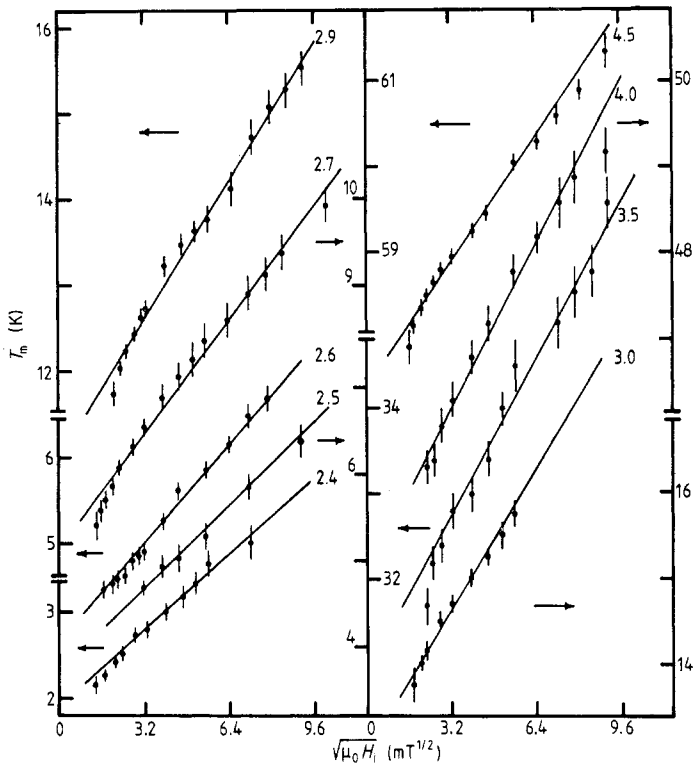

Figure 5. Plots of the critical peak temperature T_m (in K) against the square root of the internal field $\mu_0 H_i$ (in mT) for most of the samples studied. The intercepts at $H_i = 0$ enable estimates for the Curie temperatures to be made.

in this system (Kunkel *et al* 1989). The physical origin of this similarity is not clear to us at present. The slopes found from figure 5 are comparable to those estimated for intermediate concentration **Pd**Mn alloys (Ho *et al* 1981b), which also display unusual features in the zero-field susceptibility, discussed below.

3.3. Zero-field susceptibility $\chi_{AC}(0, t)$

With a value specified for T_c, it is usual to examine the remaining scaling law prediction by attempting to fit the zero-field susceptibility to

$$\chi(0, t) \propto t^{-\gamma} \qquad t > 0 \tag{6}$$

using a double-logarithmic scale (the slope of which is γ). Frequently the origin (T_c) of such plots, and similar ones based on equation (2), is adjusted slightly (by up to a few tens of mK) until an acceptable power-law behaviour is established. As figure 6 shows, such plots remain unconvincing in **Pd**Ni; the failure of the measured zero-field susceptibility $\chi(0, t)$ to approach demagnetisation limit (N^{-1})—discussed in section (3.1)—translates into a severe flattening of these plots at low reduced temperature, while at higher temperatures detailed examination reveals considerable curvature (particularly at larger Ni concentrations) with large local slopes (effective $\gamma^*(T)$ exponents), and no extended power law regime is seen. This situation is very similar

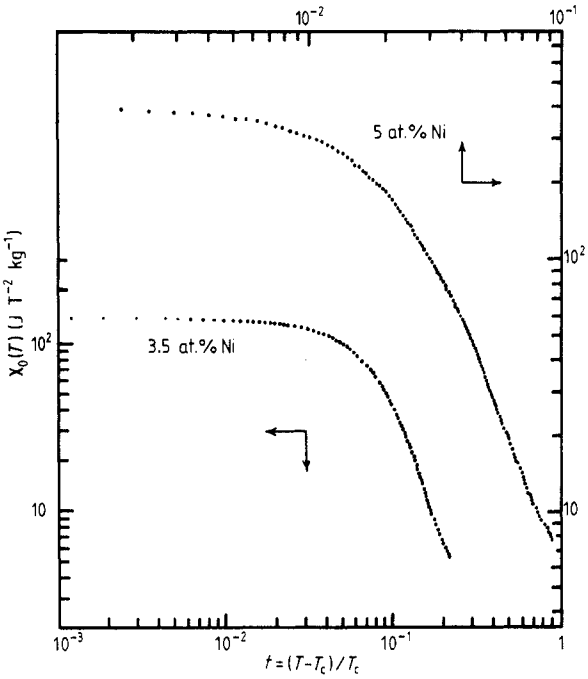

Figure 6. Plots of the zero-field susceptibility (in J M^{-2} kg^{-1}), corrected for background and demagnetising effects, against the reduced temperature $t\,(=(T-T_c)/T_c)$ for the 3.5 and 5 at.% Ni samples. The T_c values were obtained from figure 5.

to that reported recently in a variety of disordered systems (Gaunt *et al* 1981, Kaul 1985, Fähnle *et al* 1988, Seeger and Kronmüller 1989), consequently we have chosen to present our data in a form suitable for comparison, namely as effective components $\gamma^*(t)$ versus $T-T_c$ (figure 7).

In the region $T\rightarrow T_c$ such plots remain unreliable for the reasons outlined above, so they cannot be used to assess either the validity of the Harris (1974) criterion or whether the dominant interactions in this system are short-ranged ($\gamma(t\rightarrow 0)\simeq 1.38$) or long-ranged ($\gamma(t\rightarrow 0)\simeq 1.0$) (Seeger and Kronmüller 1989, Fisher *et al* 1972, Joyce 1966, Fähnle *et al* 1988); the behaviour of the effective exponent δ^*, however, would favour the former.

At intermediate temperatures this effective susceptibility exponent increases with increasing temperature, and the maximum value achieved by $\gamma^*(T'_m)$ and the temperature (T'_m) at which this maximum value occurs display some systematic variation with Ni concentration. Near the critical composition c_0, $\gamma^*(T)$ increases only slightly above the predicted value for the isotropic three-dimensional Heisenberg model over the entire range investigated. However, with further composition increases $\gamma^*(T'_m)$ and T'_m behave similarly to the slopes of T_m versus $\sqrt{\mu_0 H_i}$ (figure 5, discussed above) and the high field magnetoresistance (Kunkel *et al* 1989). Furthermore the maximum $\gamma^*(T'_m)$ value observed here ($\simeq 3.2$) while being larger than values reported ($\leqslant 2.0$) in a review of the magnetic properties of amorphous systems (Kaul 1985), is comparable with estimates obtained from **Pd**Mn samples of intermediate concentration (3–5 at.% Mn) (Ho *et al* 1981b) which also display evidence of substantial bond disorder,

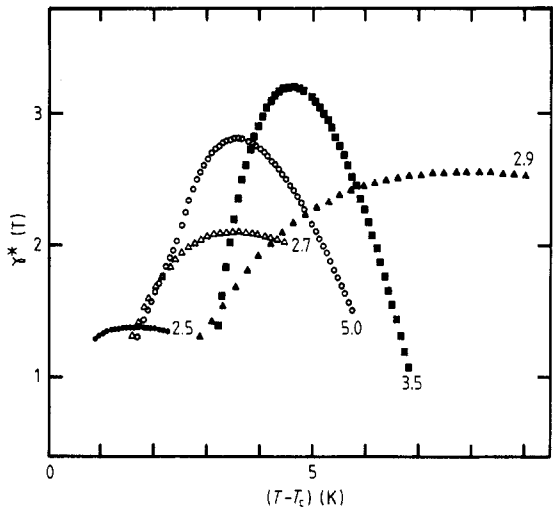

Figure 7. The effective susceptibility exponent $\gamma^*(T)$ plotted against $T - T_c$ (in K) for various samples, the Ni concentration (in at.%) of which is shown against each curve.

principally through the behaviour of δ^*. In all the **Pd**Ni samples studied, $\gamma^*(T \gg T_c)$ falls towards the mean-field value of 1.

4. Summary and conclusions

Attempts have been made to analyse in detail the magnetic response of **Pd**Ni alloys containing 2 to 5 at.% Ni. The presence of spin–orbit-induced anisotropy (associated with an orbital contribution to the total moment at the Ni site) prevents reliable estimates of the critical exponents from being made, although values for the Curie temperatures can be found. Nevertheless these data are consistent with suggestions that a spin-glass ground state would appear at low temperatures for Ni concentrations at or below 2 at.% if complications arising from the single ion Kondo compensation of a low spin state were absent.

Acknowledgments

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