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MAGNETIC REGIMES IN AMORPHOUS Ni-Fe-P-B ALLOYS

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by

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Professor Pol Duwez, principal investigator.

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ABSTRACT

A complete substitution of iron for nickel has been obtained by splat-cooling in amorphous alloys of composition $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$. Results of high field magnetization (up to 70 kOe), ac and dc low field susceptibility, Curie temperature and resistivity measurements over a temperature range of 1.7 to 300°K are reported. The $\text{Ni}_{79}\text{P}_{13}\text{B}_8$ alloy is not ferromagnetic, but the magnetization behavior as a function of field and temperature is typically that of alloys in the critical concentration range for ferromagnetism. The $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ alloy is ferromagnetic with a Curie temperature T_c of 616°K. For $y = 1$ at.%, the Fe atoms are magnetic. The variation of the moment per Fe atom as a function of y is discussed. When y is increased, the Ni atoms are likely to be polarized progressively and the moment per Ni atom would be roughly constant for $y \geq 30$ at.%. Various magnetic behaviors were defined as a function of the Fe content. The value of T_c reaches a maximum for $y \approx 90$ at.%, and extrapolates to zero for $y \approx 7$ at.%. Alloys within the range $1 \leq y \leq 10$ at.% did not exhibit well-defined Curie transition, but sharp maxima in low field susceptibility measurements were observed at T_M . The value of T_M is proportional to y for $1 \leq y \leq 4$ at.%, like in classical spin-glass regimes. For $4 < y \leq 10$ at.%, the variation of T_M as a function of y implies a more complicated type of magnetic ordering (mictomagnetism or superparamagnetism). Homogeneous ferromagnetic ordering emerges only for $y > 10$ at.%. Results of resistivity measurements are discussed in relation with the magnetic properties of different regimes in the magnetic phase diagram.

I. INTRODUCTION

Recent studies on crystalline concentrated alloys and compounds have emphasized the complex nature of the transition from the paramagnetic to the ferromagnetic state.¹ Most of the systems investigated undergo various intermediate magnetic regimes before reaching a long-range homogeneous ferromagnetic ordering. Experimentally, the main characteristics of these regimes are fairly well defined, although the physical mechanisms involved in each regime are not always clearly understood.² For example, in the classical cases, like Au-Fe, Mo-Fe....^{3,4}, one can distinguish a number of progressive steps of magnetic ordering between the Kondo concentration and the critical concentration for the onset of ferromagnetism: spin-glass regime (scaling laws ordering temperature varying linearly with concentrations, $T^{3/2}$ dependence of the resistivity...), micromagnetic regime (strong magnetic short-range order, irreversible processes, thermomagnetic historic effects, displaced hysteresis curves...), superparamagnetic regime (different ordering temperatures as determined by local or by bulk magnetic measurements...). In such systems, there is some evidence of a mixture of local antiferromagnetism and ferromagnetism attributed to long-range interactions of the RKKY type.⁵ On the other hand, such phenomena as susceptibility maxima, irreversible processes... were observed in completely different systems where the dilute impurity has no localized moment (Ni in Cu, Fe in V...) ^{6,7}, but, at higher concentrations, the magnetic moment resides in large polarization clouds. An interpretation of the

magnetic "anomalies" in this case was suggested in terms of a random orientation of the anisotropy axes of the spin clusters.⁶ Since the same phenomena may occur in physically different contexts, some authors suggested to give all the intermediate magnetic regimes the same name: mictomagnetic¹ or spin-glass regime.⁸

The onset of ferromagnetic order was already investigated by local and bulk magnetic measurements in some amorphous systems, like Pd-Si, Fe-Pd-P, Cu-Zr...⁹⁻¹⁴, and the complexity of the para-ferromagnetic transition in amorphous systems was pointed out: analogy between amorphous Fe-Pd-P and crystalline Au-Fe systems suggested by Sharon and Tsuei¹¹, some irreversible phenomena mentioned by Hasegawa⁹, susceptibility maxima in amorphous Pd-Si alloys with Fe and Co...¹⁵ Recent progress in the understanding of the intermediate regimes in the crystalline case encouraged us to undertake a detail study of the electrical and magnetic properties at low (less than 500 Oe) and high (up to 70 kOe) fields in amorphous (Ni-Fe)-P-B alloys. The (Ni, Fe)₇₉P₁₃B₈ alloys seemed to be a good candidate for such an investigation, since a complete substitution of Fe for Ni is possible in the amorphous phase.

III. EXPERIMENTAL PRODEDURES

The samples were prepared by splat-cooling from the melt. Each foil was checked by a Norelco X-ray diffractometer. Concentrations are nominal. Magnetic ordering temperatures were observed using a standard ac inductance bridge. Magnetization measurements were made between 1.7 and 290°K in fields ranging from 0.1 to 70 kOe using

the Faraday method. Electrical resistivity as a function of temperature was measured using a standard four probe technique. More detail on the experimental procedures may be found in ref. 16.

IV. RESULTS AND DISCUSSION

The magnetic properties of the $\text{Ni}_{79}\text{P}_{13}\text{B}_8$ alloys have been discussed previously.¹⁷ The initial susceptibility exhibits a temperature independent term of about $2 \cdot 10^{-6}$ emu/g and a small magnetic contribution strongly temperature dependent ($\sigma_0(0^\circ\text{K}) = 0.78$ emu/g). From a Brillouin function analysis, the magnetic part was found to arise from polarization clouds of Ni. These clouds are antiferromagnetically coupled and they have two different sizes: $\mu_1 = 5\mu_B$, $\theta_1 = 1.0^\circ\text{K}$, $c_1 = 0.14$ at.%, and $\mu_2 \approx 9\mu_B$, $0 < \theta_2 < 1^\circ\text{K}$, $c_2 = 6 \cdot 10^{-3}$ at.%. The contribution of the $\text{Ni}_{79}\text{P}_{13}\text{B}_8$ matrix will be subtracted from the magnetization data of the $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$ alloys for $1 \leq y \leq 10$ at.%. For $y > 10$, this contribution was found negligible. The variation of the Curie temperature T_c and of the mean magnetic moment per transition metal atom at 4.2°K $\mu/\text{at.}$ (Fe, Ni) as a function of the Ni content $x = 100 - y$ is shown on Fig. 1. We first comment on the low Fe content part of the magnetic phase diagram (section A); then we discuss the magnetic properties for the ferromagnetic region (section B); finally, we summarize the results of electrical resistivity measurements performed over the whole concentration range.

A. $1 \leq y \leq 10$. Intermediate Magnetic Regimes.

The magnetic parameters of the alloys in the concentration range

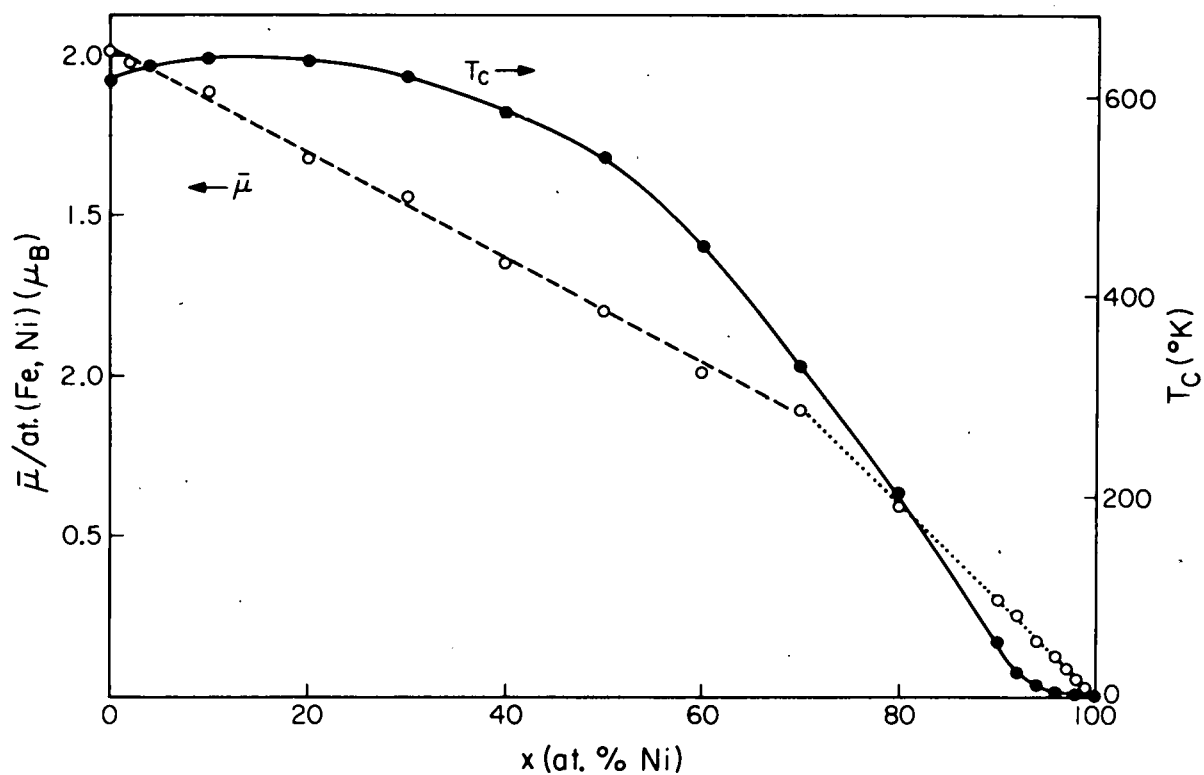


Fig. 1. Variation of the Curie temperature T_C and of the mean magnetic moment per transition metal atom $\bar{\mu}/\text{at. (Fe, Ni)}$ as a function of the Ni content x in amorphous $(\text{Fe}_{100-x}\text{Ni}_x)_{79}\text{P}_{13}\text{B}_8$ alloys.

$1 \leq y \leq 10$ are listed in Table 1. Fig. 2 shows some low temperature isotherms $M = f(H)$ for a sample with 2 at.% Fe. The saturation becomes progressively easier when y increases. Even for 1 at.% Fe, the magnetization data at low temperature cannot be fitted by an unique Brillouin function. The isolated impurity regime in this system could be reached only for a much lower concentration of Fe. Our results were analyzed in the classical way used for crystalline concentrated alloys.^{18,19} (see Table 1). Assuming no polarization of the Ni atoms (a possible polarization will be discussed in the following section) the Fe atoms would carry a moment of about $3\mu_B$, roughly constant over this concentration range. This moment is not carried by individual Fe atoms, but by some magnetic polarization clouds. The average size μ^* of these magnetic clusters is fairly constant and equal to $12-13\mu_B$, and their concentration c^* increases with the Fe content.

The paramagnetic Curie temperature θ , defined by the variation at high temperature of the initial susceptibility $\chi_o(T) = C_{cw}/(T-\theta)$, increases monotonically as a function of y . The large differences between θ and the ordering temperature T_M are a measure of the inhomogeneous short-range ferromagnetic interactions. The value of $(\theta-T_M)/T_M$ decreases from a value of about 15 for $y = 1$ down to 1.8 for $y = 10$. This variation characterizes the critical range for the onset of long-range ferromagnetic order.

Measurements of the initial susceptibility at "zero" field (ac bridge) exhibit sharp peaks at temperature T_M (Fig. 3). These peaks are rounded off when the susceptibility is measured at low dc fields ($H < 500$ Oe) as observed in crystalline cases.²⁰ A detail study of

Table 1. Magnetic properties of amorphous $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$ alloys ($1 \leq y \leq 10$ at.%).

y (at. % Fe)	T_M (°K)	θ (°K)	$\sigma_o^{(a)}$ (emu/g)	$C_{cw}^{(a)}$ (10^{-4} °K. cgs)	$\mu^{*(b)}$ (μ_B)	$c^{*(b)}$ (at. %)	$\mu/\text{Fe at.}^{(a)}$ (μ_B)
1	1.80	30	2.39	6.0	11.2	0.20	2.77
2	3.35	45	4.87	15.1	13.8	0.32	2.83
3	5.45	56	7.06	20.0	12.7	0.51	2.73
4	7.35	85	10.70	29.0	12.1	0.81	3.10
5	9.85	—	—	—	—	—	—
6	12.2	121	14.27	52.2	13.2	0.9	2.76
7	15.6	—	—	—	—	—	—
8	23.5	133	21.15	60.0	12.7	1.52	3.06
10	54	152	25.70	78.0	13.6	1.73	2.98

(a) Values obtained after correcting for the $\text{Ni}_{79}\text{P}_{13}\text{B}_8$ matrix contributions.

(b) Average values of the magnetic clouds μ^* and of their concentrations c^* calculated from $\sigma_o = c^* \mu^*$ and $C_{cw} = c^* \mu^{*2} / 3k$ (k , Boltzmann constant).

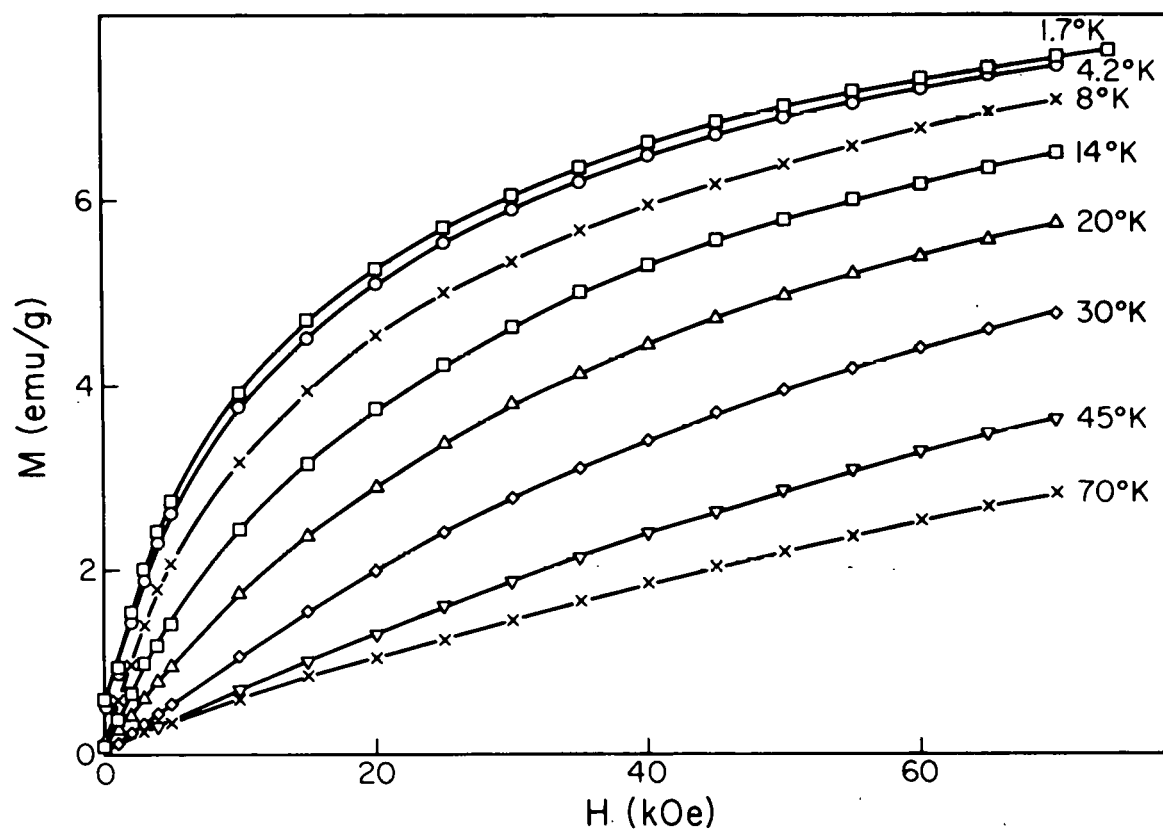


Fig. 2. Magnetization versus applied field at low temperature in amorphous $(\text{Ni}_{98}\text{Fe}_2)_{79}\text{P}_{13}\text{B}_8$ alloy.

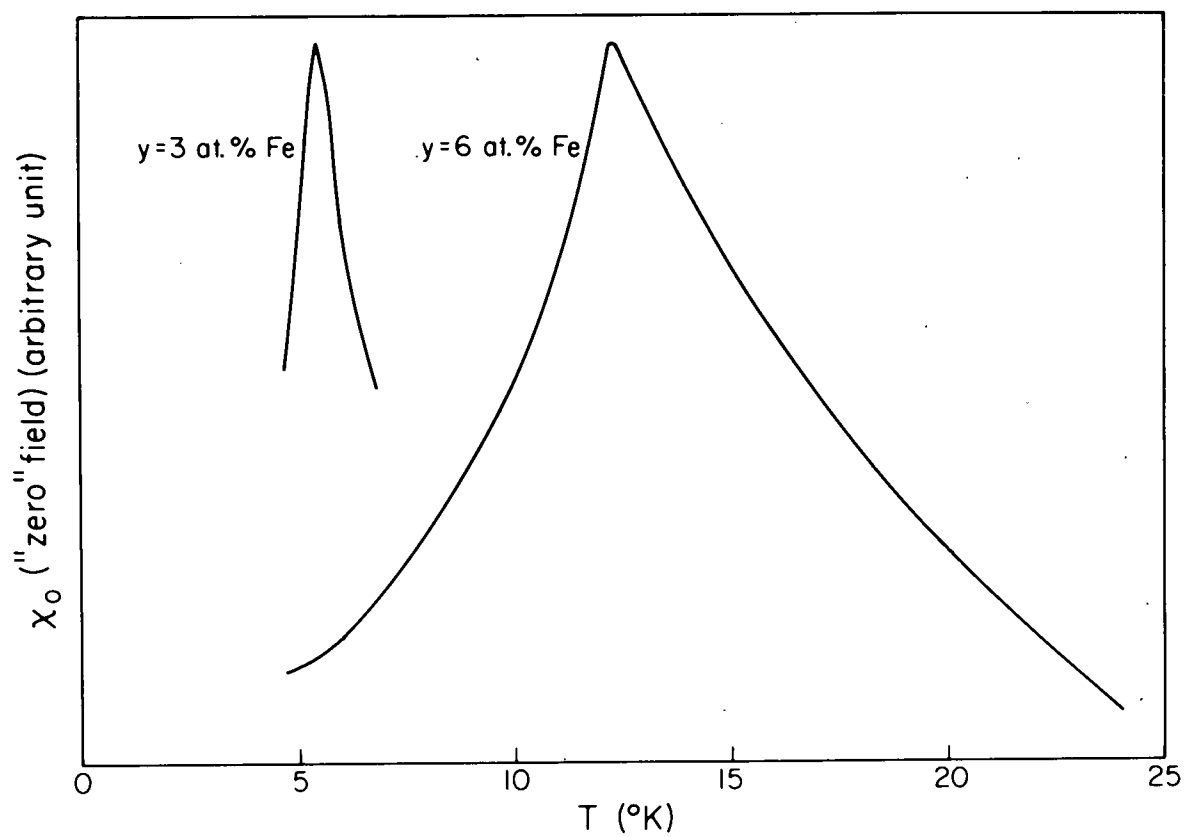


Fig. 3. Magnetic susceptibility (in arbitrary unit) as a function of temperature in "zero" field for $y = 3$ and 6 at.% Fe in amorphous $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$ alloys.

the concentration dependence of T_M (Fig. 4) and of the magnetic behavior of the different samples allows us to distinguish three different intermediate regions:

1. $1 \leq y \leq 4$. T_M is proportional to y (insert of Fig. 4), like in Au-Fe in the spin-glass regime ($y \leq 1$ at.%) in the sense of ref. 3. The analogy is only formal, since in our case the scaling laws are not obeyed and there is no evidence for a substantial long-range RKKY coupling. In this region, the Fe-Ni interactions are negligible. This point was verified by measuring T_M for $y = 1$ to 4 at.% Fe in a $\text{Ni}_{80}\text{P}_{12}\text{B}_8$ matrix (insert of Fig. 4). We found exactly the same values for $y = 1$ and 2, but the departure from linearity starts with $y = 3$ when the alloy is richer in Ni.

2. $4 < y \leq 8$. T_M as a function of y departs slightly from linearity. The Fe-Ni interactions become visible. Looking at the Arrott plots ($M^2 = f(H/M)$), one cannot define any Curie temperature, even for the low field part (about 100 Oe) of the curve.

3. $8 \leq y \leq 10$. For these alloys, a Curie temperature may be defined from the low values of H in the Arrott plots, and this T_c agrees fairly well with the values of the susceptibility maxima. On the other hand, a drastic change is observed at $y = 8$ on the variation of T_M as a function of y , and the values of T_M for $y = 8$ and 10 are in the trend of the value of T_c for $y = 20$. But the observed temperature variation of the initial susceptibility is certainly not characteristic of a good ferromagnetic transition, as compared with the 20 at.% Fe sample where no peak is observed. This region resembles that called superparamagnetic in the Fe-Cr, Fe-Mo, Au-Fe systems. It is expected

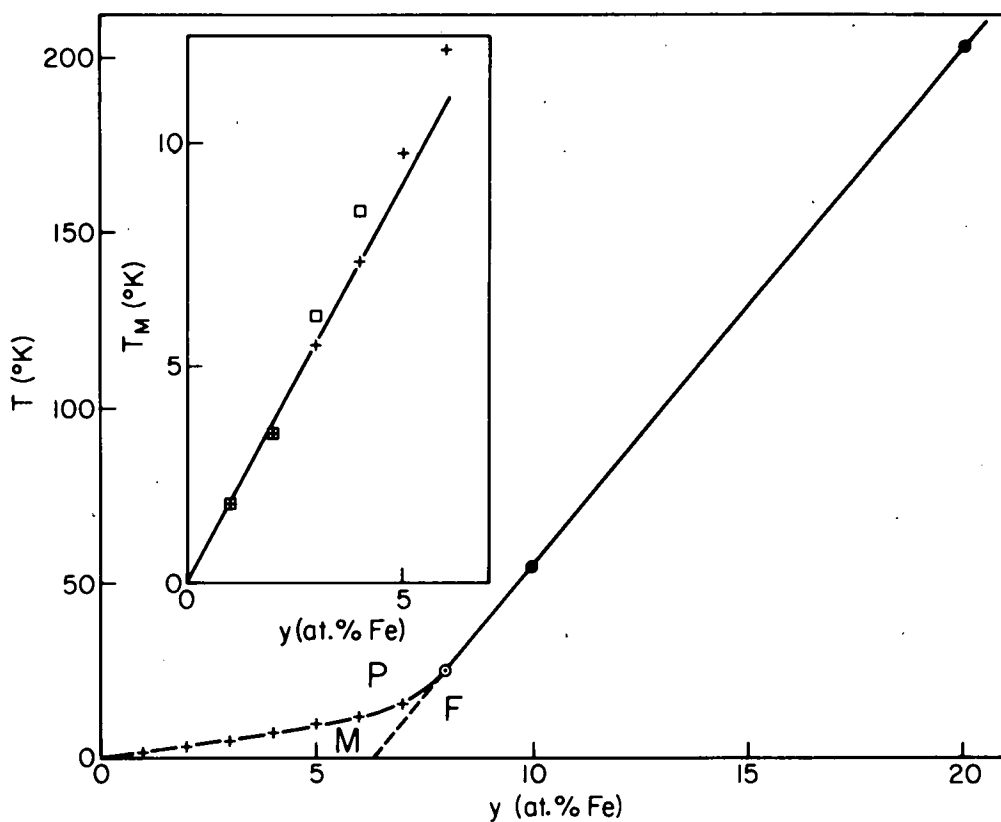


Fig. 4. Ordering temperatures (see text) as a function of Fe concentration in amorphous $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$ alloys.

Insert: variation of T_M as a function of y ($y \leq 6$ at.% Fe)

in $(\text{Ni}_{100-y}\text{Fe}_y)_{79}\text{P}_{13}\text{B}_8$ (+) and in $(\text{Ni}_{100-y}\text{Fe}_y)_{80}\text{P}_{12}\text{B}_8$ (\square) amorphous alloys.

for our amorphous alloys that the ordering temperature observed by local measurements (Mössbauer, neutrons) will not coincide with the values of T_M . This problem is under investigation. Some measurements are planned also for samples with y between 10 and 20 at.% Fe for determining the real critical concentration for the onset of long-range ferromagnetic order.

In conclusion, we found, between the paramagnetic and the ferromagnetic states, some intermediate regions, whose characteristics resemble those defined in the Au-Fe system, although the mechanisms involved in the two cases are likely to be different.

B. $y \geq 20$. Ferromagnetic Regime.

1. Variation of the Curie temperature (Fig. 1, 4, 5). At the beginning of the ferromagnetic regime (Fig. 4), T_c increases with an initial slope of about $15^\circ\text{K/at.}\%$ Fe. The curve of T_c as a function of y is rounded off and T_c reaches a maximum value of 639°K for $y = 90$ at.%. The T_c decreases down to 616°K for $\text{Fe}_{79}\text{P}_{13}\text{B}_8$. Over the same concentration range, the magnetic moment $\bar{\mu}$ was found to decrease continuously from its value in $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ (Fig. 1). When a small amount of Ni is alloyed with bcc Fe, T_c goes also through a maximum, but the variation of $\bar{\mu}$ follows that of T_c .²¹ An opposite variation of $\bar{\mu}$ and T_c was observed in dilute Fe-Cr, Fe-V and Fe-Ti alloys in the bcc phase.²² As far as we know, this anomaly has never been explained in a satisfactory way. We think that this effect has to be correlated to the crossing variation of $\bar{\mu}$ and T_c observed in amorphous Fe-P and Fe-B alloys.^{16,23} When Ni is added to a matrix having a lower T_c (amorphous $\text{Fe}_{80}\text{P}_{12}\text{B}_8$ - Fig. 5, or crystalline

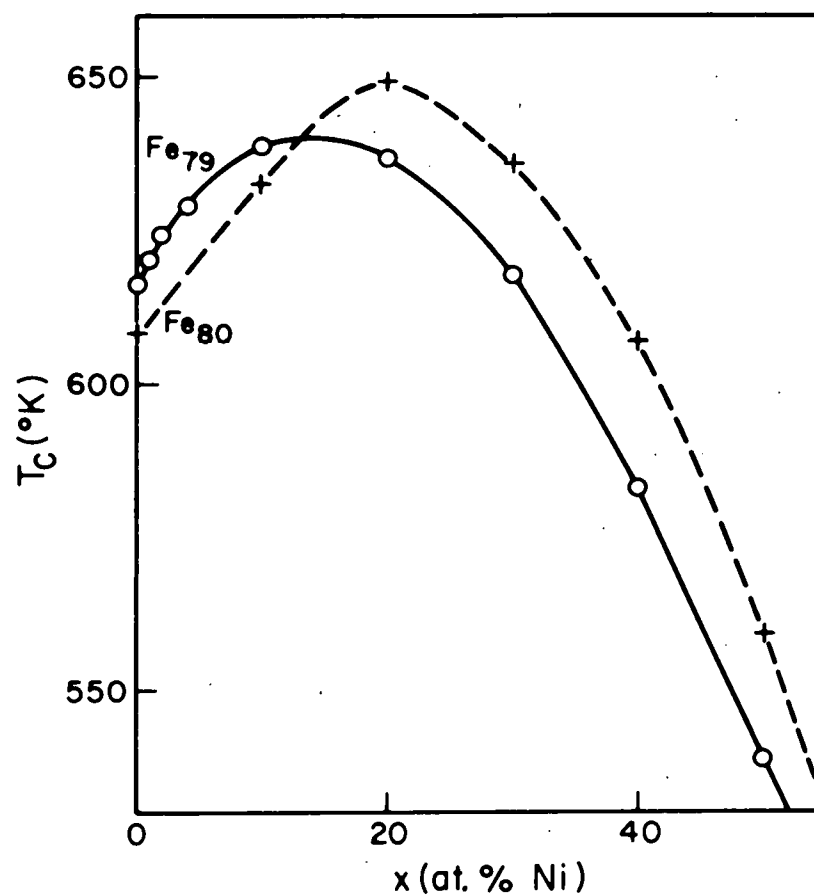


Fig. 5. Curie temperature as a function of Ni content in amorphous $(Fe_{100-x}Ni_x)_{79}P_{13}B_8(0)$ and $(Fe_{100-x}Ni_x)_{80}P_{12}B_8(+)$ alloys.

Fe_2P - ref. 24), the amplitude of the peak is increased and the Ni concentration for the maximum is shifted to a higher value.

2. Variation of the magnetic moment. The variation of the mean magnetic moment per transition metal atom $\bar{\mu}/\text{at. (Fe, Ni)}$ as a function of x is shown on Fig. 1. The curve is divided into two regions. In each region, the variation is linear. According to a least squares fit:

$$0 \leq x \leq 70 : \bar{\mu}/\text{at. (Fe, Ni)} = (2.02 - 1.64x) \mu_B \quad (1)$$

$$70 \leq x \leq 99 : \bar{\mu}/\text{at. (Fe, Ni)} = 2.99 (1 - x) \mu_B \quad (2)$$

We first comment on equation (1), which is valid down to the dilute limit (2 at. % Ni in $\text{Fe}_{79}\text{P}_{13}\text{B}_8$), so that we can discuss the electronic structure of the $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ alloy and of the transition metal impurities in this matrix. Second, we estimate the individual moments on the atoms of Fe and Ni.

2. 1-Electronic structure of $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ and of the dilute impurities of transition metals in this matrix.

As observed before²⁵, equation (1) gives for $\bar{\mu}$ a gradient of $-0.82 \mu_B/\text{e/a}$, which is very close to the value of $-1 \mu_B/\text{e/a}$ predicated by the Slater-Pauling curve. But, the Slater-Pauling approach based on a rigid band model fails to account for the behavior of $\bar{\mu}$ for impurities like Mn, Cr, V in the same matrix. The variation of $\bar{\mu}$ as a function of x is found to be linear from $x = 2$ to $x = 70$ at. % Ni (no impurity pair effect). So, this result, together with the values of the initial slopes $d\bar{\mu}/dc$ for Co, Mn, Cr, V²⁶ in $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ may

easily be interpreted in terms of electronic structure.²⁷ Let us call Z the impurity excess charge as compared with Fe, and Z_{σ} the displaced charge by one impurity in the bands of spin σ (\uparrow or \downarrow). From the Friedel's sum rule, Z_{\uparrow} and Z_{\downarrow} are related to Z by:

$$Z = Z_{\uparrow} + Z_{\downarrow} \quad (3)$$

The variation of the mean magnetic moment $\bar{\mu}(c)$ in the dilute limit is given by: (neglecting the spin-orbit coupling)

$$\bar{\mu}(c) = \mu_{Fe} + c\mu_B (Z_{\uparrow} - Z_{\downarrow}) \quad (4)$$

From equations (3) and (4), the experimental values of $d\bar{\mu}/dc$ allow us to calculate the screening in each spin sub-band of $Fe_{79}P_{13}B_8$ when alloyed with impurities of the first transition series. (Table 2)

Some interesting conclusions may be drawn from this analysis. First, the $Fe_{79}P_{13}B_8$ matrix is a strong ferromagnet. For Co and Ni in $Fe_{79}P_{13}B_8$, the screening in the \uparrow bands is about zero. So, the rigid band model (and hence the Slater-Pauling curve) seems to be roughly valid for these impurities and the variations of $\bar{\mu}(c)$ may be approximated by:

$$\bar{\mu}(c) = \mu_{Fe} - cZ\mu_B \quad (5)$$

The fact that the screening in the \uparrow bands is very small for Co and Ni implies that the d_{\uparrow} bands of the matrix are filled up (small density of states at the Fermi level, like in the crystalline Ni and Co matrix).

Table 2. Displaced charge by one impurity in the bands ↑ or ↓ of $\text{Fe}_{79}\text{P}_{13}\text{B}_8$.

Impurity		V	Cr	Mn	Co	Ni
Displaced	Z_{\uparrow}	-4.4	-4	-2.6	+0.1	+0.2
charge	Z_{\downarrow}	1.4	2	1.6	0.9	1.8

This conclusion comes out on the following way. Neglecting the s-d hybridization, the total displaced charge in a completely filled d_{\uparrow} band is zero. Moreover, in a tight-binding description of the d-bands, the local density in each cell remains the same (and equal to 5) if all the states are occupied.²⁸ Thus, the band structure of amorphous $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ is basically the same as that of compounds like Fe_2B ²⁹, Fe_3P ³⁰, Fe_3C ³¹, as suggested before.³²

On the other hand, the negative values of Z_{\uparrow} for Mn, Cr, V in $\text{Fe}_{79}\text{P}_{13}\text{B}_8$ are a classical result obtained for the same impurities in a strong ferromagnet. According to the Friedel's model²⁷, repulsive potentials are large enough to repel d bound states from the d_{\uparrow} bands. These d bound states are hybridized with the s bands through the impurity potential. The occupation $n_{d\downarrow}$ of the corresponding virtual bound states (v.b.s.) is related to the total displaced charge by $Z_{\uparrow} = n_{d\downarrow} - 5$. Thus, the v.b.s. are half-emptied for Mn and almost emptied for V in $\text{Fe}_{79}\text{P}_{13}\text{B}_8$.

2.2 - Estimate of the individual moments μ_{Fe} and μ_{Ni} . We try to give a consistent picture of equations (1) and (2) by means of reasonable assumptions on the respective variation of μ_{Fe} and μ_{Ni} . We assume that the variation of μ_{Fe} and μ_{Ni} in the amorphous (Ni, Fe) PB alloys has to be roughly the same as that observed by neutron diffraction³³ and obtained by calculation³⁴ in crystalline Fe-Ni alloys. The fact that $\text{Ni}_{79}\text{P}_{13}\text{B}_8$ is "nearly magnetic" (like crystalline Pd) suggests also an analogy with crystalline Pd-Fe alloys. Thus, we make several hypotheses on the variation of $\bar{\mu}_{\text{Fe}}$ and μ_{Ni} as Crangle did for the Pd-Fe system.³⁵ First, if μ_{Ni} remains constant and equal to zero, as in $\text{Ni}_{79}\text{P}_{13}\text{B}_8$, μ_{Fe} would increase slightly from 2.8 at $y = 1$ up to

$3\mu_B$ at $y = 30$, and then decrease down to $2.02\mu_B$ for $\text{Fe}_{79}\text{P}_{13}\text{B}_8$. Such a variation of μ_{Fe} is unlikely as compared with the case of crystalline Fe-Ni.^{33,34} Second, if μ_{Fe} increases with x according to: $\mu_{\text{Fe}} = (2.02 + 0.75x)\mu_B$, then the value of μ_{Ni} would increase from $y = 1$ to $y = 30$ at.% Fe up to a maximum of $0.15\mu_B$; for $y > 30$, μ_{Ni} would be zero (hypothesis 1 on Fig. 6). Such a variation of μ_{Ni} is unlikely according to ref. 33,34. Finally, we assume that μ_{Fe} remains constant and equal to $2.02\mu_B$. Then μ_{Ni} increases when y increases from 1 to 30 and remains roughly constant and equal to $0.40\mu_B$ for $y \geq 30$ (hypothesis 2 on Fig. 6). Such a picture seems to be most likely. Thus, the Ni atoms would be progressively polarized when the Fe content increases like Pd in the crystalline Pd-Fe system.

C. Resistivity Measurements.

The temperature dependence of the electrical resistivity $\rho(T)$ for all the samples may be expressed at low temperature ($4.2 < T < 60^\circ\text{K}$) by:

$$\rho(T) = \rho_0 + A \log T + B T^2 \quad (6)$$

At higher temperature ($T > 100^\circ\text{K}$), $\rho(T)$ becomes proportional to T . The determination of the residual resistivity ρ_0 is very inaccurate because of the difficulty in measuring the geometrical factor of the foils. This problem is avoided by using the normalized coefficients: $a = A/\rho_0$ and $b = B/\rho_0$. The coefficients a and b were calculated by a non-linear least-squares computer program used in ref. 36. The coefficient b displays a well-pronounced maximum between 10 and 20 at.% Fe, i.e.

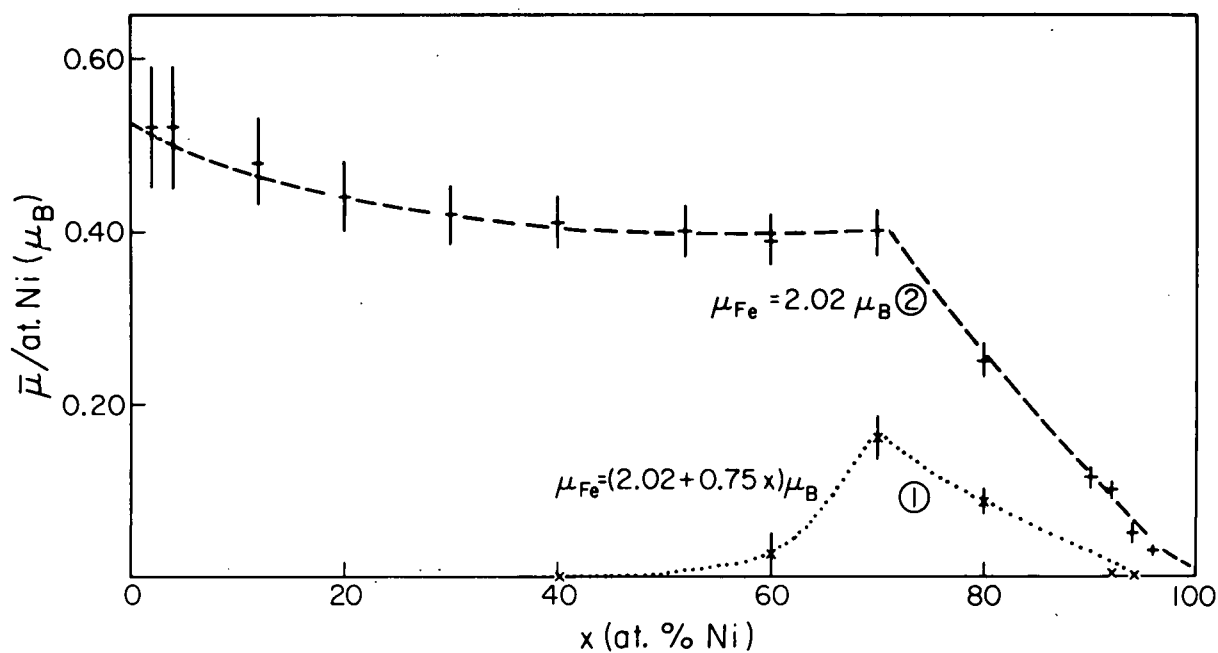


Fig. 6. Estimate of the magnetic moment per Ni atom as a function of Ni content in amorphous $(\text{Fe}_{100-x}\text{Ni}_x)_{79}\text{P}_{13}\text{B}_8$ alloys.

Hypothesis 1 : $\mu_{Fe} = (2.02 + 0.75x)\mu_B$.

Hypothesis 2 : $\mu_{Fe} = \text{constant} = 2.02\mu_B$.

around the critical concentration for ferromagnetism. Similar dependence on concentration was found for many concentrated alloys in the critical region: Ni based alloys was Pd³⁷, Rh³⁸, Cr, V, Ru, Mo¹⁹, and Fe based alloys with Cr³⁹, Ru.⁴⁰ This behavior is commonly explained by a spin fluctuation scattering especially important in this region of strong local enhancement. The coefficient α and the temperature of the resistivity minimum have a maximum value for samples with 4 to 6 at. % Fe. The fact that this maximum occurs in a region where the magnetic inhomogeneities are particularly important may suggest a magnetic origin for the resistivity minimum. Magnetoresistivity measurements are planned for clarifying this point.

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