# PROPERTIES OF AMORPHOUS Fe-P-B ALLOYS

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## California Institute of Technology W. M. Keck Laboratory of Engineering Materials

# ELECTRONIC AND MAGNETIC PROPERTIES OF AMORPHOUS Fe-P-B ALLOYS

by

J. Durand and M. Yung

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#### ABSTRACT

The ternary diagram of the amorphous phase in the splat-cooled Fe-P-B system was investigated by X-ray diffraction measurements. Alloys were found to be amorphous within the Fe concentration range  $75 \le c_{Fe} \le 83$  at.%. For a fixed Fe concentration, B substitutes for P over a large scale, the maximum substitution occurring for  $c_{F,o}$  = 80 at.%. Concentration dependence of the electronic and magnetic properties was systematically studied in alloys within the whole amorphous diagram by means of bulk magnetization  $\overline{u}$  (4.2°K), Curie temperature  $T_{c}$  and electrical resistivity  $\rho$  measurements from  $4.2^{\circ}$ K up to the amorphous-crystal transformation. The composition dependence of  $\overline{\mu}$  and  $T_{_{\hbox{\scriptsize C}}}$  when B substitutes for P at  $c_{_{\hbox{\scriptsize Fe}}}$ constant suggests the existence of two different short range orders in the amorphous alloys, corresponding to the  $\,\varepsilon\,$  and  $\,\varepsilon_{\,1}\,$  crystal structures in the Fe  $_3P_{1-x}^{}B_x^{}$  compounds. The concentration dependence of  $\overline{\mu}$  and T<sub>c</sub> when P (or B) substitutes for Fe at c<sub>B</sub> (or c<sub>D</sub>) constant is explained by a comparison between the amorphous Fe and the fcc Fe. A minimum in the variation of  $\rho$  as a function of the temperature T occurs at  $T_m$ . The temperature dependence of  $\rho$  is expressed by a phenomenological law:

$$\rho(T) = \rho_0 + A \log T + B T^2 + CT.$$

The concentration dependence of  $T_{m}$  and of the coefficients A and B is discussed in relation with the magnetic and structural properties of the amorphous Fe-P-B alloys.

#### I. INTRODUCTION

In a first report 1 it has been shown that the substitution of B for P in a series of amorphous  $Fe_{79}^{P}e_{21-y}^{B}$  alloys results in an increase of both the Curie temperature  $T_c$  and the mean magnetization  $\overline{\mu}$  averaged over all atoms in the alloy. Moreover, a change of slope for the variation of  $T_c$  and  $\overline{\mu}$  as a function of y occurs at a value of y which corresponds fairly well to a change of crystal structure ( $\varepsilon \rightarrow \varepsilon_1$ ) in the ternary Fe<sub>3</sub>P<sub>1-c</sub>B<sub>c</sub> compounds. <sup>2</sup> In order to check whether this phenomenon was fortuitous or not, we studied the variation of  $\mathbf{T}_{\mathbf{c}}$  and  $\overline{\mathbf{u}}$  for as many as possible Fe-P-B alloys available in the amorphous phase. The ternary diagram of the amorphous phase in the splat-cooled Fe-P-B system is presented in section III. The variation of  $T_c$  and  $\overline{\mu}$ as a function of y when B substitutes for P at  $c_{\mbox{Fe}}$  constant (75  $\leq$  c<sub>Fe</sub>  $\leq$  83 at.%) is discussed in section IV A. On the other hand, it has been found that  $T_c$  and  $\overline{\mu}$  vary in an opposite way, i.e.  $T_c$ increases and  $\overline{\mu}$  decreases as a function of x (or y) when P (or B) substitutes for Fe, the content of the other metalloid being kept constant. The previous measurements  $^{1}$  were made on series of Fe $_{92-x}P_{x}B_{8}$  and  $Fe_{87-v}P_{13}B_v$  alloys. In this paper, we report the results obtained for all the  $Fe_{100-x-y}^{P}$   $B_{y}^{B}$  series available in the amorphous diagram (section IV B). Thus, we were able to obtain extropolated values of  $\mathbf{T}_{\mathbf{c}}$ and  $\overline{\mu}$  for the binary  $\text{Fe}_{100-x}P_x$  and  $\text{Fe}_{100-y}B_y$  amorphous systems. These values were found to be consistent with the results obtained for electrodeposited amorphous Fe-P alloys 3 and for some splat-cooled Fe-B alloys. Besides, some resistivity measurements as a function of temperature o(T) were undertaken on samples over the whole

amorphous phase. We report also (section IV C) some preliminary results on the concentration dependence of the different parameters involved in  $\rho(T)$ . Detail on experimental procedures may be found in ref. 1.

#### II. DIAGRAM OF THE AMORPHOUS PHASE

The ternary diagram of the amorphous phase in splat-cooled Fe-P-B system is shown on Fig. 1. As in many amorphous systems obtained by splat-cooling, the most favorable concentrations of transition metal is around 80 at.%. We succeeded in obtaining some single-phase amorphous  ${\rm Fe_{80}^B}_{20}$  alloys; but alloys with 19 and 21 at. % B exhibited some traces of microcrystalline phases. On the P side, we did not obtain any good amorphous binary alloy with  $17 \le x \le 21$  at.%. Substitution of B for P is possible to a large extent for  ${\rm c_{Fe}}=79$ , 80 and 81 at.%. The limit defined by  ${\rm c_{Fe}}=82$  at.% seems to be absolute. For  ${\rm c_{Fe}}<79$  at.%, one still can obtain some single-phase amorphous, but the foils were found to be more and more brittle when  ${\rm c_{Fe}}$  decreases down to 75 at.%, and the efficiency became poor (one amorphous foil out of a set of five). In the 75 at.% line, only the  ${\rm Fe_{79}^P}_6{\rm B_{19}}$  alloy was found to be a good amorphous.

In order to obtain an estimate of the influence of P and B on the stability of the amorphous phase, we compared the temperatures at which the resistivity drops down for samples of  $Fe_{79}P_{21-y}B_y$  heated up at a rate of  $3^{\circ}K/min$ . These so called "crystallization temperatures" defined for the same rate of heating were found to increase proportionately to y up to y = 13 at.% B. The slope is about  $3^{\circ}K/at.\%$  B. For

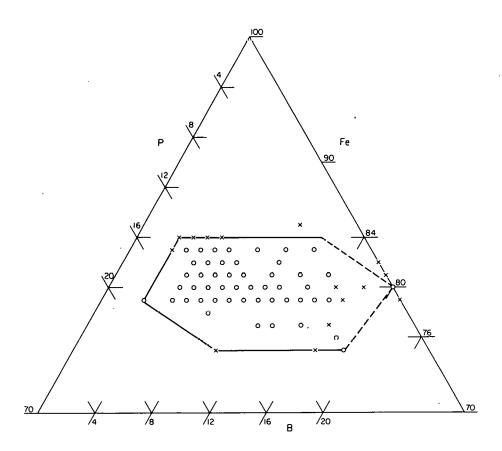


Fig. 1. Ternary diagram of the amorphous phase in splat-cooled Fe-P-B alloys. Alloys with traces of microcrystalline phases (X). Single-phase amorphous (O).

y > 13 at.% B, the "crystallization temperature" starts to decrease. These results are in qualitative agreement with the results obtained by Polk and Chen.  $^4$ 

#### III. RESULTS AND DISCUSSION

### A. Substitution of B for P at c<sub>Fe</sub> constant.

The results of our measurements of  $T_c$  (Fig. 2) and of  $\overline{\mu}$  (Fig. 3) are plotted as a function of the B content y. The results reported before are confirmed. When B substitutes for P, the Fe content being kept constant, the linear variation of  $T_c$  as a function of y changes of slope for some value of y. Below this concentration,  $T_c$  increases at a rate of 5 to  $7^{\circ}$ K/at.% B; above, the slope is smaller: between 2.5 and  $4^{\circ}$ K/at.% B. For comparison, we normalize the y critical concentration by writing:  $c_{crit} = y_{crit}/x + y$  (see Table 1).

As shown in Fig. 3, the variation of  $\overline{\mu}$  as a function of y changes drastically for some value of y. Normalized values of the critical concentration are listed on Table 1. This behavior has to be compared with a similar feature in the crystalline  $\text{Fe}_{75}\text{P}_{25\text{-y}}\text{B}_{y}$  alloys. These ternary compounds have the crystal structure of  $\epsilon$  -  $\text{Fe}_{3}\text{P}$  (tetragonal of the Ni<sub>3</sub>P type) up to y = 8.2 at.% B. For 8.2 < y < 12.3 at.% B, there is a solubility gap. For 12.3 < y < 24.4 at.% B, the compounds crystallize in a new ternary phase called  $\epsilon_{1}\text{-Fe}_{3}\text{P}_{1\text{-c}}\text{B}_{c}$  (tetragonal also, but not isomorphous with Ni<sub>3</sub>P). The variation of the magnetic properties as a function of y were found to be different in the  $\epsilon$  and  $\epsilon_{1}$  phases. The results of Fruchart et al. are collected for comparison in Fig. 3 for  $\overline{\mu}$  and in Fig. 2 (insert) for  $T_{c}$ . In the  $\epsilon$  phase,  $T_{c}$  and  $\overline{\mu}$ 

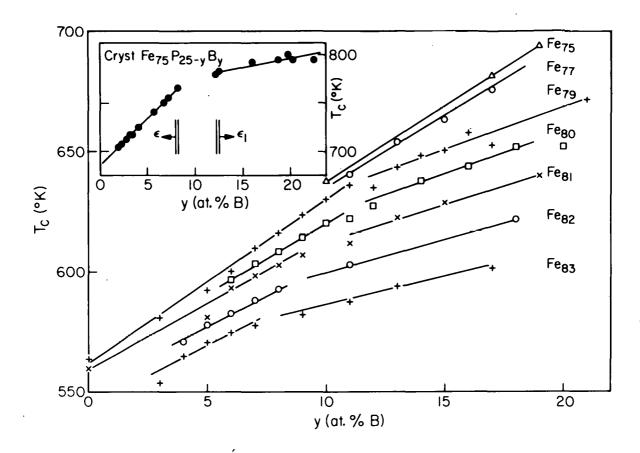


Fig. 2. Curie temperature  $T_c$  versus B content y when B substitutes for P in amorphous  $Fe_cP_{100-c-y}B_y$  alloys with  $75 \le c_{Fe} \le 83$  at.%. Insert:  $T_c$  versus y in crystalline  $Fe_{75}P_{25-y}B_y$  compounds (ref. 5).

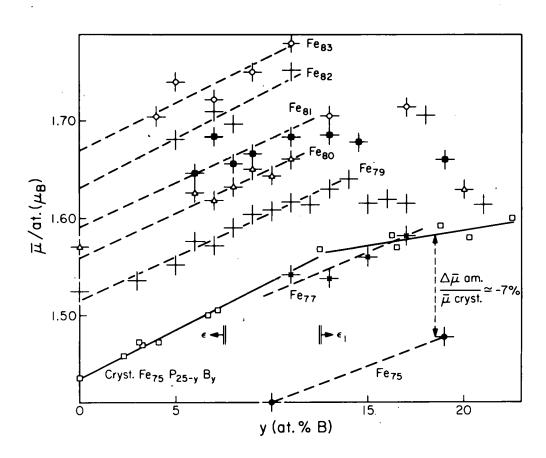


Fig. 3. Mean atomic moment per atom of alloy  $\overline{\mu}/at$ , as a function of B content y when B substitutes for P in amorphous  $Fe_c P_{100-c-y} B_y \text{ alloys with } 75 \le c_{Fe} \le 83 \text{ at.} \% \text{ Fe. Values obtained in crystalline } Fe_{75} P_{25-y} B_y \text{ compounds (ref. 5) are also included for comparison (<math>\square$ ).}

increase at a rate of 9.5 K/at.% B and 0.97  $\mu_B$ /at. B, respectively. In the  $\epsilon_1$  phase, the slopes are definitely smaller and rather poorly defined;  $d\mu/dy$  is estimated to be about 0.30  $\mu_B$ /at. B. The values of critical concentration for the change of slope are not accurate, because of the solubility gap. But, as in our amorphous alloys, this concentration is a little higher for  $\overline{\mu}$  than for  $T_c$  (see Table 1). The rigid band model predicts an increase of  $2\mu_B$ / at. B when B substitutes for P, from the difference of s p electrons between P and B. Actually, the experimental values of  $d\overline{\mu}/dy$  are much smaller than  $2\mu_B$  in both phases. Moreover, the rigid band model fails to explain that the variations of  $\overline{\mu}$  and  $T_c$  are crystal structure dependent. This phenomenon was tentatively explained by a contraction of the FeIII-FeIII distances in the  $\epsilon_1$  phase.

For the low values of y in the amorphous case, the slope of  $\overline{\mu}(y)$  ( $d\overline{\mu}/dy = 0.90 \,\mu_B/at$ . B) is quite similar to the slope in the  $\varepsilon$ -phase of the crystalline compounds. For high y values,  $\overline{\mu}(y)$  seems to be roughly constant. On the other hand, the critical concentrations are roughly the same in both amorphous and crystalline systems, so that the same physical explanation has to hold in both cases.

Two conclusions may be drawn from our study. First, the magnetic properties of these crystalline compounds and of these amorphous alloys are mainly determined by short-range interactions. The short-range order (SRO) is basically the same in the amorphous alloys and in their crystalline counterpart. It is a well established result from X-ray and neutron diffraction measurements  $^6$  that in the amorphous alloys of the transition-metal metalloid type, the coordination numbers (CN) for the metal and metalloid atoms are the same in the amorphous alloy and in the equivalent compound. In our case, the  $\varepsilon$  and  $\varepsilon_1$  phases

Table 1. Critical concentration for the change of slope in the variation of T  $_c$  and  $\overline{\mu}$  in the Fe-P-B amorphous alloys and in crystalline  ${^{Fe}3}^{P}{_{1-c}}^{B}{_c}.$ 

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<b>,</b> ·	Cryst. Fe <sub>3</sub> PB	Fe <sub>79</sub>	Fe <sub>80</sub>	Fe <sub>81</sub>	Fe <sub>82</sub>	Fe <sub>83.</sub>
T <sub>c</sub>	c <sub>crit</sub> = 0.50	0.57	0.55	0.58	0.55	0.59
μ	c <sub>crit</sub> = 0.68	0.71	0.60	0.63	0.67	0.71

have the same CN but differ only by the interatomic distances. Our study suggests that the two different SRO, corresponding to the two  $\varepsilon$  and  $\varepsilon_1$  phases, differ also by the mean interatomic distances.

Our second conclusion is about the effect of the long-range disorder on the magnetic properties of the amorphous Fe-P-B alloys. This effect is small in any case. It seems to be a little more important in the  $\varepsilon_1$  region than in the  $\varepsilon$  region where the slopes were found to be about the same in both amorphous and crystalline cases. For the  $\varepsilon_1$  region, this effect is evaluated by comparison of the values of  $T_c$  (694 and 794°K, respectively) and  $\overline{\mu}$  (1.48 and 1.59  $\mu_B$  per atom, respectively) for the amorphous Fe<sub>75</sub>P<sub>6</sub>B<sub>19</sub> alloy and the compound of same composition (Fig. 3). The values in the amorphous alloy are lowered by 7% for  $\overline{\mu}$  and by 12% for  $T_c$ . This effect is a little larger than that observed in Fe-P-C (5% for  $\overline{\mu}$  and 4% for  $T_c$ ). On the other hand, as in amorphous Gd<sub>80</sub>Au<sub>20</sub>, the effect of amorphousness seems to be more detrimental for  $T_c$  than for  $\overline{\mu}$ .

B. Substitution of P (or B) for Fe, the content of the other metalloid being kept constant.

The results reported in ref. 1 for the  $\mathrm{Fe}_{92-x}\mathrm{P}_x\mathrm{B}_8$  and the  $\mathrm{Fe}_{87-y}\mathrm{P}_{13}\mathrm{B}_y$  alloys are confirmed:  $\mathrm{T}_c$  and  $\overline{\mu}$  vary in an opposite way for all the sets of concentrations investigated (compare Fig. 4, 5 and Fig. 8, 9). The influence of the substitution of P for Fe on  $\mathrm{T}_c$  at different constant values of  $\mathrm{y}_o$  is shown in Fig. 4. The results for the substitution of B for Fe are plotted in Fig. 5 for different constant values of  $\mathrm{x}_o$ . Linear extrapolations to  $\mathrm{x}=0$  in Fig. 4 and to  $\mathrm{y}=0$  in Fig. 5 allow us to define the values of  $\mathrm{T}_c$  for the concentrations  $\mathrm{y}_o$  and  $\mathrm{x}_o$  in the binary  $\mathrm{Fe}_{100-y}\mathrm{B}_y$ 

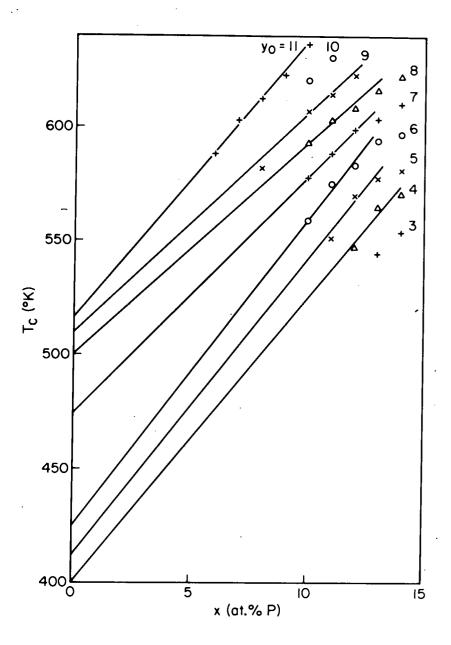


Fig. 4. Curie temperature versus P composition when P substitutes for Fe, the B content being kept constant, in amorphous  $Fe_{100-yo-x} {}^{P}_{x} {}^{B}_{yo} \text{ alloys, for some values of } \\ y_{o} (3 \le y_{o} \le 11 \text{ at. \% B}).$ 

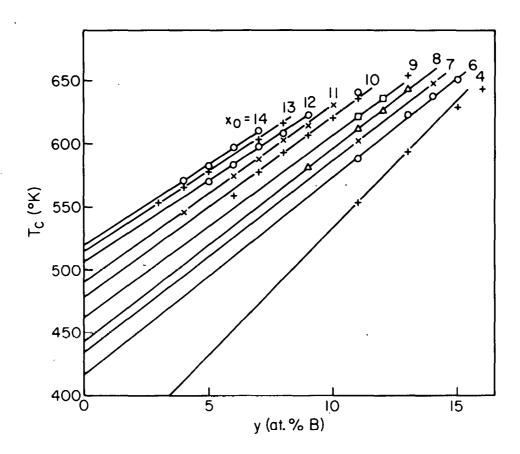


Fig. 5.  $T_c$  versus B composition when B substitutes for Fe, the P content  $x_o$  being kept constant, in amorphous  $Fe_{100-xo-y}P_{xo}B_y$  alloys, for some values of  $x_o$  (4  $\leq x_o \leq$  14 at.% P).

and Fe  $_{100-x}$  P $_x$  amorphous systems. These extrapolated values are plotted in Fig. 6 and 7, respectively. In Fig. 6 are shown also the measured values of T $_c$  for some binary Fe-B alloys (17  $\leq$  y  $\leq$  21). Among these latter alloys, only Fe $_{80}$ B $_{20}$  was found to be a single phase amorphous. The other ones (y = 17, 18, 19 and 21 at.% B) display some traces of microcrystalline phases, which do not affect sensibly the value of T $_c$  for the amorphous phase. This has been verified by measuring T $_c$  on "good" and "bad" amorphous foils of the same composition. The extrapolated values obtained from Fig. 4 are in the trend of the straight line defined by the binary Fe-B alloys. This line extrapolates for y = 0 to a value of about 320 K for T $_c$  of "pure amorphous Fe".

In Fig. 7, together with the extrapolated values of  $T_c$  obtained from Fig. 5, are plotted the values of  $T_c$  measured on electrodeposited amorphous Fe-P alloys and on microcrystalline splat-cooled alloys (x = 19, 20 and 21 at.%P). The variation of  $T_c$  as a function of x is fairly linear for x < 16 at.% P. It extrapolates at x = 0 to a value of  $T_c = 335^{\circ}$ K for "pure amorphous Fe", which is in good agreement with the estimate obtained for the Fe-B system. Let us note that the straight line defined for x < 16 at.% P would extrapolate for x = 25 at.% P to a value of  $685^{\circ}$ K very close to the value of  $T_c$  for crystalline Fe<sub>3</sub>P (716°K). For x > 16 at.%, the variation of  $T_c$  as a function of x is considerably smoother. The change in the variation of  $T_c$  occurring for  $16 \le x \le 17.5$  at.% P may be related to the singularities already observed around the same concentration in the variation of the quadrupolar electrical moment, the isomer shift, the width of the Curie transition and the electrical resistivity parameters. 3,9 These phenomena may be

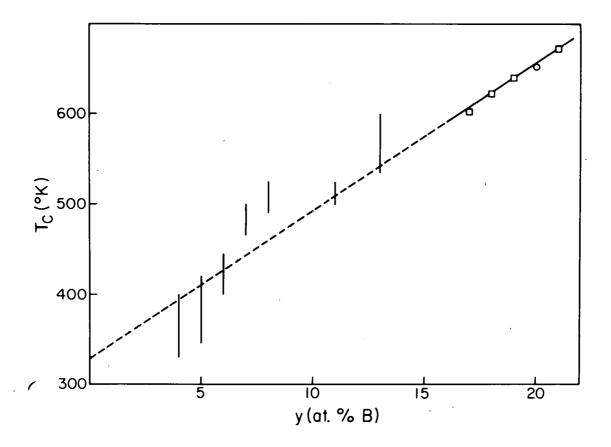
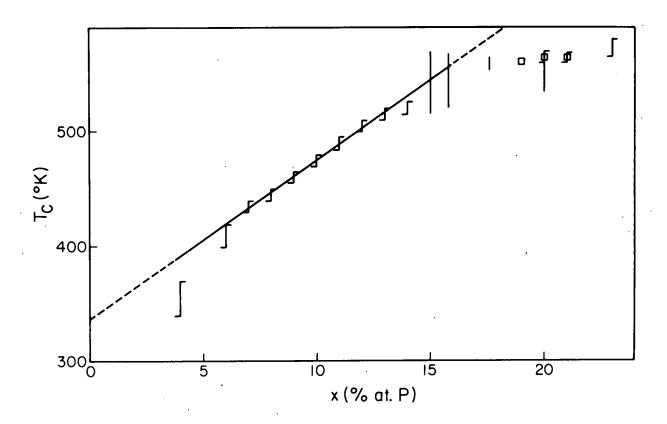


Fig. 6. T<sub>c</sub> versus B composition in amorphous Fe-B alloys.

Extropolated values obtained from Fig. 4 (bars). Values measured on binary splat-cooled Fe-B alloys ( ).



due to some structural modifications occurring around the eutectic composition.

The concentration dependence of  $\overline{\mu}$  was analyzed in the same way taking into account the two regions defined in IV A. The variation of  $\overline{\mu}$  as a function of x when P substitutes for Fe is shown in Fig. 8 for different constant values of  $y_0$ . Extrapolated values are plotted in Fig. 10. The results for the substitution of B for Fe are shown in Fig. 9 and the extrapolated values for the different sets of  $x_0$  are also plotted in Fig. 10. As for  $T_c$ , these extrapolated values are in good agreement with the measured values on splat-cooled binary Fe-B alloys, but they disagree with those obtained on electrodeposited Fe-P alloys. From a least squares fit, we obtain for Fe-B and Fe-P the following variations of the mean moment:

Fe-B: 
$$\overline{\mu}/at = (2.37 - 3.7 \text{ y}) \mu_B$$

Fe-P: 
$$\overline{\mu}/at = (2.33 - 3.9 x) \mu_B$$

The uncertainty in the values of the slopes and of the moment  $\mu_{Fe}$  for pure amorphous iron is probably as large as  $\pm$  0.2 $\mu_B$ . The agreement obtained for  $\mu_{Fe}$  for the two extrapolations is surprisingly good. Let us note that the least-squares line for Fe-P in Fig. 10 extrapolates for x = 25 at.% P to a value which is practically the experimental value for Fe<sub>3</sub>P. But, keeping in mind the dependence of  $\overline{\mu}$  and  $T_c$  on the detail of the crystal structure (or of the SRO) as described in IV A and the particular concentration dependence observed for the dilute limit in amorphous Fe-Si, Fe-Ge, Fe-O, one must be cautious about the results obtained from linear extrapolations between

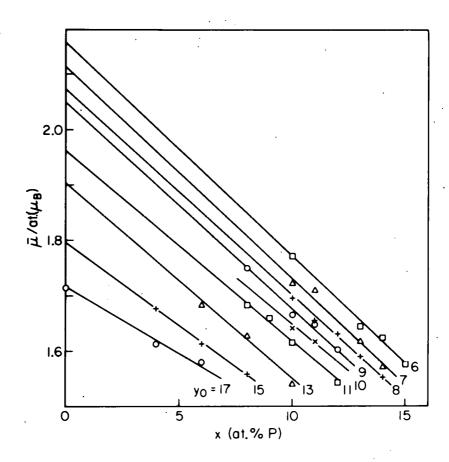


Fig. 8. Mean magnetic moment per atom of alloy as a function of P composition when P substitutes for Fe, the B content being kept constant in amorphous  $Fe_{100-yo-x}P_xB_{yo}$  alloys for some values of  $y_o(6 \le y_o \le 17 \text{ at.}\% \text{ B})$ .

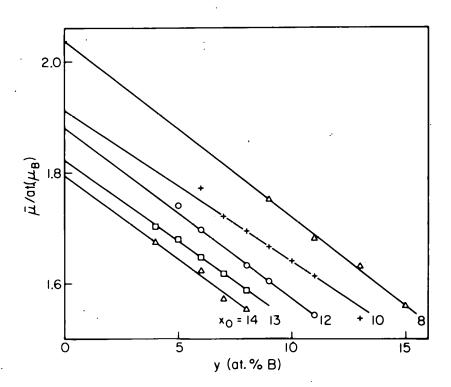


Fig. 9. Mean magnetic moment per atom of alloy as a function of B composition when B substitutes for Fe, the P content x<sub>o</sub> being kept constant in amorphous Fe<sub>100-x<sub>o</sub>-y<sup>P</sup>x<sub>o</sub> y for some values of x<sub>o</sub> (8 ≤ x<sub>o</sub> ≤ 14 at.% P).</sub>

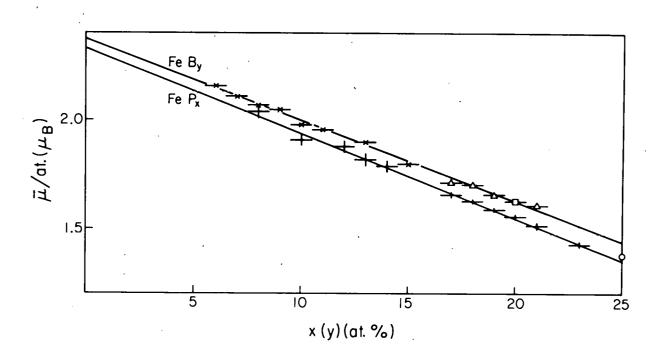


Fig. 10. Mean magnetic moment per atom of alloy as a function of P composition (+, extrapolated values) and as a function of B content (\*, extrapolated values-see text). Experimental values obtained on binary splat-cooled Fe-B alloys (Δ) are also included for comparison (only Fe<sub>80</sub>B<sub>20</sub> being a single-phase amorphous) (□).

0 and 25 at. % of metalloid. However, let us comment briefly on the values thus determined.

The concentration dependence of  $T_c$  as plotted in Fig. 6 and 7 gives for "pure amorphous Fe"  $T_c \simeq 300^{\circ} K$ . Actually the trend observed for low concentrations of P and B shows that  $T_c$  is probably less than  $300^{\circ} K$ . This is in good agreement with the extrapolated values obtained from measurements on amorphous Fe-Tb ( $T_c = 200^{\circ} K$ ) and Re-Fe ( $T_c = 270^{\circ} K$ ) alloys. <sup>14</sup> On the other hand, our value for  $\mu_{Fe}$  is close to that assumed by Yamaushi et al. <sup>15</sup> (2.6  $\mu_B$ ). The problem arising from the opposite variation of  $\pi$  and  $T_c$  in amorphous Fe-P and Fe-B may be discussed by different approaches: molecular field model <sup>16</sup>, mixture of localized and itinerant magnetism...A comparison between amorphous Fe and crystalline Fe in a fcc environment was suggested in ref. 1. More experimental information is needed for solving this problem (variation of  $T_c$  as a function of pressure, hff studies).

It has been a custom to discuss the variation of the magnetic moment in transition-metal metalloid compounds or alloys in terms of electron transfer from metalloid s-p electrons to transition metal d electrons. Assuming that this mechanism is realistic and crystal structure (or SRO) independent, B would donate 1.4 ( $\pm$  0.2) and P, 1.6 ( $\pm$  0.2) electrons to the Fe d bands. These values agree more or less with those already proposed for B <sup>15,17</sup>, but are lower than those suggested for P. They are lower than the estimates in crystalline cases. <sup>18</sup>

#### C. Resistivity measurements.

Electrical resistivity measurements as a function of temperature

 $_0$ (T) were undertaken in an effort to find a better understanding of the variation already observed for the magnetic and structural properties. A resistivity minimum was observed at a temperature  $T_m$  for all the samples investigated. The low temperature part  $(4.2 \le T \le 60^{\circ} \text{K})$  of  $_0$ (T) was found to follow the phenomenological law:

$$_0(T) = _0 + A \log T + B T^2$$

In order to avoid the uncertainties in the determination of the geometrical factor, we studied the normalized coefficients  $a = A/\rho_0$  and  $b = B/\rho_0$ . We summarize our preliminary results on the concentration dependence of a and b in the series  $Fe_{79}P_{21-y}B_y$ ,  $Fe_{87-y}P_{13}B_y$  and  $Fe_{92-x}P_xB_8$ . The coefficients a and b were calculated by a non-linear least-squares computer program used in ref. 9. When B substitutes for P at c<sub>Fe</sub> constant, both a and b increase when the B content increases. Both increase at about the same rate, so that  $T_m$  defined by  $(-a/2b)^{\frac{1}{2}}$  is roughly constant. We hardly see a slight trend to higher values of Tm for the high y values. When B substitutes for Fe at x constant, there is an increase of  $T_m$  proportional to y in the  $Fe_{87-v}P_{13}B_v$  series. In the Fe<sub>92-x</sub>P<sub>x</sub>B<sub>8</sub> series, the effect due to the substitution of P for Fe seems to be smaller than in the substitution of B for Fe. But the data scattering is too large for any slope to be determined with accuracy. Measurements are needed on more samples before the concentration dependence of a, b and T can be discussed quantitatively. But one qualitative conclusion may already be drawn out: the concentration dependence of T<sub>m</sub> resembles much more the variation of the Curie temperature than the variation of the magnetic moment. Whether the

composition dependence of  $T_c$  and  $T_m$  would be related to the existence of different magnetic states of Fe in a compact environment or to the variation of the mean interatomic distances is still an open problem. Besides, the physical meaning of the coefficient b is not apparent in our case, although it can be attributed, in other amorphous systems <sup>19</sup>, to a spin fluctuations mechanism. On the other hand, if the mechanism giving rise to a resistivity minimum seems to be magnetic in origin in our system, the explanation suggested <sup>20</sup> for this minimum in amorphous Fe-P-C does not look very likely in the amorphous Fe-P-B. From Mössbauer experiments performed on our samples <sup>21</sup>, there is no experimental evidence for a distribution of the local field  $H_o$  at Fe sites down to  $H_o = 0$ . A complete report of our resistivity measurements will be published elsewhere.

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