SOFT MAGNETIC PROPERTIES OF AMORPHOUS Fe-Co-Ni BASE ALLOYS WITH Si AND B

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SOFT MAGNETIC PROPERTIES OF AMORPHOUS Fe-Co-Ni BASE ALLOYS WITH Si AND B

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ABSTRACT

Amorphous alloys of Fe-Co-Ni-Si-B system with a wide composition have been produced by a conventional rapid quenching method, and their soft magnetic properties have been examined. The Fe-rich alloys having a high saturation magnetization ($\sim 1.6 \times 10^4$ G) exhibit a rectangular-type B-H hysteresis loop with low coercive force of about 0.01 Oe when the alloys are subjected by a magnetic field cooling. On the other hand, the Co-rich non-magnetostrictive alloys exhibit a high effective permeability ($50 - 100 \times 10^3$) at the initial magnetization and audio frequency range. The detailed compositions and heattreatments in obtaining the best soft magnetic properties, and the characteristics which may be interest in engineering applications will be reviewed.

INTRODUCTION

Recently, a number of researchers have been taking a considerable interest in the characteristics of amorphous alloys produced by a continuous rapid quenching method. The first study by using a long ribbon specimen was made on the mechanical properties of amorphous Pd-Si alloy by Masumoto and Maddin 1). Later on, many studies by using such an amorphous ribbon specimen have extensively been developed in various fields particularly magnetics, mechanics and chemistry. In 1974 - 1975, soft magnetic properties of the ferromagnetic amorphous alloys produced by the same method were examined independently and simultaneously by our group ($Fe_{80}P_{13}C_7$)2) and by Egami et al ($Fe_{80}P_{16}B_1C_3$ and $Fe_{40}Ni_{40}P_{14}B_6$)3). All of the data showed that the as-quenched alloys had low coercive force (about 0.1 - 0.01 Oe) and high permeability (about 10^5 in the maximum permeability), and the suitably annealed alloys had the values comparable to those of conventional soft magnetic materials. Subsequently, from the studies concerning the magnetostrictive effect on soft magnetic properties, non-magnetostrictive alloys were found in $Co_{70}Fe_5Si_{15}B_{10}$ by our group 4), $Co_{72}Fe_3P_{15}B_6Al_3$ by Sherwood et al 5) and $Co_{74}Fe_6B_{20}$ by O'Handly et al 6). These alloys show remarkable soft magnetic properties characterized by 0.01 - 0.005 Oe in coercive force, $30 - 90 \times 10^3$ in maximum permeability and $7 - 10 \times 10^3$ in effective permeability at 1 - 10 KHz. On the other hand, it was noted very early that the amorphous alloys produced by the rapid quenchig method possessed high mechanical strength and hardness as well as high electrical resistance. It is, therefore, not surprizing that, sice then, the investigations on the amorphous ferromagnetic alloys have been extensively performed from engineering interest point of view. Nowadays, for instance, the zero magnetostrictive amorphous alloys are considered as materials useful for Permalloy-type applications and the high B_s Fe-rich amorphous alloys such as $Fe_{80}B_{20}$ (Luborsky et al 7)) are considered to be useful for power transformer applications where the Fe-Si steel are now used.

So far, the information available to evaluate amorphous alloys for use as soft magnetic materials was reviewed by Egami et al (1975) 8), Gyorgy et al (1976) 9), Luborsky (1977) 10), Takahashi (1977) 11) and ourselves (1977) 12). On the other hand, the fundamental studies on the behaviour and origin of ferromagnetism in amorphous state were covered by Cargill (1975) 13), Mizoguchi (1976) 14), Tsuei (1976) 15) and Hasegawa et al (1976) 16).

In order to find amorphous soft ferromagnets interest in various applications, we have further investigated the low field magnetic properties for a number of amorphous ferromagnetic alloys. In this paper we will review the experimental results on the Fe-Co-Ni-Si-B amorphous alloys which have quite recently been developed by the authors and coworkers. The selected topics are listed as follows: (1) preperations of amorphous alloys, (2) soft magnetic properties of Fe-Co-Ni base amorphous alloys with samll magnetostriction, and (3) soft magnetic properties of Fe-rich amorphous alloys with high saturation magnetization.

PREPARATIONS OF AMORPHOUS FERROMAGNETIC ALLOYS

All of amorphous alloys employed in the present work were prepared by the single roller-type quenching technique. The apparatus developed by ourselves 17) is principally similar to that appear in the report by Liebermann and Graham 18). The specimens produced are in form of long ribbon with about 20 - 70 μm in thickness and about 1 - 50 mm in width, depending on alloy composition. As an example, Photo. 1 shows some of the samples.

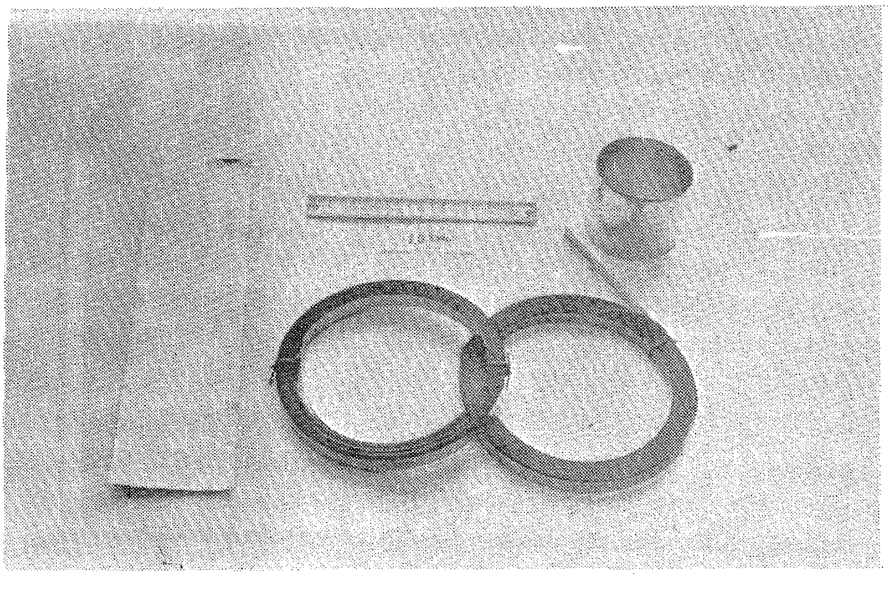

Photo. 1 Samples of amorphous magnets with a zero magnetostriction 19).

The alloy investigated are of the composition $T_{100-x}M_x$ where T represents one or more of Fe, Ni and Co, and M represents one or more of C, B, P, Si and Ge. x is in the range of about 15 - 30 at%. Figs. 1 - a and b show the formation range of amorphous phase for Fe-B-M and Fe-P-M, respectively. Also, Figs. 2 - a and b show the range for Ni-Si-B and Co-Si-B. In order to obtain technologically useful materials, the optimum composition of alloys should be chosen from various combination of metals and metalloids. Recently, our group has demonstrated that a Si-B system is mechanically very hard and structurally quite stable. Furthermore, this system is significant in soft magnetic properties as will be described as follows.

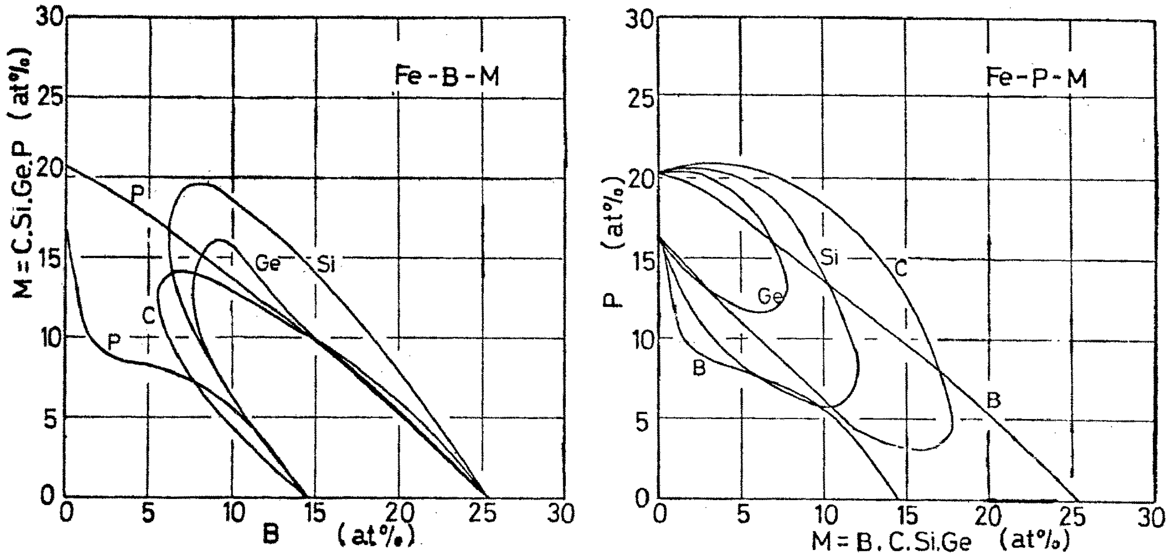

Fig. 1 Formation range of amorphous phase for Fe-B-M (figure a) and Fe-P-M (figure b). (after Naka et al [20])

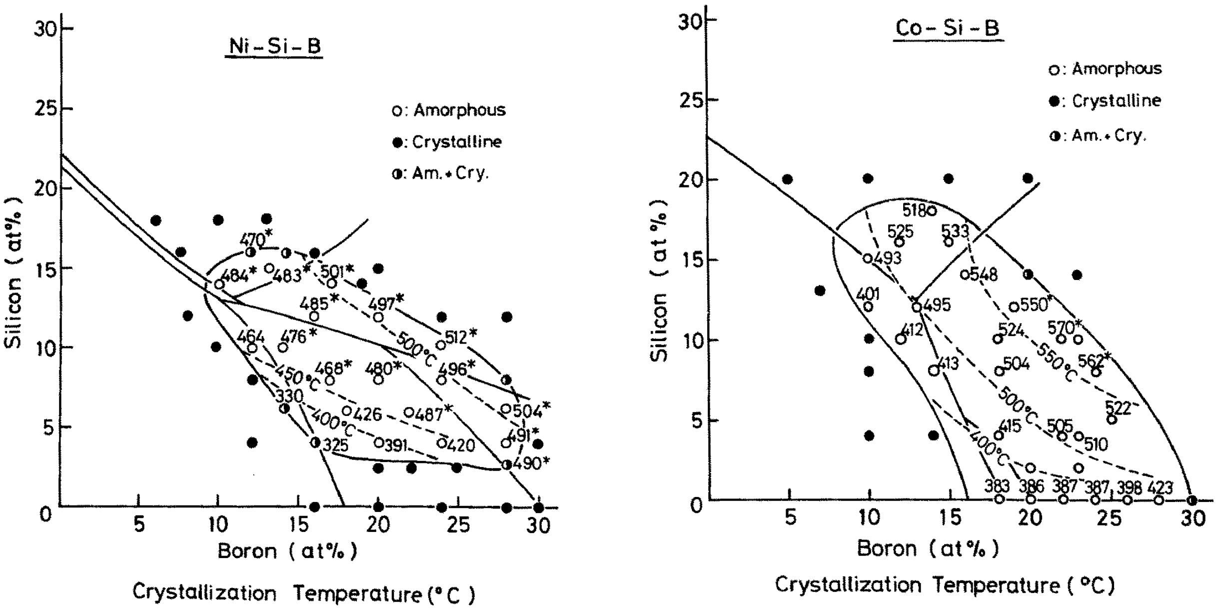

Fig. 2 Formation range of amorphous phase for Ni-Si-B (figure a) and Co-Si-B (figure b). The numbers represent the crystallization temperature (a) and the Vickers hardness (b).
(after Inoue et al[21])

SOFT MAGNETIC PROPERTIES OF Fe-Co-Ni BASE AMORPHOUS ALLOYS WITH SMALL MAGNETOSTRICTION

Because the amorphous alloy is macroscopically isotropic, it is considered that the amorphous ferromagnets are magnetically soft although the magnetostriction may play important role in the magnetization process. Previously, the authors reported that the coercive force of the Fe-Co base amorphous alloys was greatly reduced with decreasing the magnitude of the magnetostriction as a function of Co /(Co + Fe) 2). Recently, Ohnuma et al have carried out the extended study to the amorphous Fe-Co-Ni alloys and examined the magnetostrictive effect on low field magnetic properties22). Fig. 3 shows the saturation magnetic induction (B_s) at room temperature for the $(Fe\text{-}Ni\text{-}Co)_{78}Si_8B_{14}$ system. Fig. 4 represents the Curie temperature (T_c) of this system.

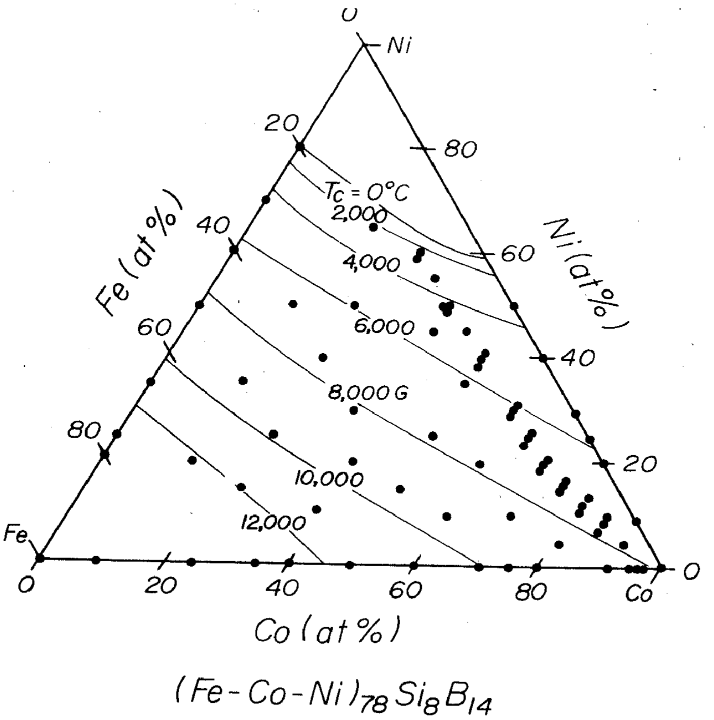

Fig. 3 Saturation magnetic induction at room temperature for the amorphous $(Fe\text{-}Ni\text{-}Co)_{78}Si_8B_{14}$ alloys. (after Ohnuma et al 22))

It can be seen that the ferromagnetism with T_c above room temperature exists over a wide compositional range, except for the Ni rich corner. In these figures, it should be noted that both B_s and T_c change continously and monotonically with respect to composition, in contrast to the discontinous change in B_s and T_c for the crystalline Fe-Co-Ni ternary alloys, which is often observed at the boundaries between different crystalline phases in the phase diagram.

Fig. 5 shows the saturation magnetostriction (λ_s) of the same amorphous alloys. The value of λ_s is as large as about 30 × 10^{-6} in the region of the Fe-rich alloys and takes a maximum of 35 × 10^{-6} near $(Co_{.15}Fe_{.85})_{78}Si_8B_{14}$. The λ_s decreases with decreasing Fe content and reaches zero at the composition indicated by $\lambda_s = 0$ in the figure. The λ_s becomes negative in the Co and/or Ni-rich side. The compositions representing zero

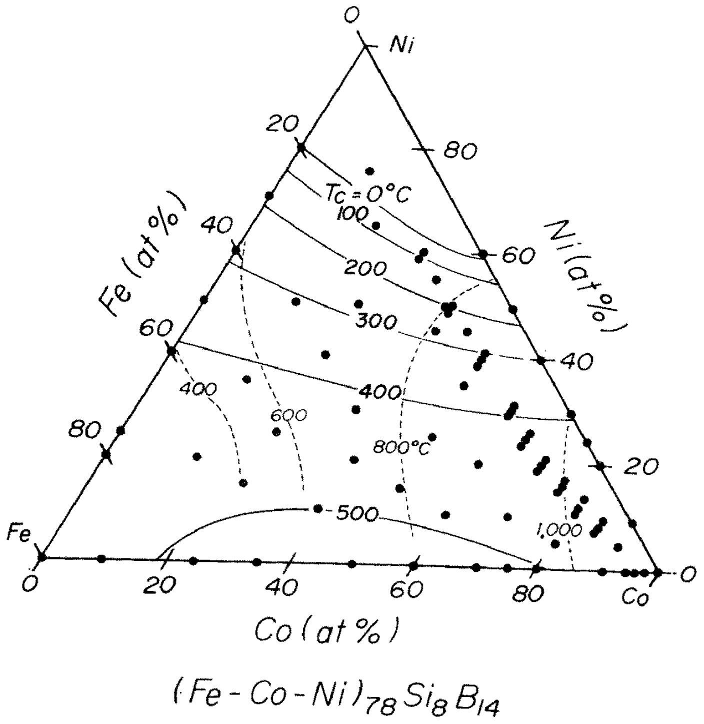

Fig. 4 Curie temperature for the same amorphous alloys in the figure 3. (after Ohnuma et al 22))

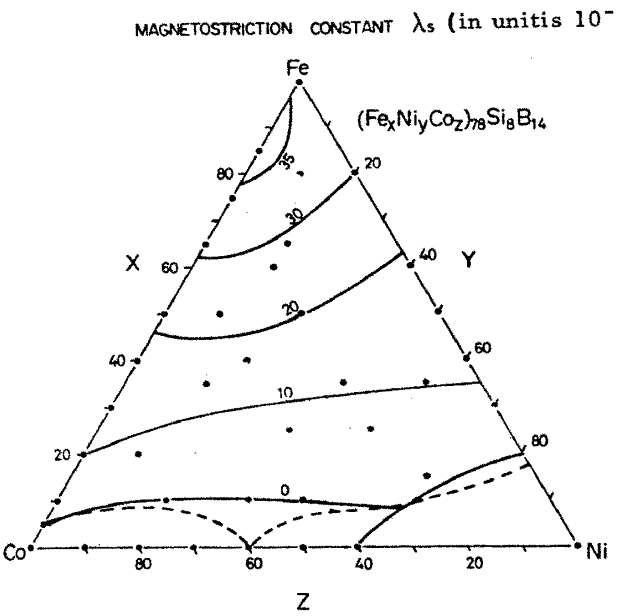

Fig. 5 Saturation magnetostriction for the same amorphous alloys in the figure 3. (after Jagielinski 23))

magnetostriction exists on a line connected smoothly from $Co_{95}Fe_5$ to $Ni_{80}Fe_{20}$. This line is different from that for the zero magnetostrictive crystalline alloys since the $Co_{60}Ni_{40}$ has also a zero magnetostriction in the crystalline state (see the dotted line in the figure).

Now, it is interesting to see the relationship between the compositional dependences of magnetostriction and of low field magnetic properties for the present amorphous alloys. As a typical quantity, the coercive forces (H_c) in the present amorphous alloys are plotted in Fig. 6. Compared with

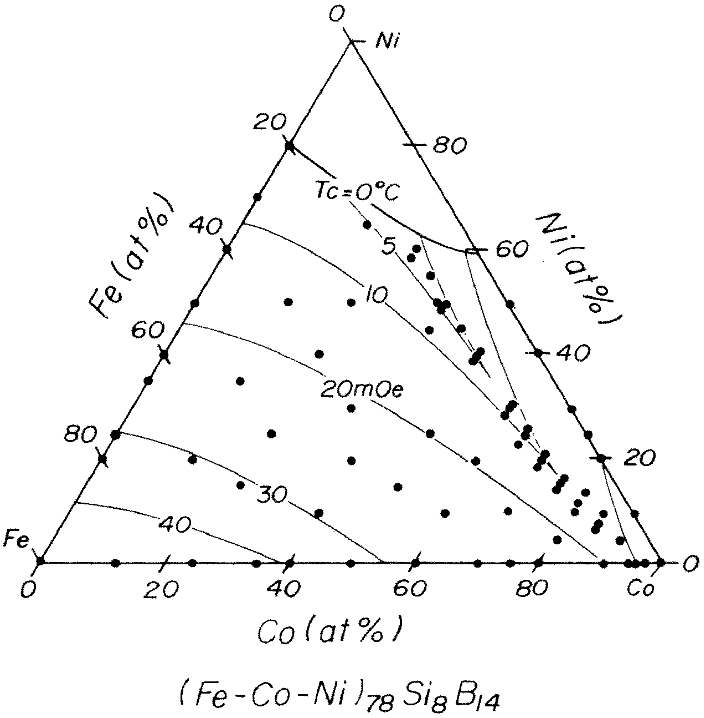

Fig. 6 Coercive force for the same amorphous alloys in the figure 3. (after Ohnuma et al [22])

the previous figure 5, one can find the significant fact that the value of H_c is extremely small at compositions where the magnetostriction is nearly zero. It is also noted that the H_c for the magnetostrictive alloys changes monotonicaly against composition. This is in contrast to the H_c vs composition curve for the crystalline alloys. That is, the coercive force in crystalline state varies in a complicated manner against composition owing to many factors such as magnetocrystalline anisotropy, magnetostriction and other parameters determined magnetostatically by the crystallographical situation. Thus, one recognizes that the amorphous alloy is magnetically very soft if the alloy has a vanishingly small magnetostriction.

According to the result by Ohnuma et al, the amorphous $(Fe-Co-Ni)_{78}Si_8B_{14}$ alloys exhibit about 2 mOe of H_c in the region of zero magnetostriction. This value is comparable to that for the conventional Permalloy. However, the B-H hysteresis loop of the as-quenched specimen is of rectangular type as shown in Fig. 7, and the initial permeability or the effective permeability at high frequency is too small to compare to that for Permalloy. In addition to the rectangular loop, the large Barkhausen effect in magnetization reversal can often be observed (see Fig. 7).

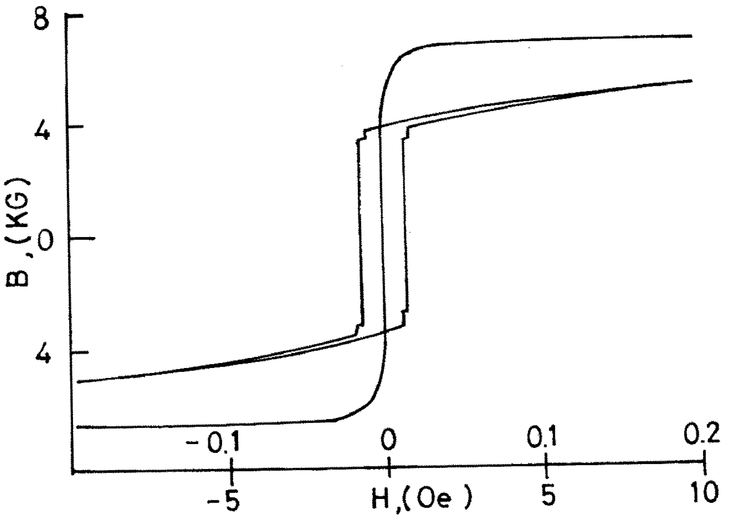

Fig. 7 Typical B-H hysteresis loop of as-quenched (Fe$_{.07}$Co$_{.82}$Ni$_{.1}$)$_{74}$Si$_{10}$B$_{16}$ amorphous alloy. (after Takahashi et al [24])

In order to improve the value of permeability, it is necessary to do the adequate heat treatment against the as-quenched alloys. The result obtained can be outlined as follows. Fig. 8 shows the effective permeability (μ_e) vs annealing temperature (T_a) curve for the amorphous alloy (Fe$_{.07}$Co$_{.82}$Ni$_{.1}$)$_{74}$Si$_{10}$B$_{16}$. The value of μ_e is in the order of 10^4 in the as-quenched state (that is, as-received state).

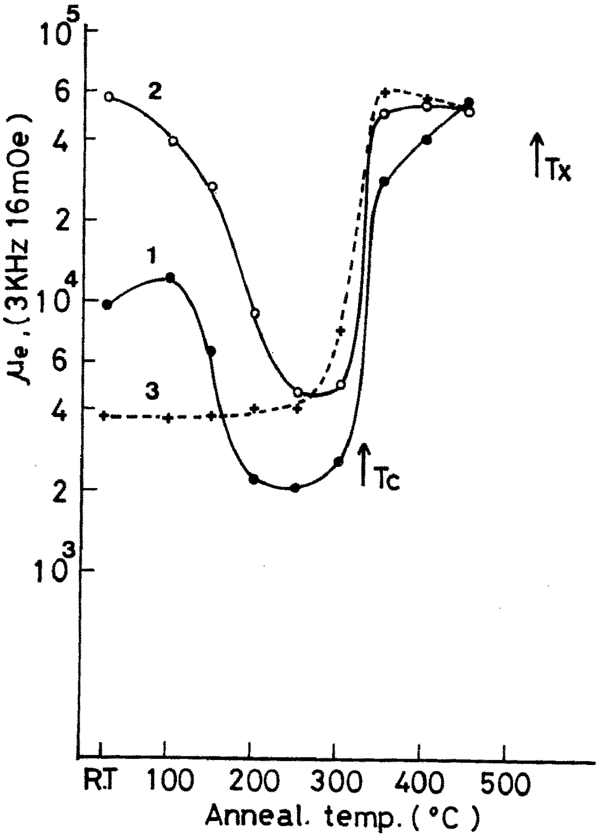

Fig. 8 Effective permeability (μ_e) vs annealing temperature (T_a) curve for the amorphous alloy (Fe$_{.07}$Co$_{.92}$Ni$_{.1}$)$_{74}$Si$_{10}$B$_{16}$.

Curve-1: annealed against the as-received specimen.
Curve-2: re-annealed against the specimen pre-annealed at 420°C.
(for Curve-1 and 2, the annealing was taken for 30 min and then the specimen was water-quenched)
Curve-3: slowly cooled with a 150°C/hr from 420°C to lower temperatures and then water-quenched. T_c and T_x represent the Curie and crystallization temperatures, respectively.
(after Takahashi et al $^{24)}$)

As seen in Curve-1, the μ_e increases up to about 6 × 10^4 by annealing at temperatures between the Curie temperature (T_c) and the crystallization temperature (T_x), although the μ_e becomes low due to the annealing below T_c. The high μ_e thus obtained decreases by re-annealing below T_c, but the same high value can be obtained when the annealing temperature is above T_c (see Curve-2). Both the results shown in Curves-1 and 2 have been obtained under the condition that the specimen is always water-quenched after the annealing. If the specimen is slowly cooled from temperatures above T_c to below T_c, only low values of μ_e can be obtained as shown in Curve-3. The observed annealing behaviour in μ_e suggests that the annealing above T_c followed by water-quenching is effective in obtaining a high μ_e. This important fact is further evident by the experimental result summarized in Fig. 9. That is, the μ_e in the case of the water-quenching takes the highest value and is nearly independent on the magnitude of T_c, while in both the cases of the air-cooling and furnace-cooling it depends strongly on T_c. For the alloys having higher Curie temperatures a faster cooling is necessarily required after annealing to obtain the highest permeability. In table 1 typical data thus obtained for several amorphous alloys are shown. The value of μ_e is superior as high as 50 - 100 × 10^3 which is comparable to those of the presently available commercial alloys.

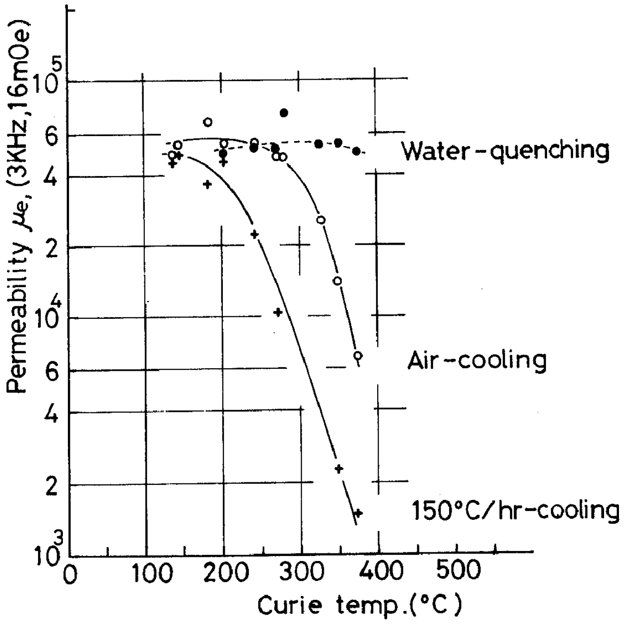

Fig. 9 Effective permeability for various compositional amorphous alloys having a nearly zero magnetostriction is plotted as a function of the Curie temperature.

Closed circles: water-quenched after pre-annealing at 450°C.
Open circles: air-cooled from 450°C to room temperature.
Crosses: furnace-cooled with a cooling rat of 150°C/hr.
(after Takahashi et al $^{24)}$)

Table 1 Amorphous magnetic alloys with high permeability

	A(Fe-Ni)	B(Fe-Co)	C(Fe-Ni-Co)	Commercial alloys	
				Supermalloy	Sendust
Sat. Mag. Ind., B_s(G)	13100	8400	6000	7700	10000
Coercive Force Hc(Oe)	0.006	0.002	0.001	0.01	0.005
Effect. Perm. (μe at 3KHz)	5000	50000	100000	30000 *	30000 *
Curie Temp. Tc(°C)	380	250	150	460	500
Satu. Magnetostric. ($\lambda s \times 10^6$)	+25	± 0.1	± 0.1	——	——
Resistivity (Ω-cm)	160×10^{-6}	180×10^{-6}	175×10^{-6}	10×10^{-6}	80×10^{-6}
Hardness (Hv)	880	910	900	120	500

* Initial Permeability (μo)

The large decrease in μ_e which is taken place by the annealing around 200 - 300°C below T_c or by the slow cooling is considered to be due to a magnetic after effect. Recently, the authors have found in the similar alloys that the coercive force and B-H hysteresis loss increase markdly during the annealing below T_c 25). And it has also been found that the hysteresis loss (W_h) obtained by annealing long enough increases with decreasing the annealing temperature and is closely related with the magnetic anisotropy induced by a magnetic field cooling. Since the induced magnetic anisotropy is large when the amorphous alloys include the Fe-Co pairs 26), the value of W_h becomes large in the Fe-Co base amorphous alloys (pseudo - binary alloy) but fairly small in the alloys based on only Fe or Co. Such an annealing behaviour in W_h may be explained in terms of the domain wall stabilization 25). That is, even in the absence of the external magnetic field, the spontaneous magnetization vectors oriented in compliance with the distribution of magnetic domain present during the annealing induce the local anisotropy. Such a local anisotropy restricts the displacement of domain walls 27). It seems that the annealing behaviour in μ_e is also originated from the domain wall stabilization. It has been observed that a reduction in μ_e takes place during aging even at room temperature particularly in the alloys having a high Curie temperature. Such an aging effect would make a serious trouble in applying the amorphous alloys as practical materials. In order to improve thermal stability in μ_e, the atomic structural relaxation by which the magnetic domain wall fixing is governed must be prevented by increasing the activation energy. This phenomenon may be related closely with the stability of amorphous structure and the problem will be solved by selecting the composition of alloys, especially the adequate combinations of metal and metalloid.

As far as low Curie temperature alloys are concerned, the permeability is fairly stable against aging. Fig. 10 shows the frequency dependence of effective permeability and loss factor for the zero magnetostrictive amorphous alloy $(Fe_{.06}Co_{.94})_{71}Si_{19}B_{10}$. From comparison with the commercial Permalloy, it is certainly seen that the amorphous alloy has excellent properties.

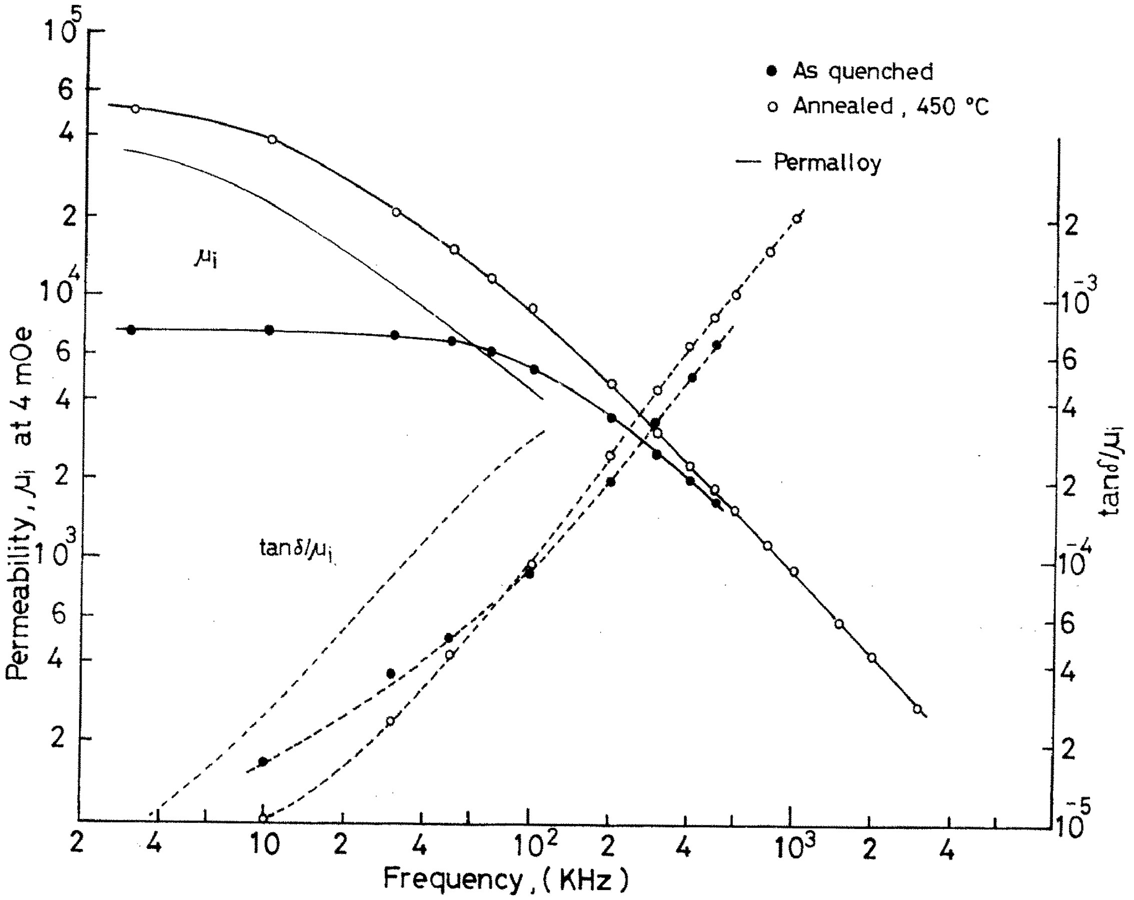

Fig. 10 Frequency dependence of effective permeability (μ_e) and loss factor ($\tan\delta/\mu_e$) for the zero magnetostrictive amorphous alloy $(Fe_{.06}Co_{.94})_{71}Si_{19}B_{10}$. The data for Permalloy are also shown for the comparison. (after Takahashi et al [24])

Next, the temperature dependence of μ_e for the low Curie temperature amorphous alloys will be examined. As shown in the previous figure 4, the Curie temperature decreases with increasing Ni content and becomes below room temperature. Fig. 11 shows the temperature dependence of μ_e for some Ni-rich amorphous alloys. It should be noted that the value of μ_e drops sharply around T_c but the value at temperature lower than T_c is extremely high on a level with that of the Ni-poor alloys. In general, crystalline alloys or ferrite also have a sharp change in μ_e around T_c presumably due to the Hoppkinson effect, but it has been known that the level of permeability is rather low in the low Curie temperature crystalline materials ($2 - 6 \times 10^3$ for the thermosensor ferrites, for example). Therefore, this type amorphous alloys may be considered as possible good thermosensor materials.

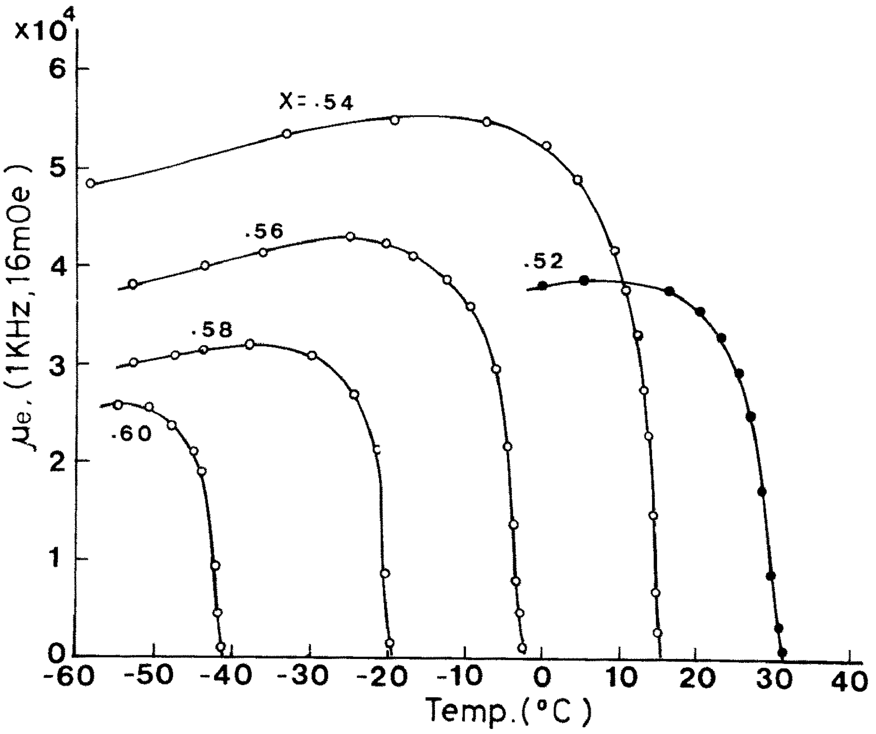

Fig. 11 Temperature dependence of effective permeability (μ_e) for the low Curie temperature amorphous alloys.
(after Okazaki et al [28])

SOFT MAGNETIC PROPERTIES OF Fe-RICH AMORPHOUS ALLOYS WITH HIGH B_s

As mentioned above, the Co and/or Ni rich amorphous alloys with a vanishingly small magnetostriction have a high permeability, but they possess the disadvantage that the saturation magnetic induction (B_s) is low. On the other hand, the amorphous alloys with high Fe content have higher values of B_s although they have a large magnetostriction. Such high B_s alloys may be useful as soft ferromagnetic materials for power devices, if the disadvantageous effect of large magnetostriction on magnetization process can be reduced.

It has already been pointed out by many investigators [29] that the values of B_s and T_c vary with the composition of metalloid elements as well as the composition of metal elements. The Fe-B alloys among various amorphous ferromagnets have considerably large values of B_s. Recently, Mitera et al [30] have further studied on the detailed properties for the $Fe_{80}(B_{1-x}Y_x)_{20}$ where Y is P, C, Si and Ge. In Figs. 12-a and b, their results on the magnetic moment per Fe atom (μ) and the Curie temperature (T_c) are shown. From these results, one can find that both μ and T_c increase in the sequence : $Fe_{80}(B\text{-}P)_{20} < Fe_{80}(B\text{-}C)_{20} < Fe_{80}B_{20} \lesssim Fe_{80}(B\text{-}Si)_{20} < Fe_{80}(B\text{-}Ge)_{20}$. Since the number of the valence electron is different between B, C and P, the result suggests that μ and T_c increase with decreasing the number of the valence electron of metalloids. On the other

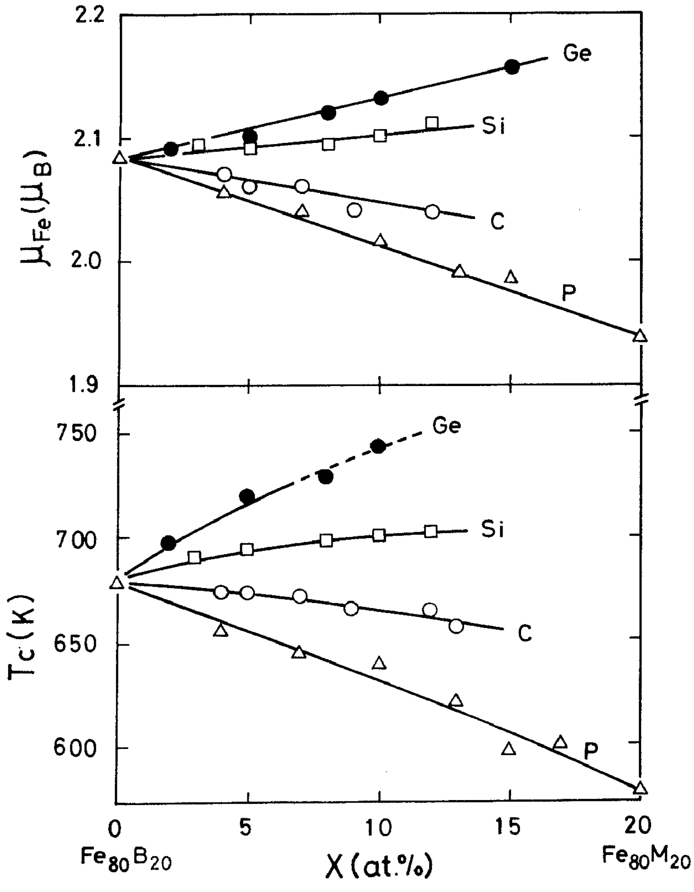

Fig. 12 Magnetic moment per Fe atom (μ) and Curie temperature (T_c) for the amorphous alloys $Fe_{80}(B_{1-x}Y_x)_{20}$ where Y is P, C, Si and Ge. (after Mitera et al 30))

hand, the number of the valence electron for C, Si and Ge is same to each other, then the difference between the values of μ (or T_c) in the cases Y = C, Si and Ge seems to be attributed from their different atomic size.

From application point of view, the above experimental result is important to know the best composition of metalloid elements to obtain the highest value of B_s at room temperature. In addition to this, it has been known that the Curie temperature markedly increases as Co is added into the Fe base amorphous alloys. Kato et al 31) have examined the soft magnetic properties in the amorphous alloys of Fe rich Fe-Co-B-Si system. Their result can be summarized in Fig. 13, where the alloy system investigated is $(Fe_{1-x}Co_x)_{80}(Si_{1-y}B_y)_{20}$. As can be seen in the figure, the alloys in the vicinity of $Fe_{65}Co_{15}B_{20}$ exhibit a large B_s as 17000 G. The B_s decreases slightly with increasing Si content. The B-H hysteresis loop for these alloys in the as-quenched state is more or less rectangular type, but the remanence magnetic induction is rather small as shown in Fig. 14 (Curve-1). This insignificant loop can not be improved by a simple annealing (see Curve-2), but the magnetic field cooling results in the pronounced increase in B_r and decrease in H_c (see Curve-3). Providing that the value of B_r / H_c corresponds approximately to the maximum permability because of the good squerness of the hysteresis loop, these values

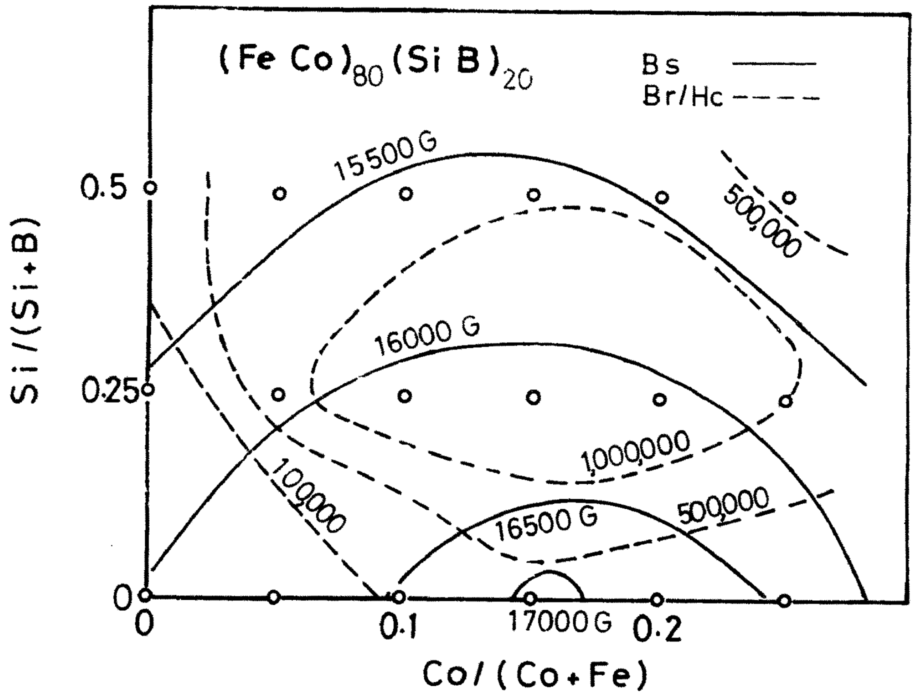

Fig. 13 Saturation magnetic induction at room temperature (B_s) and remanence to coercive force (B_r / H_c) for the amorphous alloys ($Fe_{1-x}Co_x$)$_{80}$($Si_{1-y}B_y$)$_{20}$. The data were obtained after the field cooling treatment. (after Kato et al [31])

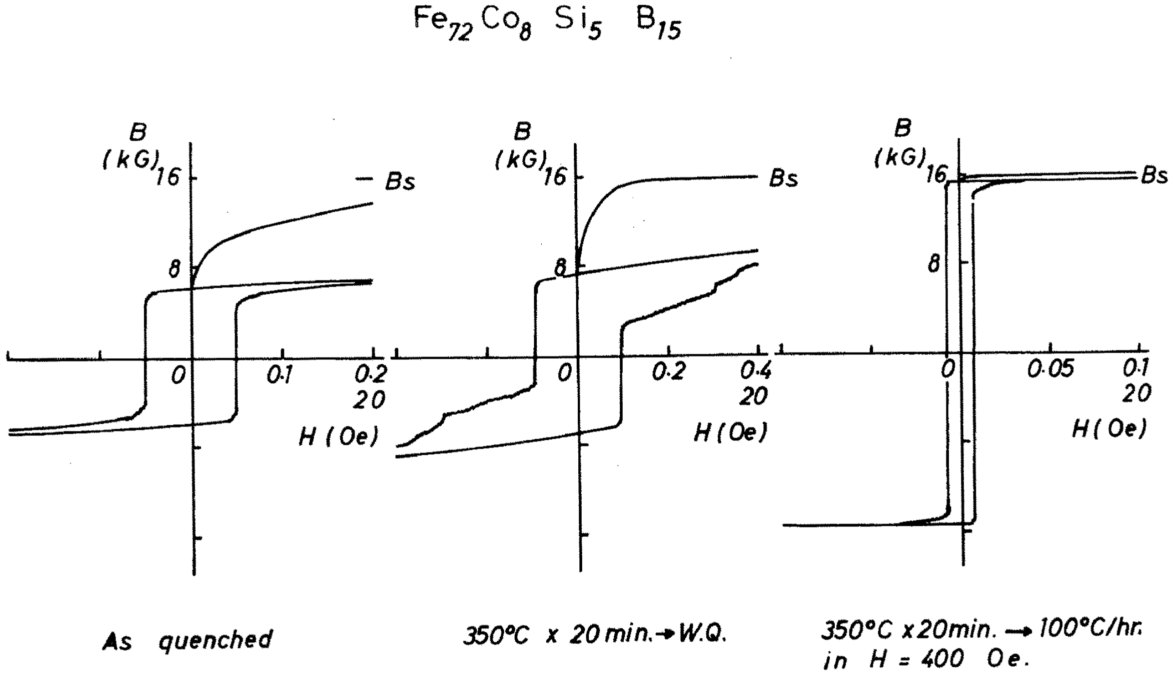

Fig. 14 As an typical example for B-H hysteresis loop of high B_s amorphous alloys, the loops of the amorphous alloy $Fe_{72}Co_8Si_5B_{15}$ are shown.

Curve-1: as-received state
Curve-2: annealed state
Curve-3: magnetic field cooled state (after Kato et al [31])

are also plotted in Fig. 13. There can be seen a wide compositional region with the maximum value of B_r / H_c of about 10^6. Although this region is slightly deviated from the compositional region at where the alloys have the maximum value of B_s, the value of B_s in this region is about 16000 G, promising that these alloys may be useful as power device materials. These alloys have the low H_c of about 10 mOe and high squareness factor of about 95%, therefore they can be magnetized up to 15000 G by applying a field of 10 - 100 mOe. The tendency that the soft magnetic properties depend on concentration of Si and B have been similarly found in the Fe-Si-B 32) and Fe-Ni-Si-B 33). Fig. 15 shows the compositional dependence of the maximum permeability in Fe-Si-B amorphous alloys, wherein it can be seen that the highest value is obtainable in the vicinity of $Fe_{78}Si_{10}B_{12}$ rather than Fe-B binary alloys. Fig. 16 shows the magnetic properties for $(Fe_{.8}Ni_{.2})_{78}Si_yB_{22-y}$. In the as-quenched state H_c, Br and μ_m are only slightly dependent on the value of Si content, while in the annealed state H_c shows a more pronounced minimum, resulting in a large peak of μ_m around y = 0.08. It is of particular interest that the slight change of Si content is remarkably reflected on the improved magnetic properties, but the mechanism is still unsolved.

Table 2 lists up the data on the high B_s amorphous alloys. The magnetic properties for these alloys can be characterrized by the high rectangularity, low coercive force and high maximum permeability.

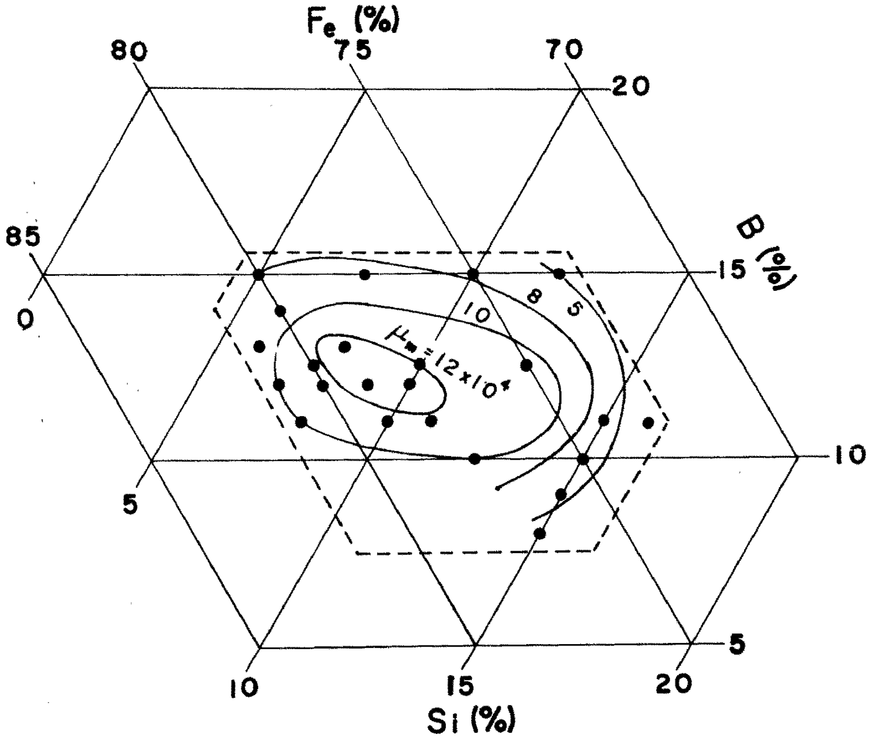

Fig. 15 Maximum permeability (μ_m) for the amorphous alloys Fe-Si-B. (after Mitera et al 32))

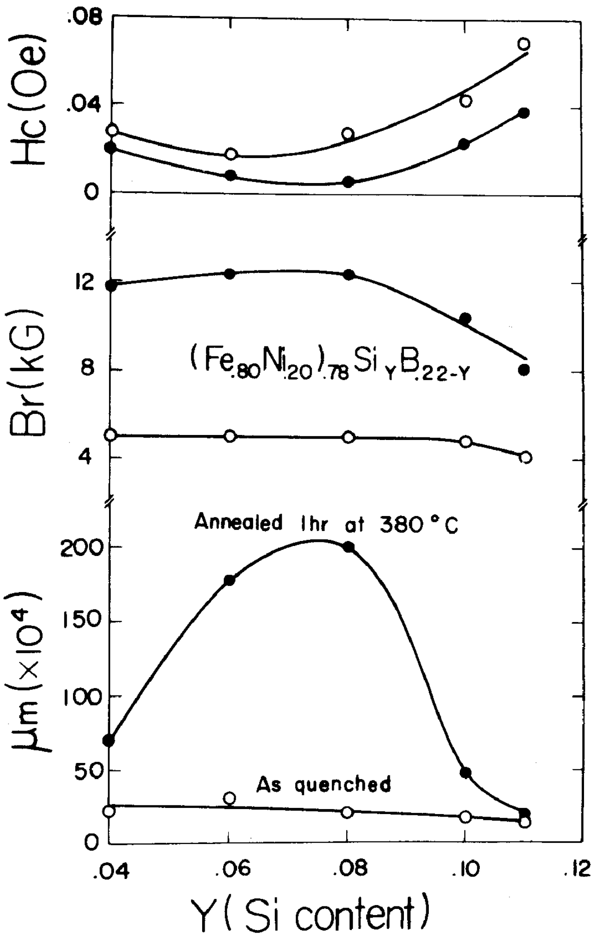

Fig. 16 Coercive force (H_c), remanence magnetic induction (B_r) and maximum permeability (μ_m) for the amorphous alloys $(Fe_{.2}Ni_{.2})_{78}Si_yB_{22-y}$. Closed circle represents the as-recieved state and open circle represents the annealed state, respectively. (after Masumoto et al [33])

Table 2 Amorphous magnetic alloys with high B_s

	Amorphous alloys			Commercial alloys	
	$Fe_{72}Co_8Si_5B_{15}$	$Fe_{80}B_{20}$*	$Fe_{62.5}Ni_{15.5}Si_8B_{14}$	Oriented Si-Fe	50%Ni-Fe
Curie Temp. (°C)	470	374	460	740	500
Satu. Magnetic Induction, B_s (G)	16000	16000	13000	20000	16000
Squareness, B_r/B_s	0.96	0.77	0.93		
Max. Permeability μm (B_r/Hc)	2200000	320000	2000000	40000	20000
Coercive Force H_c (Oe)	0.007	0.04	0.006	0.1	0.05
Resistivity ρo (μΩ·cm)	130	145	125	47	45

* from O'handley et al.

CONCLUDING REMARKS

From the experimental study on the soft magnetic properties for various amorphous alloys, we have found that non magnetostrictive alloys of Co and/or Ni rich Fe-Co-Ni-Si-B system possess a high initial or effective permeability, which are considered as Permalloy-type materials. We have further found that Fe-rich alloys possess a high saturation magnetic induction and the B-H hysteresis loops are characterized by high rectangularity with low coercive force and high maximum permeability, which are considered as materials useful for power devices. However, for practical uses there are still remained many unsolved problems. Particularly, improvements on stability of magnetic properties against aging and mass productions with low cost are strictly required.

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