Masumoto et al.

[45] July 3, 1973

[54]	-	FOR MAGNETIC ING-REPRODUCING HEADS
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[22]	Filed:	June 17, 1971
[21]	Appl. No.:	153,974
[30]	Foreign	Application Priority Data
	June 25, 19	70 Japan 45/54759
[52]	U.S. Cl	148/31.55, 148/31.57, 148/100, 148/101, 148/121, 75/170
		C04b 75/00, H01f 1/04, C22c 19/00
[58]		arch

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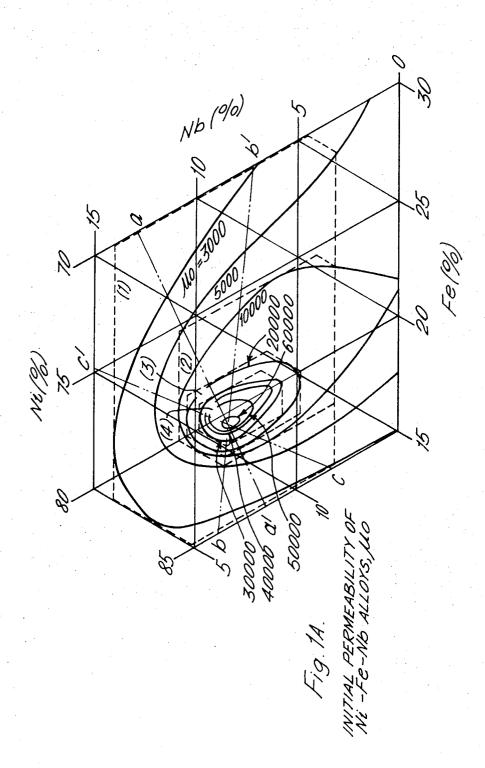
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[57] ABSTRACT

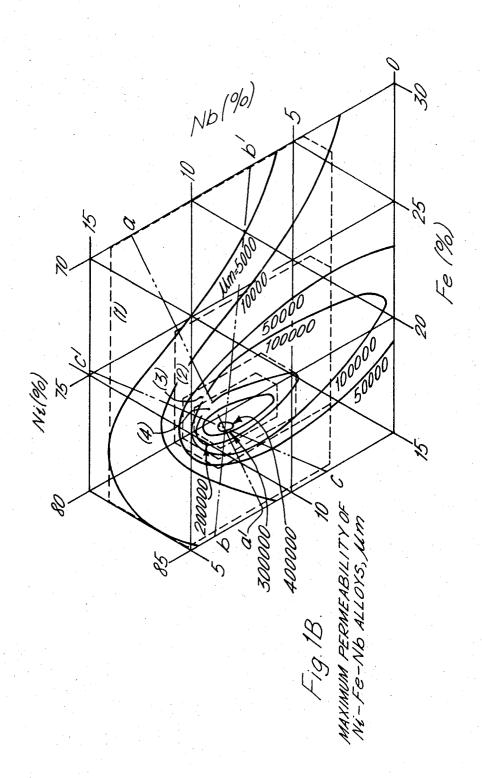
A ferromagnetic alloy of magnetic recording-reproducing heads, essentially consisting of 70.0 to 84.8 Wt.% of nickel, 5.0 to 25.5 Wt.% of iron, and 3.1 to 14.0 Wt.% of niobium, and having Vickers hardness of above 150 and a high initial permeability and a maximum permeability of above 3,000 and 5,000, respectively, and a degree of order greater than 0.1 but less than 0.6.

6 Claims, 11 Drawing Figures

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SHEET 2 OF 9



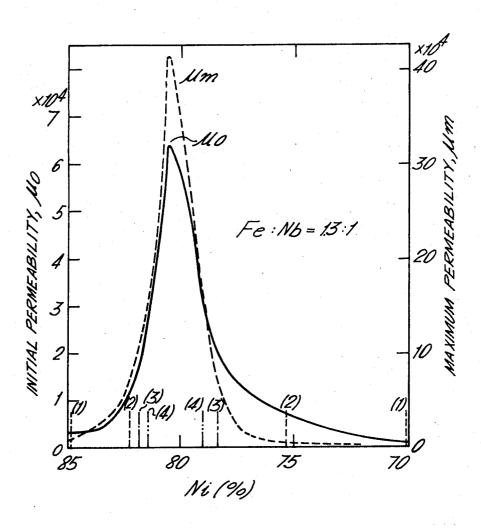


Fig. 2A.
a-al SECTIONS OF FIGS. 1A, 1B.

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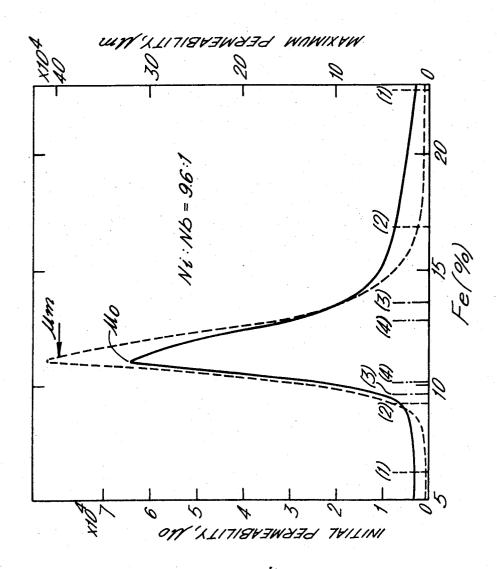


Fig. 2B. 6-6' SECTIONS OF FIGS. 14, 18.

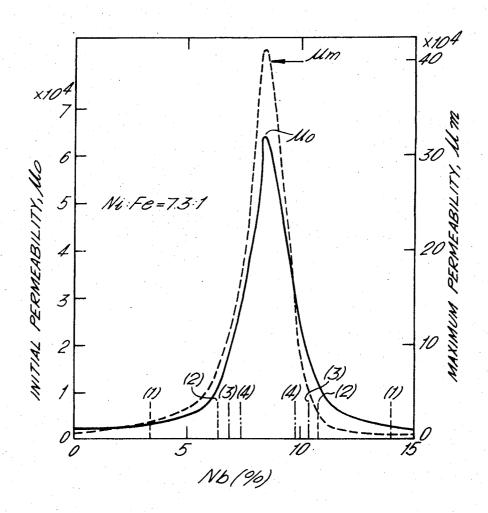


Fig. 2C.
C-C' SECTIONS OF FIGS. 1A, 1B.

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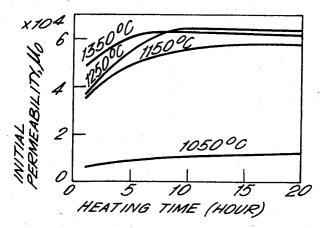


Fig. 3A. INITIAL PERMEABILITY VS. HEATING TEMPERATURE AND TIME.

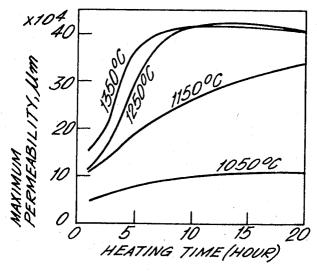
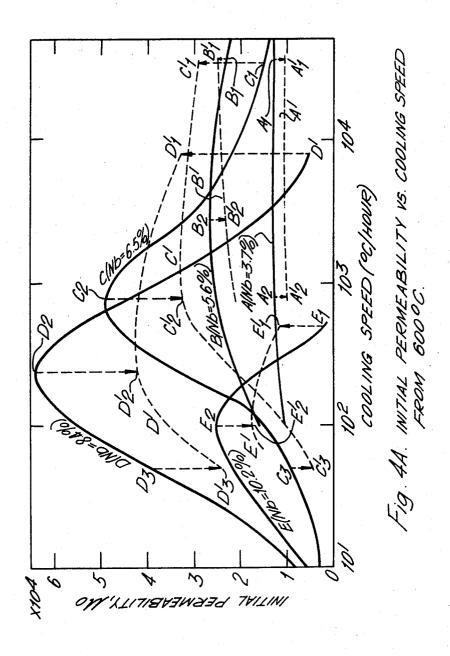
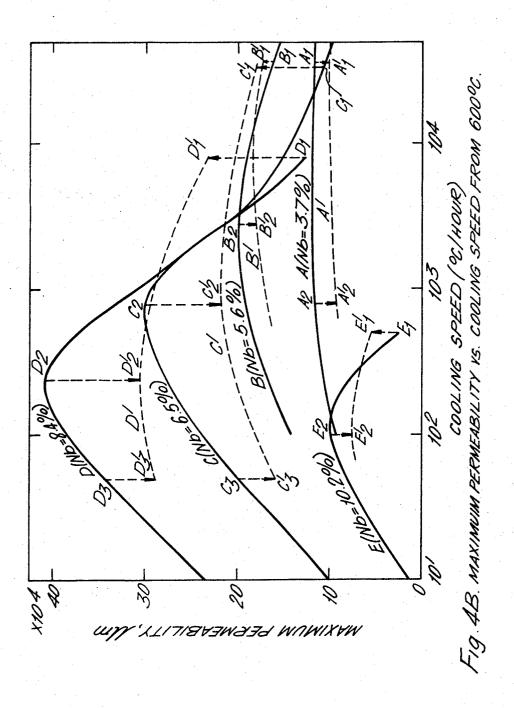


Fig. 3B. MAXIMUM PERMEABILITY
VS. HEATING TEMPERATURE
AND TIME.

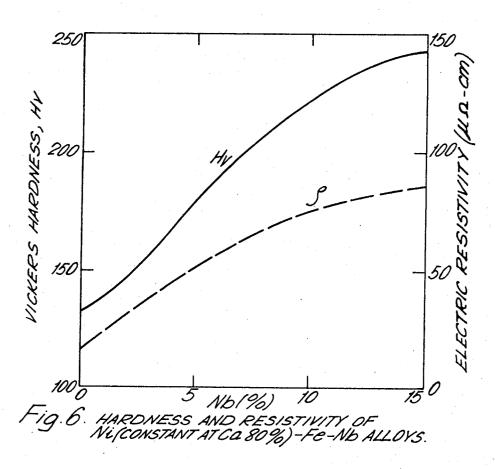
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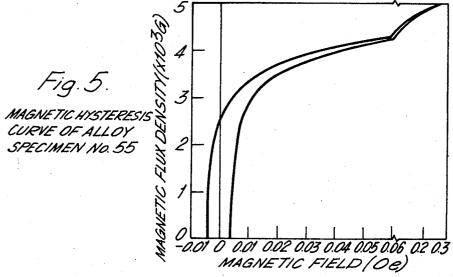


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ALLOYS FOR MAGNETIC RECORDING-REPRODUCING HEADS

BACKGROUND OF THE INVENTION

This invention relates to a high-permeability alloy for 5 magnetic recording-reproducing heads, which alloy consists of 70.0 to 84.8 Wt.% of nickel, 5.0 to 25.5 Wt.% of iron, 3.1 to 14.0 Wt.% of niobium, and an inevitable amount of impurities. With the alloy of the present invention, a high permeability, a high hardness, 10 and a high electric resistivity can be obtained through simple heat treatment, and the alloy can easily be formed into magnetic recording-reproducing heads.

Permalloy (nickel-iron alloy) is widely used at the present in magnetic recording-reproducing heads of 15 audio tape recorder, because it has a high workability. The conventional Permalloy, however, has a shortcoming in that its Vickers hardness Hv is in the order of about 130 and comparatively low, so that its abrasion a pressing need for improving the hardness and abrasion resistivity of alloy materials for magnetic recording-reproducing heads.

SUMMARY OF THE INVENTION

Therefore, an object of the present invention is to meet the aforesaid need by providing an alloy having excellent hardness and abrasion resistivity, along with a high permeability.

To achieve the object of the invention, the applicants 30 have carried out a series of tests on alloys, which have a permeability higher than that of binary Permalloy and high hardness and electric resistivity, while maintaining a high workability. As a result, the applicants have found out that, with the addition of 3.1 to 14.0 Wt.% 35 of niobium into nickel-iron alloys, magnetic and mechanical properties of the alloy can noticeably be improved.

According to present invention, there is provided an alloy consisting of 70.0 to 84.8 Wt.% of nickel, 5.0 to 25.5 Wt.% of iron, 3.1 to 14.0 Wt.% of niobium, and an inevitable amount of impurities, which alloy has a high initial permeability, e.g., 3,000 or higher, a high maximum permeability, e.g., 5,000 or higher, a Vickers hardness greater than 150, and a high electric resistivity. The alloy of the invention can easily be heat treated and formed into the shape of magnetic heads, recording or reproducing. The heat treatment, according to the present invention for providing the desired high permeability and high hardness, comprises steps of heating the alloy in vacuo or in a non-oxidizing atmosphere, for the purpose of thorough solution treatment and homogenization, at 800°C or higher, preferably 1,100°C or higher, for at least 1 minute but not longer than about 100 hours depending on the alloy composition; cooling the alloy to a temperature above its orderdisorder lattice transformation point, e.g., at about 600°C, so as to keep the alloy at the last mentioned temperature for a short while until uniform temperature is established throughout the alloy; and cooling the alloy from the temperature above the order-disorder lattice transformation point to room temperature at a rate faster than 1°C/hour but slower 100°C/second, depending on the alloy composition.

With the present invention, it is also possible to produce the desired permeability and the high hardness by a process comprising steps of heating the alloy of the

aforesaid composition in vacuo or in a non-oxidizing atmosphere, for the purpose of thorough solution treatment and homogenization, at 800°C or higher, preferably 1,100°C or higher, for at least 1 minute but not longer than about 100 hours depending on the alloy composition; cooling the alloy to a temperature above its order-disorder lattice transformation point, e.g., at about 600°C, so as to keep the alloy at the last mentioned temperature for a short while until uniform temperature is established throughout the alloy; cooling the alloy from the temperature above the orderdisorder lattice transformation point to room temperature at a rate faster than 1°C/hour but slower than 100°C/second, depending on the alloy composition; reheating the alloy at a temperature below the orderdisorder lattice transformation point for at least 1 minute but not longer than 100 hours depending on the alloy composition; and cooling it to room temperature.

The aforesaid solution treatment should preferably resistivity is rather poor. Accordingly, there has been 20 be effected at a temperature above 1,100°C, especially about 1,250°C, instead of at a temperature of 800°to 1,100°C, for an extended period of time, so as to effect the solid solution treatment as throroughly as possible. The thorough solid solution treatment results in an outstanding improvement of the magnetic properties of the alloy.

> The manner in which the alloy is cooled from the solution treatment temperature to a temperature above its order-disorder lattice transformation point, e.g., to about 600°C, does not affect its magnetic properties so seriously, regardless of whether it is cooled quickly or slowly. The cooling speed when the alloy temperature crosses its order-disorder lattic transformation point has profound effects on the magnetic properties of the alloy, and hence, it is necessary to cool the alloy from the order-disorder lattice transformation point at a rate faster than 1°C/hour but slower than 100°C/second. Such range of the cooling speed is selected in order to cause the degree of order of the alloy to fall in a range of 0.1 to 0.6, preferably 0.2 to 0.5. If the alloy is comparatively quickly cooled at about 100°C/second, its degree of order becomes comparatively small, e.g., at about 0.1. Quick cooling faster than 100°C/second results in a degree of order small than 0.1 and does not provide the desired permeability.

On the other hand, excessively slow cooling, slower than 1°C/hour, tends to make the degree of order too large in excess of 0.6, so that the desired high permeability cannot be achieved.

The inventors have found that the permeability of the alloy of the invention can be maximized when the degree of order of the alloy falls in a range of 0.1 to 0.6. The aforesaid cooling from a temperature above the order-disorder lattice transformation point of the alloy at a rate faster than 1°C/hour but slower than 100°C/second will results in the desired degree of order in the range of 0.1 to 0.6. The permeability of the alloy thus treated, especially when it is quickly cooled, may be further improved by tempering or reheating it to a temperature below its order-disorder lattice transformation point, e.g., in a range between 200° and 600°C.

In short, with the present invention, the permeability of the alloy of the aforesaid composition is maximized by making its degree of order be 0.1 to 0.6 by applying thorough solution treatment at 800°C or higher, preferably 1,100° to 1,250°C, followed by cooling at a proper rate in the aforesaid range. When quick cooling fails to provide the high permeability, the additional tempering, preferably in the range of 200° to 600°C, will improve its degree of order for raising its permeability.

Generally, a higher treating temperature tends to allow a shorter treating time, while a lower treating 5 temperature tends to require a longer treating time. Similarly, a greater mass tends to require a longer treating time, while a smaller mass tends to allow a shorter treating time.

In cooling the alloy having the aforesaid composition, 10 ent invention will now be described step by step. according to the present invention, from a temperature above its order-disorder lattice transformation point to a low temperature, e.g., to room temperature, the proper cooling speed for maximizing its permeability somewhat varies depending on its composition, but the cooling speed to be used in the method of the present invention usually so slow that cooling in a furnace is preferred. With conventional nickel-iron alloys containing no niobium, e.g., Permalloy, high permeability cannot be obtained unless it is quickly cooled, for instance by forced-air-cooling. The difference of the cooling speed between the conventional alloys and the alloy of the present invention is a very important factor in improving the properties of magnetic material.

For instance, after shaping magnetic recordingreproducing heads, such heads are usually heat treated for eliminating internal stress caused in the heads by the shaping process. To retain their proper shape and to avoid the oxidation of their surface, slow cooling in 30 a vacuo or in a non-oxidizing atmosphere is preferable. The conventional alloys requiring quick cooling for producing a high permeability is not suitable for such slow cooling. On the other hand, the alloy according to the present invention is particularly suitable for such 35 post-shaping heat treatment.

BRIEF DESCRIPTION OF THE DRAWINGS

For a better understanding of the invention, reference is made to the accompanying drawings, in which: 40

FIG. 1A is a graph illustrating the relation between the composition of nickel-iron-niobium alloys and their initial permeability;

FIG. 1B is a graph illustrating the relation between the composition of nickel-iron-niobium alloys and their 45 maximum permeability;

FIG. 2A is a graph representing sections along the lines a-a' of FIGS. 1A and 1B (Fe:Nb = 1.3:1);

FIG. 2B is a graph representing sections along the lines b-b' of FIGS. 1A and 1B (Ni:Nb = 9.6:1);

FIG. 2C is a graph representing sections along the lines c-c' of FIGS. 1A and 1B (Ni:Fe = 7.3:1);

FIG. 3A is a graph showing the relation between the initial permeability of Specimen No. 55 of the alloy according to the present invention and their heating tem- 55perature and heating time;

FIG. 3B is a graph showing the relation between the maximum permeability of Specimen No. 55 of the alloy according to the present invention and their heating 60 temperature and heating time;

FIGS. 4A and 4B are graphs showing the effects of different cooling speeds on the initial permeability and the maximum permeability of the alloys of the present invention, respectively;

FIG. 5 is a magnetic hysteresis curve of Specimen No. 55 of the alloy according to the present invention;

FIG. 6 is a graph representing the effects of different niobium contents in the alloy according to the present invention on their electric resistivity and Vickers hardness, assuming a constant nickel content of about 80 Wt.%.

DESCRIPTION OF THE PREFERRED **EMBODIMENT**

A method for making an alloy according to the pres-

In order to make the alloy of the present invention. a suitable amount of a starting material consisting of 7.0 to 84.8 Wt.% of nickel, 5.0 to 25.5 Wt.% of iron, and 3.1 to 14.0 Wt.% of niobium (instead of metallic niobium, ferro-niobium available in market may also be used) is melted by a melting furnace in air, preferably in vacuo or in a non-oxidizing atmosphere; a small amount (less than 1 Wt.%) of a de-oxidizer and a desulfurizer, e.g., manganese, silicon, aluminum, tita-20 nium, calcium alloy, and the like, is added in the melt for removing impurities as far as possible; and the molten metal thus prepared is thoroughly agitated to homogenize its composition.

For the purpose of testing, a number of different 25 alloy specimens were prepared in the aforesaid manner. Each of the alloy specimens was poured into a mold for producing an ingot. The ingot was then shaped into sheets, each being 0.3 mm thick. The alloys can be shaped into any other suitable form by forging or rolling at room temperature or at an elevated temperature.

Rings with an outer diameter of 35 mm and an inner diameter of 27 mm were punched out of the sheets thus prepared. The rings were then heated at 800°C or higher, preferably at 1,100°C or higher, for at least 1 minute but not longer than 100 hours, in vacuo or in hydrogen or other non-oxidizing atmosphere, and then cooled at a cooling speed suitable for the composition of each alloy specimen in the range of 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second. For certain alloy compositions, the specimens were further heated at a temperature below their order-disorder lattice transformation point, e.g., at about 600°C, for at least 1 minute but not longer than 100 hours, and then

The permeability of the ring specimens thus heat treated was measured by a conventional ballistic galvanometer. The highest values of the initial permeability (μ_o) and the maximum permeability (μ_m) of the specimens proved to be 64,000 and 409,600, respectively. It was also found that the specimens had a considerably high hardness and a large electric resistivity.

FIG. 1A shows contours of the highest values of the initial permeability μ_o of the nickel-iron-niobium alloys of different compositions which were obtained by the aforesaid various heat treatments. Similar contours for the highest values of the maximum permeability μ_m of the nickel-iron-niobium alloys of different compositions are shown in FIG. 1B after applying the aforesaid variety of heat treatments.

FIGS. 2A, 2B, and 2C are schematic sections of FIGS. 1A and 1B, taken along the lines a-2', b-b' and c-c', respectively, illustrating the highest values of the $_{65}$ initial permeability μ_o and the maximum permeability μ_m along such sections.

Table 1 shows the physical properties of selected alloy specimens. (The details of the process for making the alloy specimens of Table 1 will be described hereinafter).

ture. Thus, there are an optimal heating temperature and an optimal heating time for each alloy composi-

TABLE 1

				Heat trea	tment.	Cooling speed after heating		Maxi-	Residual magnetic flux density	Coercive force	Hystere- sis loss (erg per cm.3 per	Saturated flux density (G)		
		nposi				at 600° C.	Initial	mum	(G)	(oe.)	cycle)	At mag-	Electric	Vickers
Speci-	()	percen	t)	Temper-	Dura-	for 10	permea-	permea-				netic	resis-	Hard-
men	NT:	T7	NTI.	ature	tion	minutes	bility	bility	At maxir	num flux de	nsity of	field of	tivity	ness
No.	Ni	Fe	Nb	(° C.)	(hour)	(° C./hour)	(μ ₀)	(μ _m)		5,000 G		900 oc.	(μΩ-cm.)	(Hv)
10	80, 1	16, 2	3.7	1,100	3	8,100	12,700	116,000				9,010	42, 0	165
16		15, 6	4, 2	1,150	3	2,800	16, 250	58, 800	4, 110	0, 0250	42, 10	8,750	45.3	170
16	80.2	15.6	4.2	1,250	3	400	10, 430	121,700	4, 260	0.0213	37.75	· · · · · · · · · · · · · · ·		
25	80, 5	13.9	5, 6	1, 250	9	2,800	26, 500	198, 200				7, 930	54.6	186
27		14, 5	5, 6	1,350	3	400	21,150	249,400	3, 630	0.0080	16.30			
34		13.1	6, 5	1,250	18	100	15,080	345,000				7, 580	59, 7	
35	80, 0	13.5	6, 5	1, 150	3	240	3 4,800	129,000	3, 300	0, 0160	13.60	7,600	60, 2	193
35	80.0	13, 5	6, 5	1, 250	9	800	49, 300	302,800	3,500	0.0098	14, 46			190
35	80, 0	13.5	6, 5	1,350	3	400	29,000	231,600	3, 230	0.0082	13, 10	7,550	60, 3	190
45	80, 5	12.0	7.5	1, 3 50	3	400	49,300	151,000	2,600	0.0093	14. 47	6,980	65.1	
	79.4	13. 1	7.5	1,250	3	240	40,600	86, 300	3, 360	0.0105	16, 42	7,050	65, 5	203
47	79.4	13. 1	7, 5	1,250	_9	800	43,500	191,500						
47	79, 4	13.1	7.5	1, 250	18	800	34,800	254 , 3 00	3, 120	0.0088	14, 25			
52	80, 6	11.4	8.0	1, 250	18	400	33 , 000	354,500	2,860	0, 0084	13.61	6, 730	67.1	20
52	80, 6	11.4	8.0	1, 250	18	¹ 8, 100	21,900	260, 700						
55	80.5	11.1	8.4	1,150	3	240	44,500	143,000	2,850	0.0114	16,95	6, 530	69, 0	
55	80.5	11.1	8.4	1,250	9	240	64,000	409, 600	2,550	0,0038	5, 09	6, 550	69. 3	210
55	80.5	11.1	8.4	1,250	9	2 240	42,000	306, 400				6, 520	69.0.	
55	80.5	11.1	8.4	1,250	9	1 8, 100	33, 500	237, 700	2,640	0.0054	9, 63			
55	80.5	11.1	8.4	1,350	. 3	400	55, 100	221,800	2,080	0.0036	7. 47	6, 530	6 9, 4	208
55	80.5	11.1	8.4	1,350	.3	1 400	46, 300	207,000	2,440	0.0046	8. 25			
56 65	79.5 80.1	12. 1 10. 6	8.4	1,150	-3	240	43,500	212,000	2,640	0.0077	15. 10	6,550	69.3	
			9.3	1,250	18	240	43, 520	204, 000	2,580	0.0096	16. 73	6,030	73.4	
66	80.1 79.7	10.6 11.0	9.3 9.3	1,250	18	1 2,800	32,500	187, 200	2 000	0.0010	10.67			217
66	79.7	11.0	9.3	1,250 $1,250$	9	$\frac{100}{240}$	53, 650 34, 800	231,500	3, 280	0.0048	10.64	6,050	73.7	
70	79. 7	10.1	9. 3	1,250 $1,250$	9	100	25, 140	304, 500	3, 350 1, 070	0.0050	. 9. 78	5 070		
70 75	80. 0	7.8	12.2	1,250	9.			98,000 8,600	1,070	0.0108	11.57	5,370	75, 3	999
78	79.0	7.0	14.0	1,250 $1,250$	3	50 10	4,500 3,010	5,000				3,950	80.5	233 240
10	10.0	1.0	14.0	1, 200	o	10	9,010	ο , 000 .				2, 400	84.0	240

¹ And heated at 400° C. for 1 hour.

It is apparent from FIGS. 1A to 2C that the addition of 3.1 to 14.0 Wt.% of niobium into binary nickel-iron alloys greatly improves the magnetic properties of the alloys, and the heating of such ternary alloys at a temperature higher than 1,100°C further improves the permeability of the ternary alloys. Thus, with the alloys of the present invention, extremely high initial permeability and maximum permeability can easily be obtained. For instance, alloy Specimen No. 55 consisting of 80.5 Wt.% of nickel, 11.1 Wt.% of iron, and 8.4 Wt.% of niobium showed an initial permeability of 64,000 and a maximum permeability of 409,600, when it was heated at 1,250°C for 9 hours and cooled in a furance to 600°C for keeping it at 600°C for 10 minutes and then cooled to room temperature at a cooling speed of 240°C/hour. Such values of permeability are considerably larger than those obtainable by using conventional alloys; namely, a conventional nickel-iron alloy consisting of 78.5 Wt.% of nickel and 21.5 Wt.% of iron shows an initial permeability of 8,000 and a maximum permeability of 100,000, when it is heated at 1,050°1.050°C and slowly cooled to 600°C followed by quick cooling from 600°C.

FIG. 3A shows the effects of different high heating temperatures and the heating time at such temperatures on the initial permeability of the ternary alloy, for the case of Specimen No. 55 of Table 1. FIG. 3B shows similar effects on the maximum permeability of the same Specimen. The values of the permeability in FIGS. 3A and 3B were determined after cooling Specimen no. 55 from the illustrated high temperature in the range of 1,050° to 1,350°C in a special manner; namely, it was cooled to 600°C in a furnace for keeping it at 600°C for 10 minutes and then cooled to room temperature at a speed of 240°C/hour. It is apparent from FIGS. 3A and 3B that the permeability is materially influenced by the high heating temperature and the duration in which the alloy is heated at such high tempera-

tion, in order to maximize the permeability. More particularly, a heat treatment at a temperature below 1,100°C results in comparatively low permeabilities; namely, an initial permeability not greater than 12,000 and a maximum permeability not greater than 110,000. On the other hand, a high temperature heat treatment at 1,100°C or higher results in comparatively high permeabilities; namely, an initial permeability greater than 12,000 and a maximum permeability greater than 110,000.

In order to test the effects of the cooling speed and reheating after the cooling from the aforesaid high temperature, a series of tests were carried out. The results are shown in FIGS. 4A and 4B. in the figures, curves A to E correspond to selected Specimens of Table 1, as shown in Table 2.

TABLE 2

50	Curve	Specimen No. in	Composition (Wt.%)						
		Table 1	Nickel	Iron	Niobium				
	Α	10	80.1	16.2	3.7				
	В	25	80.5	13.9	5.6				
	C	35	80.0	13.5	6.5				
	D	55	80.5	11.1	8.4				
55	E	70	79.7	10.1	10.2				

In the figures, non-primed symbols in FIGS. 4A and 4B (A₁, A₂, B₂, C₁, C₂, C₃, D₁, D₂, D₃, E₁, and E₂) represent the permeability of the corresponding alloys, which were treated by heating at 1,250°C for 9 hours, cooling to 600°C in a furnace, and then further cooled from 600°C to room temperature at different speeds as specified by such non-primed symbols in the figures. The curves A to E were drawn by connecting such non-primed points for the corresponding alloys.

Primed points in FIGS. 4A and 4B (A₁', A₂'B₁', B₂', C₁', C₂', C₃', D₁', D₂', D₃', E₁', and E₂') represent the permeability of the corresponding alloy Specimens

² And heated at 400° C. for 30 minutes.

after further treating them from the corresponding non-primed conditions, respectively. The heat treatments for the primed points were as follows.

A₁': After A₁, Specimen No. 10 was reheated at 350°C for 1 hour and cooled in air.

A₂': After A₂, Specimen No. 10 was reheated at 350°C for 1 hour and cooled in air.

B₁': After B₁, Specimen No. 25 was reheated at 400°C for 30 minutes and cooled in air.

B₂': After B₂, Specimen No. 25 was reheated at 10 400°C for 30 minutes and cooled in air.

C₁': After C₁, Specimen No. 35 was reheated at 350°C for 1 hour and cooled in air.

C₂': After C₂, Specimen No. 35 was reheated at 350°C for 30 minutes and cooled in air.

C₃': After C₃, Specimen No. 35 was reheated at 400°C for 1 hour and cooled in air.

d₁': After D₁, Specimen No. 55 was reheated at 400°C for 1 hour and cooled in air.

D₂': After D₂, Specimen No. 55 was reheated at 20 400°C for 30 minutes and cooled in air.

 D_3' : After D_3 , Specimen No. 55 was reheated at 400° C for 1 hour and cooled in air.

E₁': After E₁, Specimen No. 70 was reheated at 400°C for 2 hours and cooled air.

E₂': After E₂, Specimen No. 70 was reheated at 400°C for 2 hours and cooled in air.

The following trends are noticed in FIGS. 4A and 4B. For alloys with 3.7 Wt.% of niobium, quick cooling is necessary in order to obtain a high permeability, initial or maximum, and reheating, e.g., at 350°C for 1 hour, tends to reduce the permeability. As the niobium content increases, e.g., toward 5.6 to 14.0 Wt.%, high permeabilities can more frequently be obtained by slower cooling. In general, if the alloys are comparatively quickly cooled from 600°C the succeeding reheating tends to noticeably increase their permeability, while if the alloys are comparatively slowly cooled from 600°C the succeeding reheating tends to jeopardize their permeability. The aforesaid trends are noticed both in the initial permeability and the maximum permeability.

FIG. 5 illustrates the hysteresis curve for the Specimen having the highest permeability, i.e., Specimen No. 55. It is apparent from the figure that the hysteresis 45 loss of Specimen No. 55 is extremely small.

The present invention will not be described in further detail by referring to specific Examples. (Detailed explanation will be made in the following Examples on Specimens selected from those listed in Table 1, and similar treatment may be applied on non-selected Specimens for obtaining similar effects.)

EXAMPLE 1

Alloy Specimen No. 35 consisting of 80.0 Wt.% of nickel, 13.5 Wt.% of iron, and 6.5 Wt.% of niobium, as listed in Table 1, was made by using 99.8%-pure electrolytic nickel, 99.97%-pure electrolytic iron, and 99.8%-pure niobium. An ingot of the Specimen was

formed by melting 800 grams of the starting pure metals in vacuo by using a crucible disposed in a high-frequency electric induction furnace, agitating the molten metal so as to produce a homogeneous melt of the alloy, and pouring the melt into a metallic mold having a cylindrical hole of 25 mm diameter and 170 mm height. The ingot was hot forged at about 1,000°C into 7 mm thick sheets. The sheets were hot rolled at about 600° to 900°C to a thickness of 1 mm, and then cold rolled at room temperature to make them into thin sheets of 0.3 mm thickness. Rings with an inner diameter of 27 mm and an outer diameter of 35 mm were punched out from the thin sheets.

The rings thus formed were subjected to different heat treatments, as shown in Table 3. Physical properties of the rings after the treatments are also shown in Table 3.

EXAMPLE 2

Alloy Specimen No. 55, consisting of 80.5 Wt.% of nickel, 11.1 Wt.% of iron, and 8.4 Wt.% of niobium, was made by using the same materials in a similar manner as Example 1, so as to make similar rings. Different heat treatments were applied to the rings of Specimen No. 55, as shown in Table 4, together with the physical properties of the rings thus treated.

EXAMPLE 3

Alloy Specimen No. 70, consisting of 79.7 Wt.% of nickel, 10.1 Wt.% of iron, and 10.2 Wt.% of niobium, was made by using the same material in a similar manner as Example 1, so as to make similar rings. Different heat treatments were applied to the rings of Specimen No. 70, as shown in Table 5, together with the physical properties of the rings thus treated.

In examples 1 to 3, 99.8%-pure metallic niobium was used as the starting material, but instead of such metallic niobium, ferro niobium available in market can also be used as the starting material of the alloys according to the present invention. Since the use of ferro niobium tends to make the alloy increasingly brittle, it is preferable to add a suitable deoxidizer and/or a desulfurizer into the melt of the alloy of the invention, so as to improve the ductility by deoxidation and desulfurization.

Thus, with the method according to the present invention, the heat treatment may be completed only by a primary treatment, which consists of heating a ternary alloy with a composition falling in the specific range of the invention, in a non-oxidizing atmosphere or in a vacuo at 800°C or higher, preferably above 1,100°C, for at least 1 minute but not longer than about 100 hours, depending on the alloy composition, cooling the alloy to about 600°C in a furnace, and then cooling the alloy from about 600°C to room temperature at a cooling speed of 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second.

4 According to the present invention, it is also possible 99.8%-pure niobium. An ingot of the Specimen was 60 to apply a secondary heat treatment to the alloy

TABLE 3

		Initial permea-	Maximum permea-	Residual magnetic flux density (G)	Coercive force (oc.)	Hysteresis loss (erg per cm. ³ per cycle)	Saturated flux density (G) At magnetic	Electric resis-	Vickers hard-
Item No.	Heat treatment	bility (μ ₀)	bility				fleld of 900 oe.	tivity (μΩ-em.)	ness (Hv)
Ī	Heated at 1,150° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room tempreature at 240° C./hour.	34, 800	129,000		0, 0160		7,600	60. 2	193
II	 After I, reheated at 350° C. in vacuo for 1 hours. Heated at 1,250° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 10° C./sec. 	29, 500 15, 000	113,000 106,000				7,620	61.0	191
IV V	 After III, reheated at 350° C. in vacuo for 1 hour. Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room temperature at 800° C./hour. 	29, 000 49, 300	173, 000 302, 800	3,380 3,500	0, 0124 0, 0098	15.30 14.46	7,580	60, 5	190
VI	After V, reheated at 350° C., in vacuo for 30 minutes.	32,500	216, 400					·	195
VII	. Heated at 1,250° C. in hydrogen for 9 hours,	16, 400	246, 000	3, 410	0, 0145	18, 66	7,600	60, 0	194
VIII	cooled in furnace to 600° C., and cooled to room temperature at 100° C./hour. After VII, reheated at 350° C. in vacuo for 1 hour. Heated at 1,350° C., in hydrogen for 3 hours cooled in furnace to 600° C., and cooled to room temperature at 400° C./hour.	11, 200 29, 000	178, 000 2 31, 600	3, 230	0.0082	13, 10	7,550	60, 3	195 190
X ·	After IX, reheated at 350° C. in vacuo for 1 hour.	18,500	188, 300						
	·		TABLE 4	! 					
	 Heated at 1,150° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 240° C./hour. 	44,500	143, 000	2,850	0. 0114		6, 530	69.0	210
	After I, reheated at 400° C. in vacuo for 30 minutes.	38,000							
111	Heated at 1,250° C. in hydrogen for 9 hours, coled in furnace to 600° C., and cooled to room temperature at 8,100° C./hour.	5,500	124, 000				6, 470	69, 5	208
	After III, reheated ta 400° C./nour. After III, reheated ta 400° C. in vacuo for 1 hour, Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room temperature at 240° C./nour.	33,500 64,000	237, 700 409, 600	2, 640 2, 550	0. 0054 0. 00 3 8	9. 63 5. 09	6,550	69. 3	210
VI	. After v, reneated at 400° C. in vacuo for 30	42,000	306, 400				6,520	69.0	
VII	minutes. Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room temperature at 50° C./hour. After VII reheated at 400° C. in rease for these	39, 200	346, 500	2,730	0, 0047	7. 53	6, 530	69. 2	212
* * * * * * * * * * * * * * * * * * * *	After VII, reheated at 400° C. in vacuo for 1 hour. Heated at 1,350° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 400° C./hour.	24,000 55,100	287, 400 221, 800	2,080	0.0036	7. 47	6, 530	69. 4	212 208
X	After IX, reheated at 400° C. in vacuo for 1 hour.	46, 300	207,000	2, 440	0, 0046	8. 25			
			TABLE &	i .					
		-	****				Saturated		
				Residual magnetic flux	Coercive	Hysteresis loss (erg per em.3	flux density (G)		
		Initial permea-	Maximum permea-	density (G)	force (oe.)	per eyele)	At mag- netic	Electric resis-	Vickers
Item No.	Heat treatment	bility	bility	At maximum		·	field of 900 oe.	tivity (μΩ-em.)	hard ness (Hv)
	Heated at 1,150° C. in hydrogen for 3 hours, cooled in furnace to 600°C., and cooled to room	11,600	55, 250	1,550	0. 0142	15, 62	5, 250	75. 5	220
III	After I, reheated at 400° C. in vacuo for 1 hour- Heated at 1,250° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room	10, 3 00 20, 3 00	46, 500 38, 100	800	0.0180	16. 25	5, 270	75. 6	223
IV V	After III, reheated at 400° C. in vacuo for 2 hours. Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room	17, 000 2, 000	43, 000 27, 900	1,740	0. 0116	12. 26	5, 310	76. 0	224 222
VI VII	Heated at 1,150° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 100° C./hour. After I, reheated at 400° C. in vacuo for 1 hour Heated at 1,250° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 100° C./hour. After III, reheated at 400° C. in vacuo for 2 hours. Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room temperature at 400° C./hour. After V, reheated at 400° C. in vacuo for 2 hours. Heated at 1,250° C. in hydrogen for 9 hours, cooled in furnace to 600° C., and cooled to room temperature at 100° C./hour. After V, reheated at 400° C. in vacuo for 2 hours. The total furnace to 600° C., and cooled to room temperature at 100° C./hour. After VII, reheated at 400° C. in vacuo for 2 hours.	10, 250 25, 140	56, 100 98, 000	1,070	0.0108	11.57	5, 370	75.3	222
VIII	After VII, reheated at 400° C. in vacuo for 2	17, 500	72, 500				5, 350	74.8	
ıx	hours. Heated at 1,350° C. in hydrogen for 3 hours, cooled in furnace to 600° C., and cooled to room temperature at 100° C./hour. After IX, reheated at 400° C. in vacuo for 2 hours.	22, 400	86,000	1, 100	0.0125	13.36	5, 320	75. 5	220
X	After IX, reheated at 400° C. in vacuo for 2 hours.	18,600	75, 200			· • • • • • • • • • • • • • • • • • • •			

treated by the aforesaid primary heat treatment, which 60 netic properties with conventional nickel-iron binary secondary heat treatment comprises steps of heating the alloy in a non-oxidizing atmosphere or in vacuo at a temperature below the order-disorder lattice transformation point of the alloy, preferably at about 600°C, for at least 1 minute but not longer than about 100 65 hours, and then gradually cooling.

The optimal cooling speed to obtain excellent mag-

alloys is comparatively quick. In the case of nickeliron-niobium ternary alloys, according to the present invention, the optimal cooling speed for obtaining excellent magnetic properties decreases as the niobium content in the alloy increases. With the niobium content of 8.4 Wt.% which gives the highest permeability among all the Specimens, the optimal cooling speed is so slow that it is preferable to cool it in a furnace. It is one of the important features of the present invention that the outstandingly high permeability of the alloy can be produced by a very simple heat treatment.

Conventional materials for magnetic recording and 5 reproducing heads have a shortcoming in that the passage of magnetic tape in contact with such heads tends to abrade the heads, which head abrasion may cause deterioration of the quality of the signals, e.g., sound quality, recorded or reproduced by the head. Accord- 10 ingly, the alloy for magnetic heads should preferably have a high hardness and a high abrasion resistivity. Conventional nickel-iron alloys for magnetic heads have a Vickers hardness in the order of about 130, which is not high enough for ensuring a high abrasion 15 resistivity. On the other hand, the Vickers hardness of the alloy according to the present invention increases with the niobium content, as shown in FIG. 6 and Table 1, and a Vickers hardness as high as 157 to 241 can be obtained by adding 3.1 to 14.0 Wt.% of niobium. The alloy having the highest permeability, which contains 8.4 Wt.% of niobium, shows a Vickers hardness of 210. Thus, the abrasion resistivity of magnetic material for recording and reproducing heads is noticeably improved by the present invention. It is one of the impor- 25 tant features of the present invention that the abrasion resistivity of magnetic recording and reproducing heads is improved by increasing the hardness of the alloy constituting the heads.

The electric resistivity of magnetic recording and reproducing heads should preferably be high, for suppressing the eddy current loss therein. The electric resistivity of conventional binary alloy consisting of about 79 Wt.% of nickel and about 21 Wt.% of iron is in the order of 16 $\mu\Omega$ -cm. On the other hand, with the alloys 35 according to the present invention, the electric resistivity comparatively rapidly increases with the niobium content, as can be seen from FIG. 6 and Table 1. The use of 3.1 to 14.0 Wt.% of niobium in the alloy of the present invention results in an electric resistivity of 38 to 84 $\mu\Omega$ -cm. For the niobium content of 8.4 Wt.%, which provides the highest permeability, the alloy according to the present invention shows an electric resistivity of 70 $\mu\Omega$ -cm. The high electric resistivity of the alloy is also one of the important features of the present 45 invention.

Magnetic heads are usually made by laminating thin sheets of the alloy material, which sheets are in turn formed by rolling and cutting into suitable shape by punching. Thus, the alloy for magnetic heads should have a high workability. The alloys according to the present invention are as easily workable as conventional nickel-iron binary alloys; namely, the alloy of the invention can easily be forged, rolled, drawn, swaged, or punched.

The high hardness of the alloy according to the present invention makes the alloy particularly suitable for magnetic recording and reproducing heads, as pointed out in the foregoing. Furthermore, the outstandingly high permeability and the high electric resistivity of the alloy of the invention are also attractive in conventional electric and magnetic devices of various other types.

The contents of nickel, iron, and niobium are restricted to 70.0 to 84.8 Wt.%, 5.0 to 25.5 Wt.%, and 3.1 to 14.0 Wt.%, respectively, according to the present invention, because the alloy composition in the

aforesaid range produces a high permeability and a high hardness suitable for magnetic heads, but alloy compositions outside the aforesaid range result in too low values of permeability and hardness to use the alloy for magnetic heads.

The suitable contents of the ingredients in the alloy according to the present invention will now be described in further detail.

1. 70.0 to 84.8 Wt.% of nickel:

With the nickel content of 70.0 to 84.8 Wt.%, excellent magnetic properties can be achieved, i.e., an initial permeability μ_o of 64,000 and a maximum permeability μ_m of 409,600. With nickel content less than 70.0 Wt.%, the initial permeability μ_o and the maximum permeability μ_m are reduced to levels below 3,000 and 5,000, respectively. On the other hand, with nickel content in excess of 84.8 Wt.%, a comparatively high maximum permeability μ_m can be obtained but the initial permeability μ_o becomes less than 3,000. Thus, the nickel content is restricted to 70.0 to 84.8 Wt.%.

2. 5.0 to 25.5 Wt.% of iron:

With the iron content of 5.0 to 25.5 Wt.%, excellent magnetic properties can be obtained. On the other hand, with iron content of less than 5.0 Wt.% or in excess of 25.5 Wt.%, the initial permeability μ_o and the maximum permeability μ_m are always below 3,000 and 5,000, respectively. Thus, the iron content is restricted to 5.0 to 25.5 Wt.%.

3. 3.1 to 14.0 Wt.% of niobium:

With the niobium content in the aforesaid range, excellent magnetic properties and hardness can be obtained. On the other hand, with the niobium content of less than 3.1 Wt.%, it becomes difficult to ensure the Vickers hardness Hv to be not smaller than 150. When the niobium content increases in excess of 14.0 Wt.%, the initial permeability μ_o and the maximum permeability μ_m become smaller than 3,000 and 5,000, respectively. The excessively high niobium content also results in the deterioration of the workability of the alloy, especially its forgeability and rollability. Thus, the niobium content is restricted to 3.1 to 14.0 Wt.%.

In short, the alloy according to the present invention consists of 70.0 to 84.8 Wt.% of nickel, 5.0 to 25.5 Wt.% iron, 3.1 to 14.0 Wt.% of niobium (instead of metallic niobium, ferro niobium in market may also be used), and an inevitable amount of impurities. An ingot of the alloy of the invention may be made by pouring a melt of the alloy into a suitable mold. The ingot may be shaped into a desired form by working it at room temperature or at an elevated temperature, for instance by forging, rolling, drawing, swaging, or the like.

After the shaping, the alloy is heat treated by heating it at 800°C or higher (preferably higher than 1,100°C) in a non-oxidizing atmosphere, e.g., hydrogen, or in vacuo for at least 1 minute but not longer than about 100 hours, and cooled to room temperature at a cooling speed of from 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second, depending on the alloy composition. For certain alloy compositions, the alloy may be reheated to a temperature below its orderdisorder lattice transformation point, e.g., below about 600°C, for at least 1 minute but not longer than about 100 hours. With such heat treatment, high permeability 65 including an initial permeability μ_0 of 64,000 and a maximum permeability of 409,600 can be obtained. In addition to the high permeability, the alloy according to the present invention has a number of properties

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suitable for magnetic recording and reproducing heads; namely, a comparatively high electric resistivity, a high hardness, and a high workability at room temperature and at an elevated temperature in terms of forgeability, rollability, drawability, and swageability.

With the alloy of the present invention, extremely high values of initial and maxiumu permeability can be generated by using the following composition and applying any of the following heat treatments to the alloy.

- al. An alloy consisting of 70.0 to 84.8 Wt.% of 10 nickel, 5.0 to 25.5 Wt.% of iron, 3.1 to 14.0 Wt.% of niobium, and an inevitable amount impurities is heated at a temperature above 800°C, preferably above 1,100°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, 15 cooled in a furnace to an intermediary temperature slightly above the order-disorder lattice transformation point of the alloy, for instance to about 600°C, and cooled to room temperature from the intermediary tempterature at a cooling rate in a range of 1°C/hour 20 100°C/second, 10°C/hour preferably 10°C/second. Whereby, one can obtain an initial permeability of about 3,000 to 64,000 and a maximum permeability of about 5,000 to 409,600.
- a2. After the cooling to room temperature, the alloy 25 of the preceding item (a1) may be reheated at a temperature below its order-disorder lattice transformation point, e.g., below about 600°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not meabilities of the item (a1).
- b1. An alloy consisting of 75.3 to 82.2 Wt.% of nickel, 9.2 to 20.6 Wt.% of iron, 3.1 to 10.8 Wt.% of niobium, and an inevitable amount impurities is heated at a temperature above 800°C, preferably above 35 1,100°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, cooled in a furnace to an intermediary temperature slightly above the order-disorder lattice transformation point of the alloy, for instance to about 600°C, and 40 cooled to room temperature from the intermediary temperature at a cooling rate in a range of 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second. Whereby, one can obtain an initial permeability of about 10,000 to 64,000 and a maximum permeability of about 50,000 to 409,600.
- b2. After the cooling to room temperature, the alloy of the preceding item (b1) may be reheated at a temperature below its order-disorder lattice transformation mosphere or in vacuo for at least 1 minute but not longer than about 100 hours, so as to generate the permeabilities of the item (b1).
- c1. An alloy consisting of 78.3 to 81.8 Wt.% of nickel, 09.6 to 15.2 Wt.% of iron, 4.8 to 10.4 Wt.% of 55 niobium, and an inevitable amount impurities is heated at a temperature above 800°C, preferably above 1,100°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, cooled in a furnace to an intermediary temperature slightly above the order-disorder lattice transformation point of the alloy, for instance to about 600°C, and

cooled to room temperature from the intermediary temperature at a cooling rate in a range of 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second. Whereby, one can obtain an initial permeability of about 20,000 to 64,000 and a maximum permeability of about 100,000 to 409,600.

- c2. After the cooling to room temperature, the alloy of the preceding item (c1) may be reheated at a temperature below its order-disorder lattice transformation point, e.g., below about 600°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, so as to generate the permeabilities of the item (c1).
- d1. An alloy consisting of 79.0 to 81.4 Wt.% of nickel, 10.1 to 14.2 Wt.% of iron, 5.7 to 9.8 Wt.% of niobium, and an inevitable amount impurities is heated at a temperature above 800°C, preferably above 1,100°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, cooled in a furnace to an intermediary temperature slightly above the order-disorder lattice transformation point of the alloy, for instance to about 600°C, and cooled to room temperature from the intermediary temperature at a cooling rate in a range of 1°C/hour to 100°C/second, preferably 10°C/hour to 10°C/second. Whereby, one can obtain an initial permeability of about 30,000 to 64,000 and a maximum permeability of about 200,000 to 409,600.
- d2. After the cooling to room temperature, the alloy longer than about 100 hours, so as to generate the per- 30 of the preceding item (d1) may be reheated at a temperature below its order-disorder lattice transformation point, e.g., below about 600°C, in a non-oxidizing atmosphere or in vacuo for at least 1 minute but not longer than about 100 hours, so as to generate the permeabilities of the item (d1).

What is claimed is:

- 1. An alloy for magnetic recording and reproducing heads, consisting essentially of 70.0 to 84.8 Wt. % of nickel, 5.0 to 25.5 Wt. % of iron, and 3.1 to 14.0 Wt. % of niobium, said alloy having a Vickers hardness of greater than 150 and having an initial permeability above 3,000 and a maximum permeability above 5,000, and a degree of order greater than 0.1 but less than 0.6.
- 2. An alloy according to claim 1, wherein the nickel 45 content is 75.3 to 82.2 Wt.%, the iron content is 9.2 to 20.6 Wt.%, and the niobium content is 3.1 to 10.8 Wt.%.
- 3. An alloy according to claim 1, wherein the nickel content is 78.3 to 81.8 Wt.%, the iron content is 9.6 to point, e.g., below about 600°C, in a non-oxidizing at- 50 15.2 Wt.%, and the niobium content is 4.8 to 10.4
 - 4. An alloy according to claim 1, wherein the nickel content is 79.0 to 81.4 Wt.%, the iron content is 10.1 to 14.2 Wt.%, and the niobium content is 5.7 to 9.8
 - 5. An alloy according to claim 1, wherein the initial permeability is greater than 12,000 and the maximum permeability is greater than 110,000.
 - 6. An alloy according to claim 1, wherein the electric resistivity is greater than 38 $\mu\Omega$ -cm.