

[54] **MAGNET ALLOYS**  
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1965, abandoned.  
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[51] Int. Cl. .... **H01f 1/04**, C22c 31/00  
[58] Field of Search ..... 148/31.57; 75/134, 134.9 M

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[57] **ABSTRACT**

A ternary permanent magnet alloy which is composed of 69.5 to 73.0 percent by weight of Mn, 26.4 to 29.5 percent by weight of Al and 0.6 to (1/3 Mn—22.16) percent by weight of C, and primarily consists of a body-centered tetragonal structure phase, and which has magnetic characteristics of  $BH_{max} \geq 1.0 \times 10^6$  G.Oe,  $Br \geq 2800$  G. and  $BH_c \geq 1400$  Oe., excellent magnetic stability, mechanical properties, weather resistance and corrosion resistance, and are not susceptible to spontaneous disintegration in the atmosphere.

**1 Claims, 6 Drawing Figures**

FIG. 1

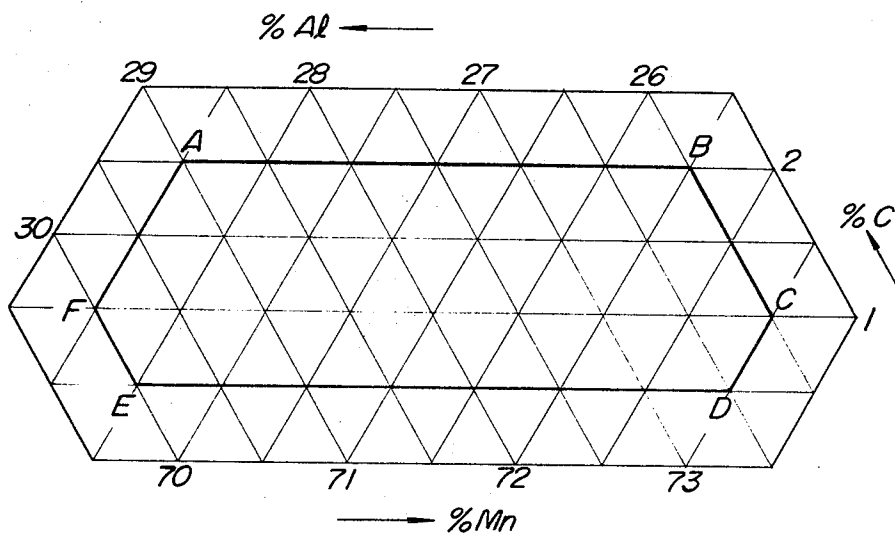
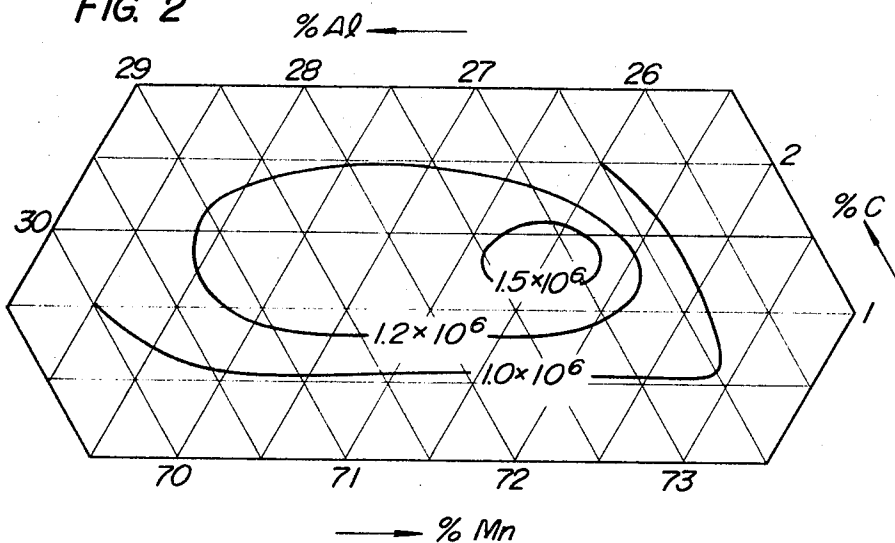


FIG. 2



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FIG. 3

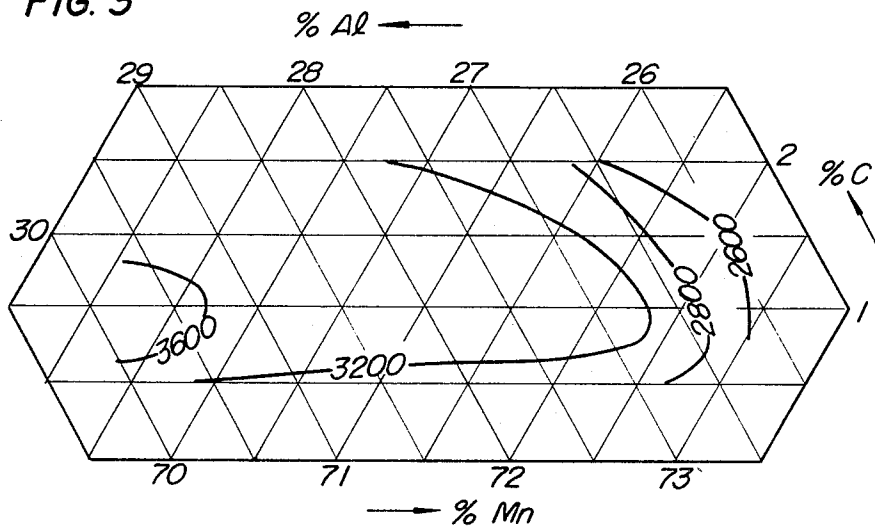
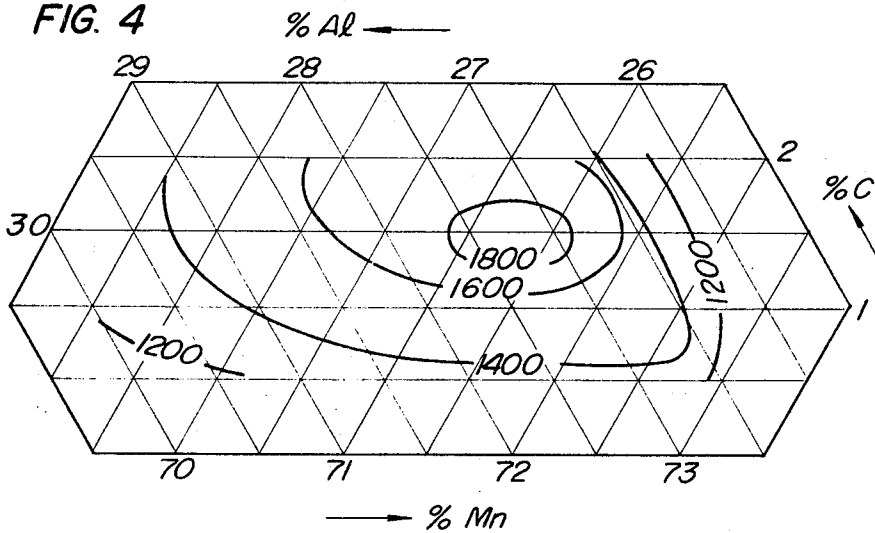


FIG. 4



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FIG. 5

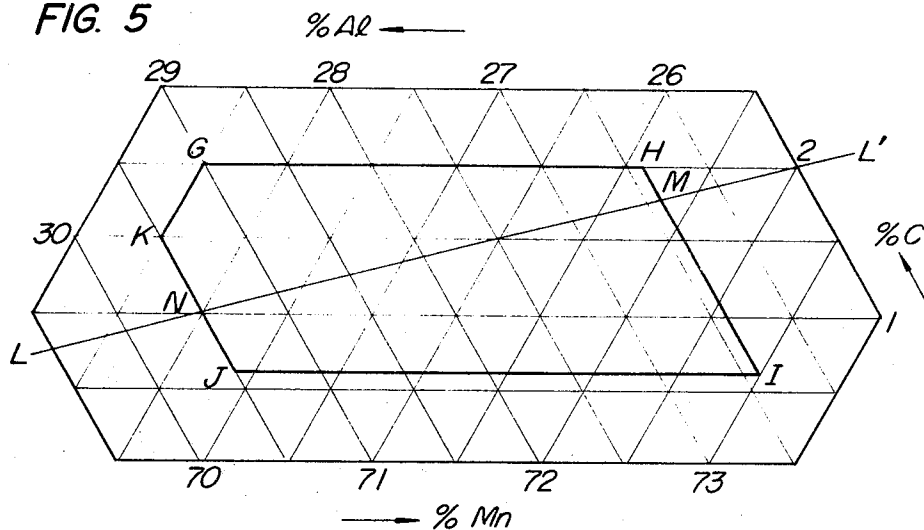
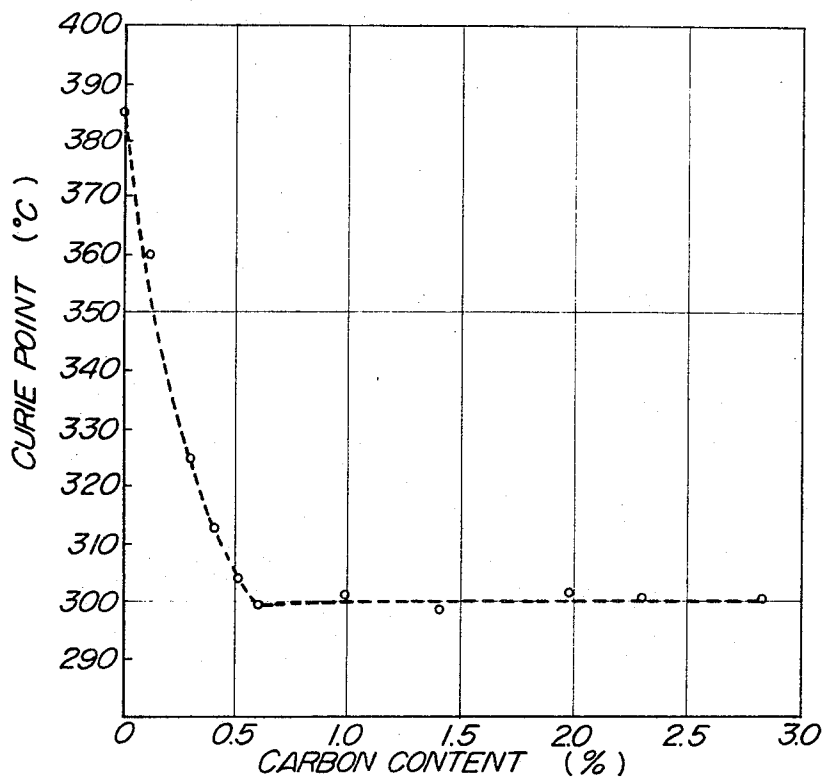


FIG. 6



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## MAGNET ALLOYS

## CROSS-REFERENCE TO RELATED APPLICATION

The present application is a Continuation-in-Part application of Ser. No. 429,260 filed Feb. 1, 1965, now abandoned.

## BACKGROUND OF INVENTION

The present invention relates to manganese-aluminum-carbon (Mn-Al-C) ternary alloys which have excellent magnetic characteristics, corrosion resistance, stability and mechanical properties to be used as a permanent magnet.

Conventional Mn-Al binary alloys have a potential utility as a magnet material but their properties are not sufficiently satisfactory for practical use as a magnet material. For instance, an alloy composed of about 72 percent by weight of Mn and about 28 percent by weight Al, when transformed from the hexagonal crystal structure of  $\epsilon$ -phase, which emerges at elevated temperatures, into the tetragonal crystal structure of metastable phase ( $\gamma$ -phase), by subjecting it to a suitable heat treatment, exhibits ferromagnetism and tentatively shows the properties suitable for use as a permanent magnet. However, the BHmax value of the alloy is not greater than about  $0.6 \times 10^6$  G.Oe. and, in addition, because of its magnetic phase being a metastable phase, the alloy is poor in stability as well as in mechanical properties, so that it is entirely unserviceable for practical applications. There have also been known Mn-Al-C ternary alloys of body-centered tetragonal system containing 2.0 to 5.5 percent by weight of carbon and 0.4 to 15.5 percent by weight of aluminum, but these alloys have magnetic characteristics which are only as good as to be used as a substitute for nickel which does not show the properties required for permanent magnet at all, and thus are not adapted for use as a material for permanent magnet.

## SUMMARY OF INVENTION

The object of the present invention, therefore, is to provide a novel permanent magnet material which has excellent magnetic characteristics, stability, mechanical properties and corrosion resistance, and which primarily consists of a Mn-Al-C ternary alloy, by incorporating into the conventional Mn-Al binary alloy carbon as the third element, to thereby convert said binary alloy into a ternary alloy within a specific composition range so as to improve the magnetic characteristics of the binary alloy drastically.

More specifically, the present invention relates to a magnet material which is composed of 69.5 to 73.0 percent by weight of Mn, 26.4 to 29.5 percent by weight of Al and not less than 0.6 percent by weight but not more than 166 Mn—22.16 percent by weight of C, and which consists primarily of a body-centered tetragonal crystal structure phase which is stable with a sufficient amount of carbon dissolved therein in the solid state, or is unsusceptible to decomposition into a low temperature phase; and has for its object the provision of a permanent magnet material which has an excellent maximum energy product (BHmax), residual magnetic flux density (Br) and coercive force (BHc), and is also excelling in the other properties essential for a permanent magnet, such as magnetic characteristics, stability and mechanical properties. Namely, according to the present invention, there is provided a new permanent magnet material consisting primarily of a Mn-Al-C ternary alloy which has excellent magnetic characteristics, i.e.  $BH_{max} \geq 1.0 \times 10^6$  G.Oe.,  $Br \geq 2,800$  G. and  $BHc \geq 1,400$  Oe., and excellent stability and mechanical properties.

Another object of this invention is to provide a permanent magnet material which is completely free from disintegration in the atmosphere caused upon hydrolysis of aluminum carbides, e.g.  $Al_4C_3$ , by the water present in air in the case where a ternary alloy constituting a magnet material contains an excessive amount of carbon therein, and which therefore has an excellent corrosion resistance.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a diagram illustrating the compositions of the magnet alloys used in the example of the present invention;

FIGS. 2 to 4 inclusive are diagrams illustrating the relationship between the composition of the alloys and the magnetic characteristics thereof, in which FIG. 2 is a BHmax equivalent value curve, FIG. 3 is a Br equivalent value curve and FIG. 4 is a BHc equivalent value curve;

FIG. 5 is a diagram illustrating the compositions of the magnet alloys according to this invention; and

FIG. 6 is a diagram illustrating the relationship between the carbon amount in the magnet alloys and the Curie point of said alloys.

## DESCRIPTION OF THE PREFERRED EMBODIMENTS

The Mn-Al-C alloys used in the experiment were selected from those whose compositions fall in the range defined by points A, B, C, D, E and F in FIG. 1 and were composed of 69 to 73 percent (by weight and so hereinafter) of Mn, 26 to 30 percent of Al and 0.5 to 2 percent of C. Within these ranges of the respective elements, a total of 62 sample alloys were prepared while varying the Mn and Al contents by 0.5 percent and the C content by 0.2 percent from one sample to another. Namely, the sample alloys were prepared for each of the carbon contents consisting of 0.5, 0.7, 0.9, 1.1, 1.3, 1.5, 1.7 and 1.9 percent, and, when the carbon content was, e.g. 1.3 percent, the alloys prepared with this carbon content had the compositions of 69% Mn — 29.7% Al — 1.3% C, 69.5% Mn — 29.2% Al — 1.3% C, 70% Mn — 28.7% Al — 1.3% C, 70.5% Mn — 28.2% Al — 1.3% C, 71% Mn — 27.7% Al — 1.3% C, 71.5% Mn — 27.2% Al — 1.3% C, 72% Mn — 26.7% Al — 1.3% C and 72.5% Mn — 26.2% Al — 1.3% C.

These alloys may be cast by melting them in argon gas, carbon oxide gas, hydrogen gas or in the atmosphere and desired alloys may be obtained by melting the ingredients under the conditions suitable for the specific atmosphere used. In the example to be illustrated hereunder, the constitutional elements were molten mainly in an argon gas atmosphere. The melting points of these ternary alloys, of course, vary somewhat depending upon the compositions thereof but are about 1,260° C. (or in the range of 1,240° to 1,280° C.).

In order to obtain Mn-Al-C ternary alloys having excellent magnetic characteristics, stability, mechanical properties and corrosion resistance, however, three elements of Mn, Al and C must be thoroughly alloyed and, for this purpose, it is necessary to establish the melting temperature within the range from 1,380° to 1,500° C.

Upon melting at the temperature specified above, Mn, Al and C form a homogeneous melt, with carbon dissolved therein the solid state. Successively thereafter, the molten alloy is cast in a suitable mold at that temperature. In practice, the mold to be used must be suitably selected in accordance with the purpose, configuration and dimensions of the alloy desired, but the casting operating can be carried out relatively simply by the use of a die for the sake of experiment. In the production of a magnet adapted for use in power-generating lamps, motors and speakers, the shell mould casting, investment casting and sand mould casting methods are suitably employed. Upon completion of the casting, the alloys may be subjected to homogenizing and hot forging but such treatments are not always required.

Although the Mn-Al-C ternary alloys cast in the manner described show the characteristics required for permanent magnet as such, as will be described later, the magnetic characteristics may be improved greatly by subjecting them to a heat treatment in the manner described hereunder. Namely, the heat treatment basically consists of two steps, i.e. hardening and tempering (aging). The hardening is carried out by rapidly cooling the alloys from a temperature of 900° C. or higher in water, oil, air or an inert gas, irrespective of composition of said alloys, and the cooling rate must be 300°

C./min. or higher. In this case, the temperature of the water, oil or air is only required to be 650° C. or below. In other words, the alloys must be cooled from 900° C. to 600° C. at the rate of at least 300° C./min. In general, the hardening is simply effected at the normal temperature.

Upon completion of the hardening, the alloys are subjected to tempering successively thereafter. The conditions under which the tempering is carried out will largely affect the magnetic characteristics of the product alloys, and effective results can be obtained only under certain range of conditions. Namely, the tempering is effectively carried out for all compositions at a temperature of 480° to 650° C., and the period of tempering becomes longer as the temperature becomes lower and shorter as the temperature becomes higher. For instance, at a temperature of 600° C. a suitable period of tempering is about 80 minutes, and for some composition a period of 1.5 minutes was optimum at a tempering temperature of 650° C., while for some other composition a period of 24 hours was optimum at a tempering temperature of 550° C. In general, it appears that the characteristics of alloys containing a relatively large amount of Mn are improved with an elevating temperature and a prolonged period of tempering, and this relation is reversed for alloys containing a relatively small amount of Mn. However such relations have not been ascertained as yet. When alloys are hardened, for example, in a salt bath at 600° C. from a temperature of 900° C. or higher, tempering of the same may be accomplished successively at that temperature.

By the hardening and tempering as described above, the cast Mn-Al-C ternary alloys are transformed from the hexagonal crystal structure of high temperature phase into a body-centered tetragonal crystal structure of stable magnetic phase having carbon forcibly dissolved therein in the solid state. It is to be noted, however, that the thus heat-treated alloys are not entirely transformed into the body-centered tetragonal crystal structure at every portions thereof but occasionally contain a very small amount of the other phases mixed therein which have no magnetism. These other phases include  $\beta$ Mn type solid solution, AlMn type compound phase, and some sorts of carbides of the hexagonal crystal structure of high temperature phase and of the phases described above with carbon dissolved therein in the solid state or combined therewith.

The Mn-Al-C ternary alloys obtained in the manner described are much tougher than Mn-Al binary alloys and, for instance, the tensile strength, compressive strength and breaking strength of the former are about 5 to 10 times greater than those of the latter. The ternary alloys also have an improved workability providing for cutting work.

It is also to be noted that in the Mn-Al-C ternary alloys, the magnetic phase (body-centered tetragonal crystal structure) thereof is highly stable to such an extent as to be considered as a stable phase, in contrast to that of the Mn-Al binary alloys which is considered as a metastable phase, and such stable magnetic phase is hardly transformed into a low temperature phase.

As stated above, the Mn-Al-C ternary alloys show the characteristics essential for a permanent magnet as they are cast and this is believed to be attributed to such a highly stable magnetic phase as mentioned above.

The magnetic characteristics of the Mn-Al-C ternary alloys, as they are cast, obviously vary depending upon the compositions thereof, but some of them shows a BHmax value of as high as about 0.7 to 0.8 M.G.Oe. However, these alloys as cast contain a considerable amount of high temperature phase remaining therein along with the magnetic phase. In contrast thereto, it has been acknowledged that the alloys which had been subjected to the aforesaid heat treatment successively after casting, had substantially the entire phase thereof transformed into magnetic phase.

It has also been confirmed that the Mn-Al-C ternary alloys show better magnetic characteristics when they contain a very small amount of carbon-containing non-magnetic phase,

peculiar to the ternary alloys, in addition to the above-mentioned magnetic phase.

This indicates that the particularly excellent magnetic characteristics of the Mn-Al-C ternary alloys are attributed, not only to the aforementioned stable magnetic phase having carbon dissolved therein the solid state but also to the kind, amount and the state of distribution of the carbon-containing non-magnetic phase, peculiar to the Mn-Al-C ternary alloy system. However, the detail reasons are not clearly understood at the present stage because the non-magnetic phase is present in only an extremely small amount and distributed extremely finely. In any event, the non-magnetic phase is brought into existence when the amount of carbon contained in the alloys is 0.6 percent or more, and the alloys containing carbon in an amount of 0.6 percent or more are believed to be different in character from those which contain carbon in an amount of less than 0.6 percent. The fact that the characteristics of alloys change at the border of carbon content of 0.6 percent, has also been confirmed by other experiment, e.g. by measuring the Curie point. The relationship between the carbon content and Curie point of alloys containing Mn and Al in the proportion of 72 to 28, is shown in FIG. 6. From this Figure, it is assumed that the maximum content of carbon in the alloys with respect to Mn and Al is about 0.6 percent. It has also been acknowledged that the non-magnetic phase emerges in a large amount as a result of incomplete or no transformation of the structure into the body-centered tetragonal crystal structure or magnetic phase, when the tempering is carried out at temperatures outside the temperature range from 480° to 650° C.

The results of chemical analysis of the alloys formed in the manner described and the magnetic characteristics of said alloys are shown in Table 1 below.

TABLE 1

Sample No.	% Mn	% Al	% C	Br(g)	BHc(Oe)	BHmax (x10 <sup>6</sup> G.Oe.)
1	69.5	30.0	0.5	3300	1050	0.70
2	70.0	29.5	0.5	3200	1150	0.75
3	70.5	29.0	0.5	3150	1300	0.75
4	71.0	28.5	0.5	3150	1300	0.75
5	71.5	28.0	0.5	3100	1300	0.75
6	72.0	27.5	0.5	3000	1300	0.75
7	72.5	27.0	0.5	2900	1300	0.75
8	73.0	26.5	0.5	2700	1100	0.65
9	69.5	29.8	0.7	3600	1200	1.00
10	70.0	29.3	0.7	3550	1300	1.10
11	70.5	28.8	0.7	3500	1400	1.10
12	71.0	28.3	0.7	3400	1450	1.10
13	71.5	27.8	0.7	3400	1450	1.10
14	72.0	27.3	0.7	3250	1450	1.10
15	72.5	26.8	0.7	3000	1450	1.10
16	73.0	26.3	0.7	2650	1050	0.80
17	69.5	29.6	0.9	3700	1300	1.10
18	70.0	29.1	0.9	3500	1400	1.20
19	70.5	28.6	0.9	3500	1450	1.25
20	71.0	28.1	0.9	3400	1500	1.25
21	71.5	27.6	0.9	3400	1550	1.30
22	72.0	27.1	0.9	3300	1500	1.25
23	72.5	26.6	0.9	3050	1450	1.10
24	73.0	26.1	0.9	2550	1000	0.70
25	69.0	29.9	1.1	3900	1300	1.00
26	69.5	29.4	1.1	3650	1350	1.15
27	70.0	28.9	1.1	3550	1500	1.30
28	70.5	28.4	1.1	3500	1550	1.35
29	71.0	27.9	1.1	3400	1600	1.40
30	71.5	27.4	1.1	3300	1600	1.45
31	72.0	26.9	1.1	3250	1550	1.40
32	72.5	26.4	1.1	2800	1350	1.05
33	69.0	29.7	1.3	3650	1300	1.10
34	69.5	29.2	1.3	3550	1400	1.20
35	70.0	28.7	1.3	3500	1500	1.30
36	70.5	28.2	1.3	3450	1600	1.40
37	71.0	27.7	1.3	3350	1800	1.45
38	71.5	27.2	1.3	3300	1850	1.70
39	72.0	26.7	1.3	3200	1550	1.35
40	72.5	26.2	1.3	2700	1200	0.80
41	69.0	29.5	1.5	3500	1350	1.15
42	69.5	29.0	1.5	3500	1450	1.25
43	70.0	28.5	1.5	3450	1550	1.35

44	70.5	28.0	1.5	3400	1700	1.40
45	71.0	27.5	1.5	3300	1900	1.45
46	71.5	27.0	1.5	3250	1900	1.75
47	72.0	26.5	1.5	2850	1450	1.10
48	72.5	26.0	1.5	2600	1000	0.65
49	69.0	29.3	1.7	3500	1350	1.10
50	69.5	28.8	1.7	3450	1500	1.25
51	70.0	28.3	1.7	3450	1650	1.30
52	70.5	27.8	1.7	3300	1750	1.30
53	71.0	27.3	1.7	3250	1800	1.40
54	71.5	26.8	1.7	2950	1750	1.40
55	72.0	26.3	1.7	2650	1300	0.90
56	69.0	29.1	1.9	3450	1400	1.10
57	69.5	28.6	1.9	3400	1500	1.20
58	70.0	28.1	1.9	3300	1650	1.25
59	70.5	27.6	1.9	3200	1700	1.25
60	71.0	27.1	1.9	3050	1700	1.20
61	71.5	26.6	1.9	2800	1600	1.05
62	72.0	26.1	1.9	2400	1000	0.75

The magnetic characteristics shown in Table 1 were plotted into equivalent value curves as shown in FIGS. 2 to 4. The optimum conditions for the tempering, which is greatly influential on the magnetic characteristics of the product alloys, are variable depending upon the composition of the specific sample alloy but the equivalent value curves shown in FIGS. 2 to 4 were drawn using the values of the alloys which were tempered at 600° C. for 80 minutes, which are the temperature and the period to enable excellent characteristics to be obtained on all sample alloys.

As can be seen from FIG. 2, the value of BHmax is greatest when the compositions of the alloys are approximating to 71.5 percent Mn — 27.2 percent Al — 1.3 percent C or 71.5 percent Mn — 27 percent Al — 1.5 C and, within the composition ranges of the alloys used in the experiment, the value of BHmax becomes equal to our greater than  $1.0 \times 10^6$  G.Oe. when the amount of carbon is 0.6 percent or more and the amount of aluminum is 26.3 percent or more.

The value of Br, as is seen in FIG. 3, is highest with the composition in the vicinity of 69.5 percent Mn — 29.5 percent Al — 1 percent C but, with an amount of aluminum in excess to 26.4 percent, the value of Br becomes 2,800 G. or higher. Thus, the particularly excellent composition range for practical application with respect to Br value is extremely wide. Now, the value of BHc becomes 1,400 Oe. or higher when the aluminum content is 26.5 percent or more and carbon content is 0.6 percent or more, and reaches as high as 1,800 Oe. or even higher when the alloy composition is in the vicinity of 71.5 percent Mn — 27 percent Al — 1.5 percent C, as shown in FIG. 4.

From the overall results of the experiment described above and with particular reference to the BHmax which is most critical for magnet material, it may be said that alloys in the composition range of 69 to 73 percent Mn, 26.4 to 29.5 percent Al and 0.6 to 2 percent C show particularly excellent magnetic characteristics. This composition range is indicated in FIG. 5 by the area defined by points G, H, I, J and K. A further experiment conducted using additional samples has revealed the fact that the magnetic characteristics vary drastically between the carbon content of 0.5 percent and 0.6 percent, that the effect of carbon is particularly noticeable when it is added in an amount of 0.6 percent or more, and that the magnetic characteristics vary greatly at the boundary of aluminum content of 26.4 percent.

The Mn-Al-C alloys in the composition range set out above have excellent magnetic characteristics for use as a permanent magnet but are not entirely satisfactory to be used for practical applications because of the following drawback. Namely, alloys containing carbon in an amount more than a certain limit form  $Al_4C_3$ , an aluminum carbide, which is hydrolyzed upon reaction with water present in air. This reaction is represented by the reaction formula provided below and the chemical analysis shows that this reaction is accompanied by the generation of methane gas.  $Al_4C_3 + 12 H_2O \rightarrow 4Al(OH)_3 + 3CH_4$

For this reason, the alloys are disintegrated into powder with the lapse of time after formation, making them unserviceable for practical uses. In the atmosphere, this spontaneous disintegration occurs in about one day after casting at the earliest and in several months after casting at the latest.

The periods before the commencement of disintegration in the atmosphere of the 62 sample alloys depicted in Table 1 are shown in Table 2 below for comparison. These periods were measured from the time of casting to the time when decomposition of  $Al_4C_3$  was observed through a microscope. Those sample alloys which are not depicted in the Table below were not subjected to disintegration and therefore were proved to be excellent not only in moisture resistance and water proof but also in acid resistance and alkali resistance.

TABLE 2

Sample No.	Period before Disintegration	Sample No.	Period before Disintegration
20		25	
25		30	
30		35	
35		40	
40		45	
45		50	
50		55	
55		60	
60		65	
65		70	
70		75	
75		80	
80		85	
85		90	
90		95	
95		100	
100		105	
105		110	
110		115	
115		120	
120		125	
125		130	
130		135	
135		140	
140		145	
145		150	
150		155	
155		160	
160		165	
165		170	
170		175	
175		180	
180		185	
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790		795	
795		800	
800		805	
805		810	
810		815	
815		820	
820		825	
825		830	
830		835	
835		840	
840		845	
845		850	
850		855	
855		860	
860		865	
865		870	
870		875	
875		880	
880		885	
885		890	
890		895	
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960		965	
965		970	
970		975	
975		980	
980		985	
985		990	
990		995	
995		1000	

The line L — L' in FIG. 5 is a border line established one year after the production of the alloys and those alloys containing carbon in an amount of more than that represented by the line L — L' are susceptible to spontaneous disintegration. The disintegration tends to occur earlier as the carbon content moves away from the line L — L'. The observation through a microscope has revealed that the alloys which are susceptible to disintegration contain more amount and larger size of  $Al_4C_3$  phase, whereas those which are less susceptible to disintegration contain only a trace amount of  $Al_4C_3$  phase in their Mn-Al-C phases. Those sample alloys which were not disintegrated even one year after the casting contain no  $Al_4C_3$ . The border line is assumed to pass through the points representing the compositions of alloys of 69.5 percent Mn — 29.5 percent Al — 1 percent C, 71 percent Mn — 27.5 percent Al — 1.5 percent C and 72.5 percent Mn — 25.5 percent Al — 2 percent C, and in order to determine the position of said border line, the following experiment was conducted.

Namely, seven sample alloys were prepared which were composed of a fixed amount and 1.5 percent of C; 70.6 percent, 70.7 percent, 70.8 percent 70.9 percent, 71.0 percent 71.1 percent and 71.2 percent of Mn respectively; and the remainder of Al, and the periods before the commencement of disintegration of those sample alloys were measured, the result of which is shown in Table 3 below. In this case, the sample alloys containing 71.0 percent or more Mn were not disintegrated even after one year.

TABLE 3

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Composition (%)			Period before disintegration	
70	70.6 Mn	27.9 Al	1.5 C	6 months
	70.7 Mn	27.8 Al	1.5 C	8 months
	70.8 Mn	27.7 Al	1.5 C	10 months
	70.9 Mn	27.6 Al	1.5 C	11 months

Consequently, it may be concluded that, when the carbon content is located on or below the line L — L' in FIG. 5, that is,

in the composition range defined by points M, I, J and N, those alloys are not susceptible to spontaneous disintegration.

The line L - L' can be obtained from the relationship between the amounts of C and Mn, as expressed by the formula,

$$C = \frac{1}{2}Mn - 22.16 \text{ (percent)},$$

provided that the amount of Mn is in the range of 69 to 73 percent.

In the sample alloys mentioned above, the impurities in a total amount of up to 2 percent, which are usually contained in Mn, Al and C, will not particularly disadvantageously affect the magnetic characteristics of the alloys.

The present invention which provides a magnet material which is composed of 69.5 to 73.0 percent Mn, 26.4 to 29.5

percent Al and not less than 0.6 percent but not more than ( $\frac{1}{2}$ Mn—22.16 percent of C and which has excellent magnetic characteristics, stability and mechanical properties as a permanent magnet and is excelling in corrosion resistance and unsusceptible to spontaneous disintegration in air, as described hereinabove, is a great industrial value.

What is claimed is:

1. A ternary magnet alloy composed of 69.5 to 73.0 percent by weight of manganese, 26.4 to 29.5 percent by weight of aluminum, not less than 0.6 percent but not more than ( $\frac{1}{2}$ Mn — 22.16 percent) by weight of carbon and essentially free of  $Al_4C_3$ .

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 3,661,567 Dated May 9, 1972

Inventor(s) Hiroshi Yamamoto

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Please insert the following missing claim for priority:

--Japanese No. 5653/64 of February 1, 1964 and  
Japanese No. 12679/64 of March 3, 1964--

Signed and sealed this 24th day of April 1973.

(SEAL)  
Attest:

EDWARD M. FLETCHER, JR.  
Attesting Officer

ROBERT GOTTSCHALK  
Commissioner of Patents