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METHOD OF PRODUCING MAGNETICALLY ANISOTROPIC SINGLE-CRYSTAL MAGNETS

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Fig. 1

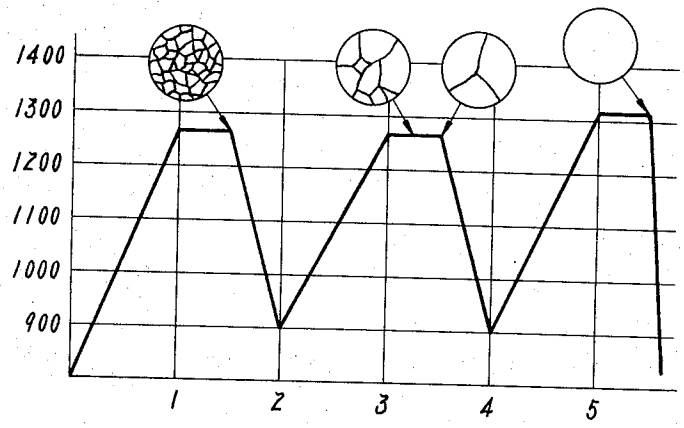
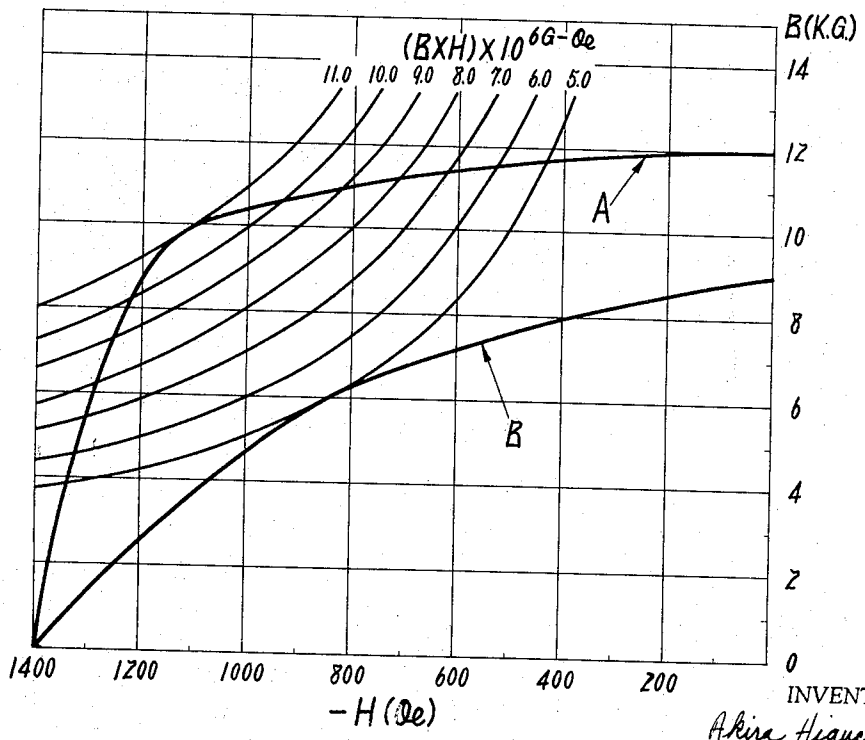


Fig. 2



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METHOD OF PRODUCING MAGNETICALLY ANISOTROPIC SINGLE-CRYSTAL MAGNETS

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ABSTRACT OF THE DISCLOSURE

A method is disclosed for producing a magnetically anisotropic single-crystal ferroalloy magnet containing alumina, nickel, cobalt, copper, titanium and iron. The method comprises subjecting a polycrystal oriented magnet of said composition to a series of more than two heat-treatment cycles, each cycle comprising heating said magnet to 1250–1280° C. at a heating rate of 5–20° C. per minute and then slowly cooling to about 900° C. The single-crystal magnet is then heat-treated in a magnetic field and aged.

This invention relates to a method of producing magnetically anisotropic single-crystal ferroalloy magnets containing Al, Ni, Co, Cu and Ti as principal elements.

The requirement for the increase of the maximum energy product per unit volume of permanent magnet has become larger with the increase of demand owing to the progress of the electronic industries. It is well known that by using a hot mold or a chill plate in casting, a crystal texture and a high magnetic anisotropy will be given to a ferroalloy magnet known as Alnico V containing 8% Al, 14% Ni, 24% Co and 3% Cu as principal elements. It is also reported that by the adoption of a recrystallization to Alnico V alloy, a single-crystal magnet can be obtained and higher permanent magnet properties can be illustrated.

It is further reported that a ferroalloy magnet containing 6–10% Al, 12–20% Ni, 20–42% Co, 0–7% Cu and less than 6% Ti and adding at one of less than 1% Zr, less than 2% Si and less than 3% Nb thereto as required, the balance being substantially iron, can obtain high coercivity by solution treatment followed annealing in a magnetic field and then aging. It is expected theoretically and experimentally that a ferroalloy magnet known as Alnico VIII containing 7% Al, 14% Ni, 34% Co, 4% Cu and 5% Ti as principal elements, may be made as one of the highest performance magnets among the Alnico series permanent magnet alloys. Due to its high coercivity, the maximum energy product of single-crystal Alnico VIII will be more remarkably increased than in the case of Alnico V. However, said Alnico VIII alloy contains in such larger amount of Ti as a principal element that it is very difficult to make it a magnetically anisotropic single-crystal magnet, due to the following reasons:

(1) Its crystal grain size is so small that the average diameter of the grain as ordinally cast is only about 0.3–0.5 mm.

(2) When a hot mold or chill plate is used, slight columnar crystals may be formed. But, in such case, too, the grain is small and short that its permanent magnet properties are not so improved.

(3) When a recrystallization for making a single-crystal magnet from poly-crystal one is applied, where the first heat treatment at a temperature of 920–1175° C. for the precipitation γ-phase and the following heat treatment at a temperature of 1220–1320° C. for the solution treatment and the grain growth are preformed, the grain is made somewhat coarser. But, in such case, the crystal texture is quite random and the permanent magnet properties is little increased.

(4) As the coexisting range of the α- and γ-phases becomes wide as compared with the case of Alnico V, it is not so effective to add specific elements as C, N, Mn, Ru, Pd, etc. for accelerating γ-phase precipitation. Besides, the addition of such elements is nearly impossible from viewpoint of the production technique.

As described above, it is impossible to make industrially this kind of permanent magnet alloy single crystal by the well-known methods. As a result of further investigations for obtaining single-crystal magnet in this kind of alloy, the following new facts have been discovered.

(1) By a high frequency zone-melting method to which the principle of the hot mold method or Bridgeman's method is applied, very fine but columnar crystals can be made in this kind of permanent magnet alloy.

(2) A crystal texture is given in advance to a poly-crystal magnet by the abovementioned method and the magnet is heated to 1250–1280° C. in a heating rate of 5–20° C./min. and then slowly cooled. By applying this cycle of heat treatment at least twice, the magnet can be positively made single crystal in a wide composition range.

(3) By carrying out the heat treatment mentioned in (2) above, in the atmosphere of cracked ammonia or carbon monoxide, coarsening of crystals can be remarkably accelerated.

(4) In the above treatment, it is unnecessary to add said specific elements for promoting the γ-precipitation.

(5) After the treatment in (1) and (2) above, by quenching the magnet from 1250–1280° C., subjecting it to standard magnetic field annealing and then aging, the maximum energy product of the magnet can reach to 11 m.g.o. (million gauss oersted).

It seems to be caused from the fact that on repeating of the heat treatment cycle, lattice relationship between the γ-precipitate and α-matrix is always kept

$$\gamma(111)//\alpha(110)$$

and γ-precipitates in grain boundary play a role to reduce boundary energy and bring easily the boundary migration required for recrystallization.

Thus, the present invention provides an industrial means of making magnetically anisotropic single-crystal magnets. A ferroalloy containing Al, Ni, Co, Cu and Ti as principal elements is melted in a high frequency furnace and then cast in a mold, where the crystal texture is regulated by using a hot mold method or a high frequency zone-melting method to make a <100> orientation. This poly-crystal texture or columnar magnet is heated to 1250–1280° C. in a heating rate of 5–20° C./min. and then slowly cooled to about 900° C. This heat treatment cycle is repeated at least twice. The magnet is then quenched from 1250–1280° C. to obtain a homogenized single-crystal magnet. Then, subjecting the magnet heated about 800° C. near to the Curie point of the magnet to the annealing in a magnetic field parallelly to the crystal orientation and then aging at about 600° C., gives permanent magnet properties excellent in the initial crystal orientation.

In the accompanying drawings;

FIGURE 1 is a diagram of a heat treatment cycle explaining the conversion of a poly-crystal magnet to a single-crystal one according to the present invention.

FIGURE 2 is a B-H curve of the single-crystal magnet based on the present invention as compared with that of a conventional poly-crystal one.

The conversion from the poly-crystal magnet to single-crystal one caused by the heat treatment according to the present invention is as shown in FIGURE 1 and the heat

treatment cycle is very effective to this purpose. Further, as the initial crystal orientation does not collapse, as shown in FIGURE 2, remarkable increases of permanent magnet properties represented by A to B in Alnico V alloy and C to D in Alnico VIII alloy can be brought by the present invention. An example thereof is now mentioned in Table I.

TABLE I

Alloys	Alnico V Series	Alnico VIII Series
Compositions	8% Al; 14% Ni; 24% Co; 3% Cu; 0.5% Si; Bal. Fe.	7% Al; 14% Ni; 36% Co; 3% Cu; 5.5% Ti; 0.5% Nb; Bal. Fe.
(1) Random crystal orientation heat-treated in magnetic field and aged at 600° C.	Br=12.5-13.2 kg., Hc=600-650 oe., $(BH)_m$=4.5-5.5 m.g.o.	Br=8.5-9.5 kg., Hc=1,350-1,550 oe., $(BH)_m$=4.5-5.5 m.g.o.
(2) Random crystal orientation recrystallized and coarsened, heat-treated in a magnetic field and aged at 600° C.	Br=13.5-14.5 kg. Hc=600-730 oe., $(BH)_m$=5.5-8.0 m.g.o.	Br=8.5-5.5 kg., Hc=1,300-1,500 oe., $(BH)_m$=4.5-5.5 m.g.o.
(3) (100) crystal orientation, heat-treated in a magnetic field and aged at 600° C.	Br=13.0-14.0 kg., Hc=650-730 oe., $(BH)_m$=6.5-8.0 m.g.o.	Br=10.0-11.0 kg., Hc=1,350-1,550 oe., $(BH)_m$=6.5-8.0 m.g.o.
(4) (100) crystal orientation, single crystallized and coarsened by 3 cycles, heat-treated in a magnetic field and aged at 600° C. (according to the present invention).	Br=13.5-14.5 kg., Hc=730-780 oe., $(BH)_m$=10.0-10.5 m.g.o.	Br=11.0-12.0 kg., Hc=1,400-1,600 oe., $(BH)_m$=10.5-11.5 m.g.o.

That is to say, the permanent magnet properties obtained by repeating heat treatment cycle for recrystallization at least twice after carrying out the crystal orientation has little deviation and shows a uniformly highest value. Specifically, the Alnico VIII alloy in which crystal coarsening is hard to obtain, can be well single-crystallised. Without cutting single-crystal magnet out of a part of a casting as before, the casting itself is of single-

crystal having a fixed orientation. The discovery that such heat treatment cycle can be easily embodied in in a continuous furnace is a feature of the present invention and very effective to the industry.

What is claimed is:

1. A method of producing a magnetically anisotropic single-crystal ferroalloy magnet having a composition consisting of 6–10% Al, 12–20% Ni, 20–42% Co, .0–7% Cu, less than 6% Ti and the balance Fe, comprising subjecting a polycrystal oriented magnet of said composition to a series of more than two heat-treatment cycles, each said cycle comprising heating said magnet to 1250–1280° C. at a heating rate of 5–20° C. per minute and then slowly cooling to about 900° C., and then heat-treating the resultant single-crystal magnet in a magnetic field and aging said magnet.

2. A method of producing a magnetically anisotropic single-crystal ferroalloy magnet having a composition consisting of 6–10% Al, 12–20% Ni, 20–42% Co, .0–7% Cu, less than 6% Ti and at least one taken from the group consisting of less than 1% Zr, less than 2% Si, and less than 3% Nb, comprising subjecting a polycrystal oriented magnet of said composition to a series of more than two heat-treatment cycles, each said cycle comprising heating said magnet to 1250–1280° C. at a heating rate of 5–20° C. per minute and then slowly cooling to about 900° C., and then heat-treating the resultant single-crystal magnet in a magnetic field and aging said magnet.

References Cited

UNITED STATES PATENTS

1,738,307	12/1929	McKeehan	148—1.6
3,027,281	3/1962	Osborn et al.	148—1.6
3,175,901	3/1965	Jesmont et al.	148—103
3,219,495	11/1965	Steinort	148—1.6
3,219,496	11/1965	Steingrover et al.	148—1.6
3,226,266	12/1965	Jesmont et al.	148—103

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