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G. HEIMKE

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PREPARATION OF SINTERED PERMANENT ALNICO MAGNETS

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Fig. 1

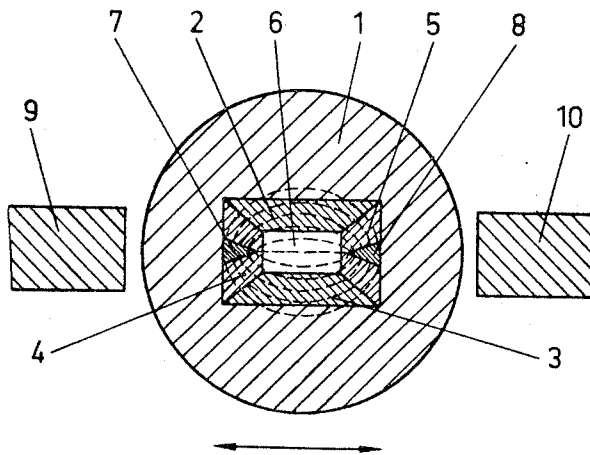
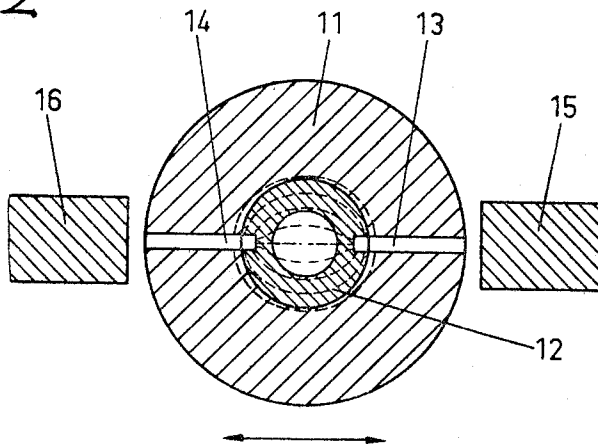


Fig. 2



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PREPARATION OF SINTERED PERMANENT ALNICO MAGNETS

Gunther Heimke, Buschdorf, Germany, assignor to Magnetfabrik Bonn G.m.b.H., vorm. Gewerkschaft Windhorst, Bonn, Germany

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2 Claims

ABSTRACT OF THE DISCLOSURE

Method of making a sintered permanent alnico magnet by filling a die with the powdered component materials while subjecting them to an inhomogeneous magnetic field having a concentrated transverse field extending in the direction of movement of the powder into the cavity, compacting the powder, sintering and heat treating the compacted body in a magnetic field.

This invention relates to the preparation of sintered metallic permanent magnets.

Permanent metallic magnets are prepared from alloys which, in addition to iron, contain essentially 15–42% of cobalt, 11–20% of nickel, 5–11% of aluminum, 0–6% of copper, and 0–8% of titanium. In addition, vanadium, niobium, sulfur, and impurities may be present, jointly, alone, or in various combinations, in such alloys.

Roughly, such permanent magnets can be divided into four groups:

(1) Magnets whose magnetic properties are the same in all spatial directions (isotropic magnets). Such sintered or cast magnets show magnetic values of about $(BH)_{\max} = 2 \times 10^6$ gauss-oersteds.

(2) Magnets where a suitable heat treatment in a magnetic field has produced a magnetically preferred spatial direction. With such magnets, magnetic values in the range of 5×10^6 gauss-oersteds, measured in said preferred direction, can be attained.

(3) Magnets which are given first a crystallographically preferred direction so that the major portion of the crystallites lies parallel with one of their (100)-directions (columnar crystallization); subsequently, said magnets receive a heat treatment in a magnetic field so as to render the magnetically and the crystallographically preferred direction coincident. With cast magnets of this type, magnetic values up to $(BH)_{\max} = 9 \times 10^6$ gauss-oersteds could be obtained. Magnets of this type prepared by sintering have not yet been commercially offered to any larger extent.

(4) Magnets which consist of a monocrystal and where the magnetically preferred direction lies parallel to a (100)-direction of the crystal. Such magnet types are not commercially available in larger amounts. Heretofore, they could be prepared only by drawing from a melt under laboratory conditions, or by the quite expensive zone melting procedure from rods previously prepared from melts. The preparation of such monocrystals by means of an continuous casting process has also been considered.

Though sintered magnets having a preferred crystallographic direction have not yet been produced commercially to any large extent, a number of preparation methods has already been proposed:

(a) Monocrystalline particles of an alloy which has the same or a similar composition as the alloy involved, are inserted into the powdery metal composition used to prepare the alloy. Before said mixture is compacted to the final form, it is subjected to a magnetic field which

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orients the alnico monocrystal particles with one of their (100)-directions parallel to the magnetic field. Said oriented crystallites have then the function to act as crystal seeds during the sintering of the pressed magnets and, due to their orientation, to produce a preferred crystallization in the polycrystal. Said procedure requires application of a strong homogeneous magnetic field to the mold shortly before the metal powder mixture is compressed. The magnetic field must be strong in order to ensure turning of the monocrystal particles used as seeds, with respect to the friction forces of the surrounding powder particles; the field must be homogeneous in order to avoid translatory forces on the seeds and the powder mixture and the resulting inhomogeneities of the density.

(b) In the mixture of the metal powders making up the alloy, there are embedded wires of one of the components of the alloy in such a way that the axes of the wires coincide with the later columnar axis. In proportioning the components of the powder mixture, the amount of the component introduced as wire has to be taken into account. This procedure is complicated for commercial application because the manipulation of the wires, which have a diameter of only about $\frac{1}{10}$ mm., and their proper arrangement in the mold is time consuming.

(c) It has also been proposed to obtain preferred crystallization in sintered permanent magnets of the type here involved by producing a concentration differential of one of the alloy components in the powder mixture within a sintered magnetic body. The thus produced distinction of a spatial direction in the sintered body can result in a preferred crystallization in one spatial direction and therewith in formation of a column. This procedure requires multiple compression steps for producing the concentration differential, which results in additional cost.

(d) A recrystallization treatment has also been proposed to produce preferred recrystallization. There, an already sintered magnet body is heated to a temperature which is close to the melting point, and is then subjected to a temperature gradient in direction of the desired preferred crystallization. This procedure, as disclosed and claimed in application Ser. No. 257,835, filed Feb. 12, 1963 is well suited only for rather simple magnet shapes.

(e) A single finished monocrystal, or a piece of a finished columnar crystal is inserted in the metal powder composition to be compressed to a compact magnet body, whereby such insert is placed in the direction of the desired orientation, i.e. with one (100)-direction or the columnar axis parallel to the desired preferred direction. This procedure is disclosed e.g. in Patent No. 2,617,723, column 6, lines 6–27.

(f) In further development of the procedure (e), Herbert W. Höner and I have magnetized said monocrystals or columnar crystal particles before placing the same into the metal powder composition, and we obtained a considerably increased yield of magnets with properly oriented monocrystals or coarse crystals.

In further development of the line of research set forth above under (d), I found that still better and more reliable results are obtained when, instead of seeding the magnetic powder with an actual magnetized monocrystal or the like, there is applied to the powder mixture while being filled into the mold a magnetic field approximately simulating the stray field of said magnetized seed crystal.

Therefore, I do no longer place a seed crystal into the alloy powder but I produce in the mold cavity a very inhomogeneous magnetic field whose strength in the maximum of the field distribution is below 500 oersteds. I have obtained particularly good results with a magnetic field having a maximum value of 50 oersteds.

In contrast to the methods proposed heretofore, the new method makes use of a strong inhomogeneity of a relatively weak magnetic field. Thereby, the preferred direc-

tion is determined by the axis of the magnetic dipole placed against the mold. The inhomogeneous field distribution within the mold cavity should be as symmetrical as possible.

In the accompanying drawing, I have shown, by way of example, two devices suitable for carrying out the invention in FIGS. 1 and 2, and I will describe said devices in connection with the following examples. Of the examples, Example 1 describes the method referred to above under (f) which, to the best of my knowledge, is also new and which has been disclosed in German application M 61,982 VIIIe/21g filed by Magnetfabrik GmbH. of Bonn, Germany, on Examples 2 and 3 show the further development which is the object of this invention.

EXAMPLE 1

Method using magnetized monocrystals to produce orientation

Alnico alloy powder composed of 13% of nickel carbonyl powder, 32.5% of a powdered cobalt-aluminum prealloy, 0.2% of a ferro-aluminum prealloy, 3% of copper powder, and 51.3% of iron carbonyl powder, was placed into a hard metal die so as to fill $\frac{2}{3}$ of the available capacity. Then, a columnar crystal was placed into the charge. Such columnar crystals had been obtained from finished cast magnets of the same composition by fracturing the same with a hammer. Said cast magnets were in the magnetically optimal state. The columnar crystals had irregular shapes; their largest diameter was about 3 mm., and their length in the preferred direction, i.e. measured parallel to the columnar axis, at least 5 mm. Said columnar crystals were placed parallel to their columnar axis in a magnetic field of 1800 oersted and magnetized. Then, they were placed with their columnar axis parallel to the direction which was to become the preferred direction of the sintered magnet body. After filling up the mold cavity with the alnico powder, the magnet bodies were prepared by applying a pressure of 2400 kg./cm.². In this way, 20 samples were made. Said samples were sintered at a temperature of 1320° C. and under a vacuum of 3.10^{-4} torr for 7 hours. The sintered samples were subjected to the conventional thermomagnetic and annealing after-treatments, as described, e.g. in Patent No. 2,499,860 and in the paper "Permanent Magnets With High Coercive Force on the Basis of Fe-Co-Ni-Al Alloys" by A. S. S. Koch, M. G. van der Steeg and K. J. de Vos, published in "Berichte der Arbeitsgemeinschaft Ferro-magnetismus," 1959, pp. 130-139.

The magnetic field applied during the heat treatment was directed parallelly to the direction of the embedded seeds. The samples were magnetically measured in said direction and fractured to observe the orientation of the crystallites. Fourteen of the samples showed a $(BH)_{\max}$ value of more than 5.5×10^6 gauss-oersteds.

EXAMPLE 2

Novel method

The same powdered mixture for the preparation of an alnico alloy, as used for Example 1, was placed in the 15.10 mm. cavity of a hard metal die to a height of 10 mm. Two soft iron knife edges were inserted in the hard metal wall of the die as shown in FIG. 1, where the reference numeral 1 designates a clamping ring of non-magnetic steel; 2, 3, 4, and 5 are hard metal parts, 6 the die cavity and 7 and 8 the two soft iron wedges. 9 and 10 are the pole pieces of a small electromagnet arranged outside the die. The current intensity for the electromagnet was so adjusted that in front of the edges of the soft iron wedges inside the die, a field strength of 30 oersteds could be measured. After charging the alloy powder, a pressure of 2400 kg./cm.² was applied by means of a non-magnetic ram, and the compressed body was ejected in the usual manner after the current through the small magnet yoke had been turned off.

20 of the thus prepared samples were then sintered and after treated exactly as described above for the samples of Example 1. In this case, however, 19 of the samples showed a $(BH)_{\max}$ value in excess of 5.5×10^6 gauss-oersteds.

The visual control after fracturing the samples showed that 14 of said 19 high-value magnets had monocrystal structure; of the residual 5 good magnets, one had 2, one had 3, one had 5, and two had 6 massive crystals of essentially favorable orientation.

EXAMPLE 3

Novel method

Alloy powder of the composition set forth in the preceding examples was filled into the cavity of a hard metal die of 30 mm. inside diameter. The die was of the type shown in FIG. 2 and consisted of a ring 11 of non-magnetic steel and a hard metal bushing 12. Bores 13 and 14 had been made by electroerosion through the ring 11 into the bushing 12, which bores extended to 3 mm. from the inner wall of the bushing. Soft iron wires were introduced into said bores in such a way that they touched the bottom of the bores and projected with the other ends just 2 mm. beyond the periphery of the die. 15 and 16 are the pole pieces of a small electromagnet disposed outside the die. The current intensity through the coil of the electromagnet was so adjusted that in front of the tips of the iron wires inserted into the bores 13 and 14 inside the hard metal bushing, there was a field strength of 40 oersteds. The powder was compressed with a pressure of 2400 kg./cm.² by means of non-magnetic rams, and the compacted body was ejected from the die after the current for the coil of the magnetizing yoke had been turned off. In this way, 20 such pressed bodies were prepared. They were sintered and heat-treated in a magnetic field as described in the preceding examples. The direction of the magnetic field applied during the heat treatment conformed to the line connecting the tips of the iron wires in the die. The finished sintered bodies were compared with ten sintered bodies which had been similarly prepared but with the two differences that, during the filling of the powder into the die cavity, the magnetic field was not switched on, and that the sintering time was not seven but only two hours. When the samples were placed into a soft iron return ring whose inner diameter was 4 mm. larger than the outer diameter of the samples, the field strength of the samples prepared in a magnetic field was 12-18% greater than that of the samples which had been compressed without a magnetic field. Thereby, the field was measured at those places of the periphery of the magnets which in the die cavity had been opposite the iron wires. After fracturing the 20 magnets compacted in the magnetic field, the visual control showed monocrystal structure in 11 cases; in six cases, the magnets consisted of two to five massive crystals of approximately correct orientation. In three magnets, there was, in addition to 2 or 3 massive crystals, still a portion of fine crystalline structure in the inside.

All ten comparison magnets which had been prepared without a magnetic field and with shortened sintering times, showed the polycrystalline fine grain structure typical of sinter magnets.

Compared with the insertion of seed crystals, my method has the advantage of eliminating the need of interrupting the operating cycle for such insertion. Such insertion had to be done very carefully because the proper placing of the small seeds in the loose alloy powder controlled the proper orientation in the subsequent preferred crystallization. This step is completely omitted in the method of this invention. The electric current for the small magnetizing yoke to produce the magnetic field across the die cavity while it is being filled with, and contains, the alnico powder can be automatically switched on and off by, or simultaneously with, the operation of the die.

Compared with previously proposed methods which operate with a magnetic orientation of the alloy powder or of the seed inserts, my method has the advantage of requiring very small field strengths whereby, at the same time, the yield of magnets having the desired crystal orientation is increased to 90–100%. While heretofore it has been always assumed that strong magnetic fields are required to produce magnetic orientation in view of the relatively weak anisotropy of the powdery alloy components, I have found that, with an arrangement as shown, very weak magnetic fields give excellent results. Though I am not yet in a position to give a satisfactory theoretical explanation for the produced effect, I assume that the different magnetizability of the components of the powdery mixture used has a bearing on the observed phenomenon.

I claim:

1. A method of preparing sintered permanent alnico magnets consisting of a monocrystal or few massive crystals oriented with a (100)-direction parallel to the direction of a magnetic field, said method comprising filling a die cavity with powders of the respective individual elements, or compounds or prealloys of said elements, which are effective to make up said alnico magnet, subjecting said powder while it is being filled to an inhomogeneous magnetic field said inhomogeneous magnetic field having a maximum strength smaller than 500 oersteds and the greatest field gradient parallel to a (100)-direction of the finished magnet, pressing said powder to compacted bodies,

sintering said bodies, and subjecting said compacted sintered bodies to a heat treatment in a magnetic field.

2. The method as claimed in claim 1 wherein the field distribution in the die cavity is symmetrical, the axis or plane of symmetry corresponding to the direction of preferred magnetic orientation in the final magnet.

References Cited

UNITED STATES PATENTS

10	3,387,972	6/1968	Inoue	75—200 XR
	2,188,091	1/1940	Baermann	148—103 XR
	2,384,215	9/1945	Toulmin	75—226 XR
	3,066,355	12/1962	Schloemann et al.	148—103 XR
15	2,694,790	11/1954	Studders	148—103 XR
	2,825,670	3/1958	Adams et al.	75—200 XR
	2,849,312	8/1958	Peterman	75—201
	3,066,355	12/1962	Schloemann	148—103 XR

FOREIGN PATENTS

1,045,006 3/1958 Germany.

L. DEWAYNE RUTLEDGE, *Primary Examiner.*

25 P. WEINSTEIN, *Assistant Examiner.*

U.S. Cl. X.R.

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