

April 9, 1963

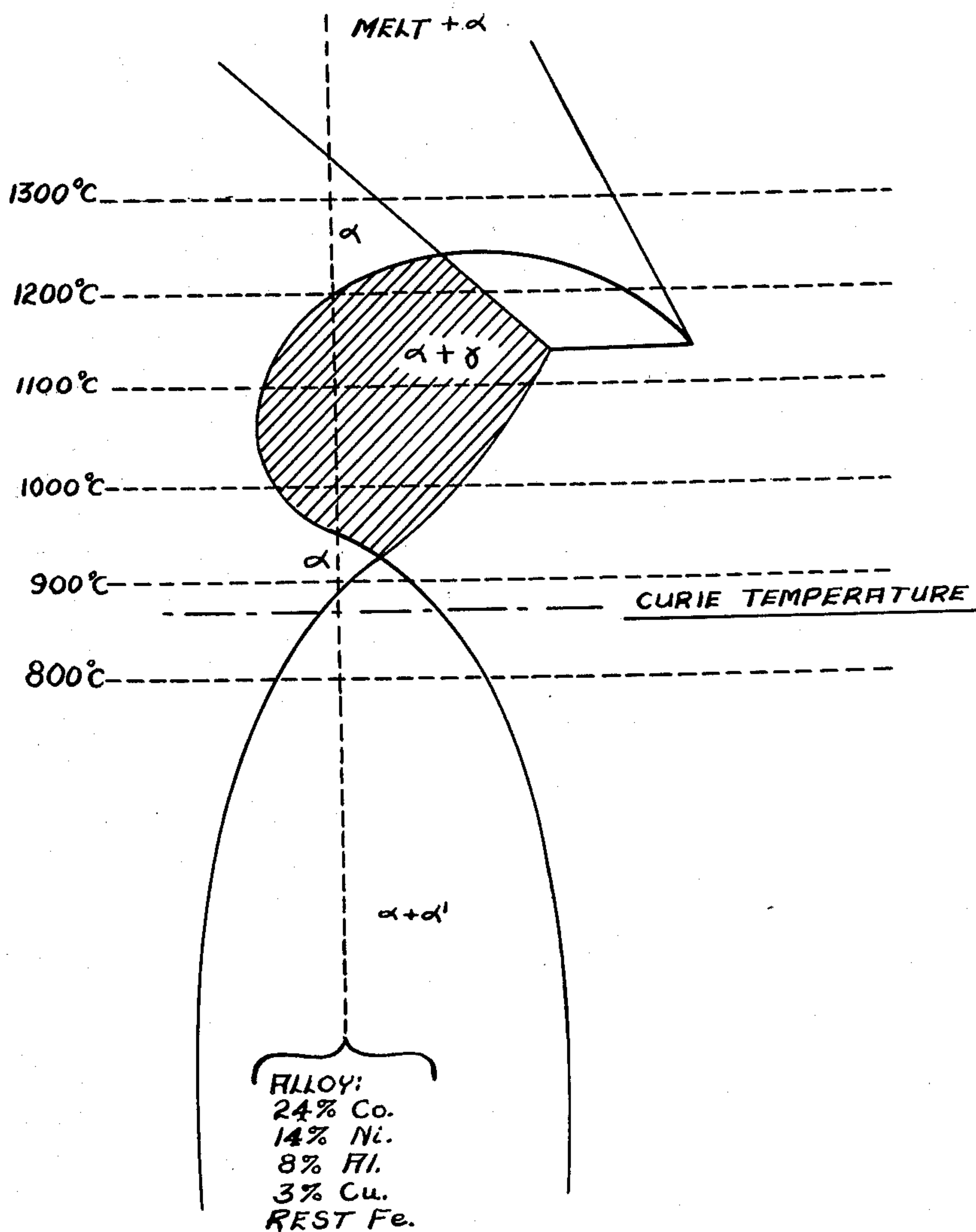
E. STEINORT
MONOCRYSTALLINE PERMANENT MAGNETS
AND METHOD OF MAKING THEM

3,085,036

Filed March 11, 1960

2 Sheets-Sheet 1

FIG. 1



INVENTOR.

EBERHARD STEINORT,
BY

Schley, Dask & Jenkins
ATTORNEYS.

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2 Sheets-Sheet 2

FIG. 2

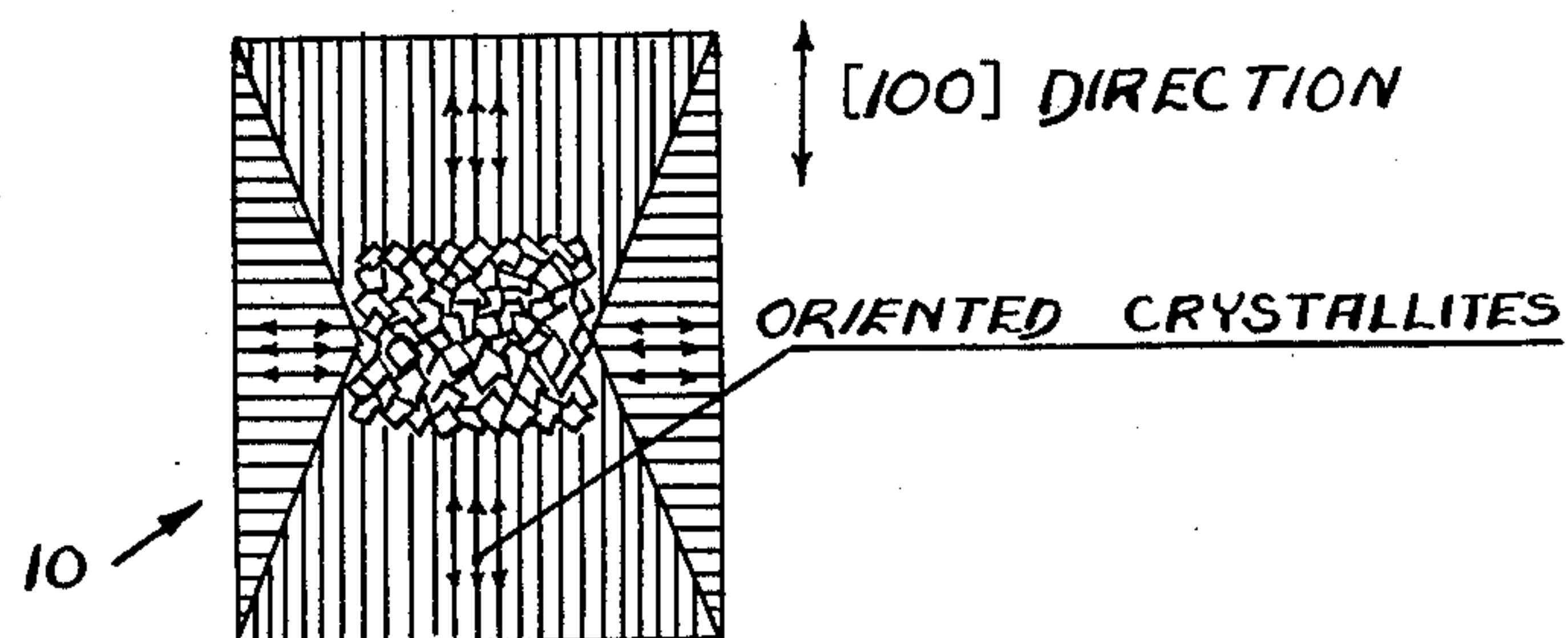


FIG. 3

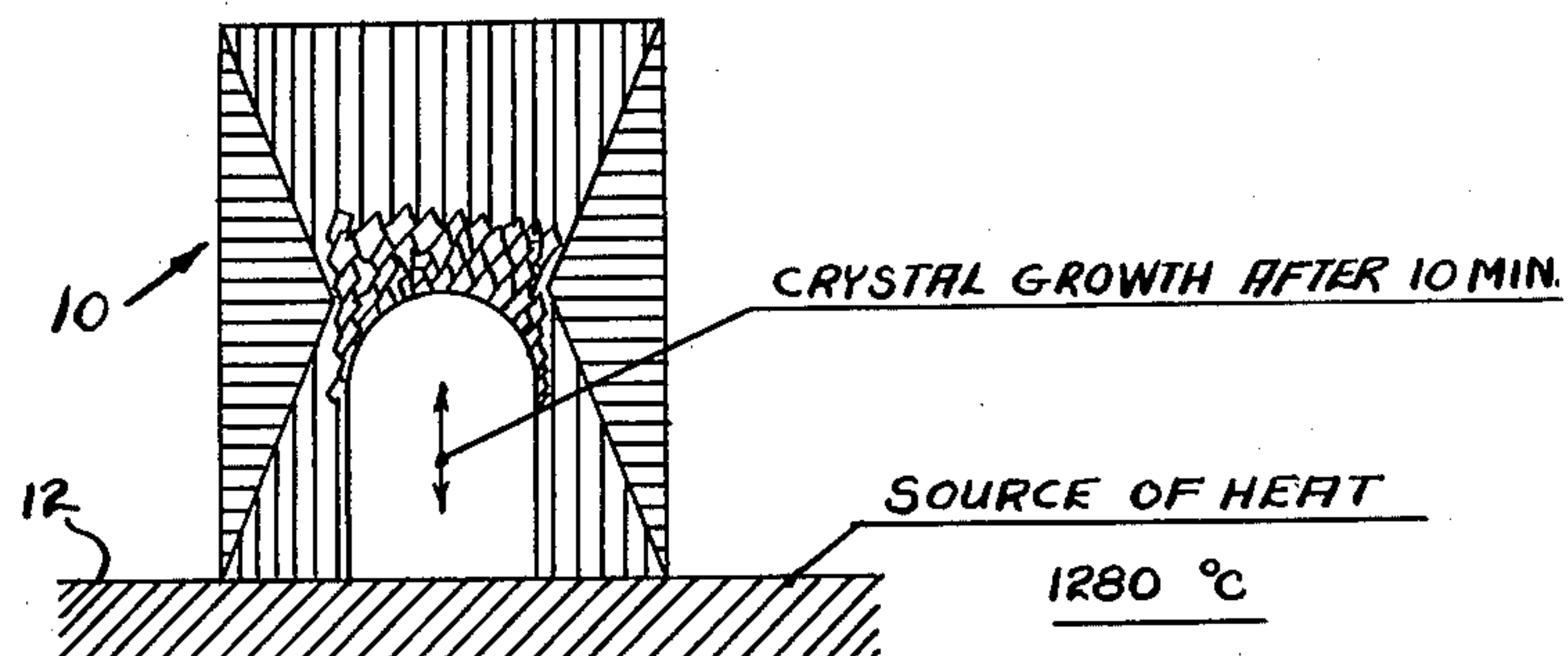


FIG. 4

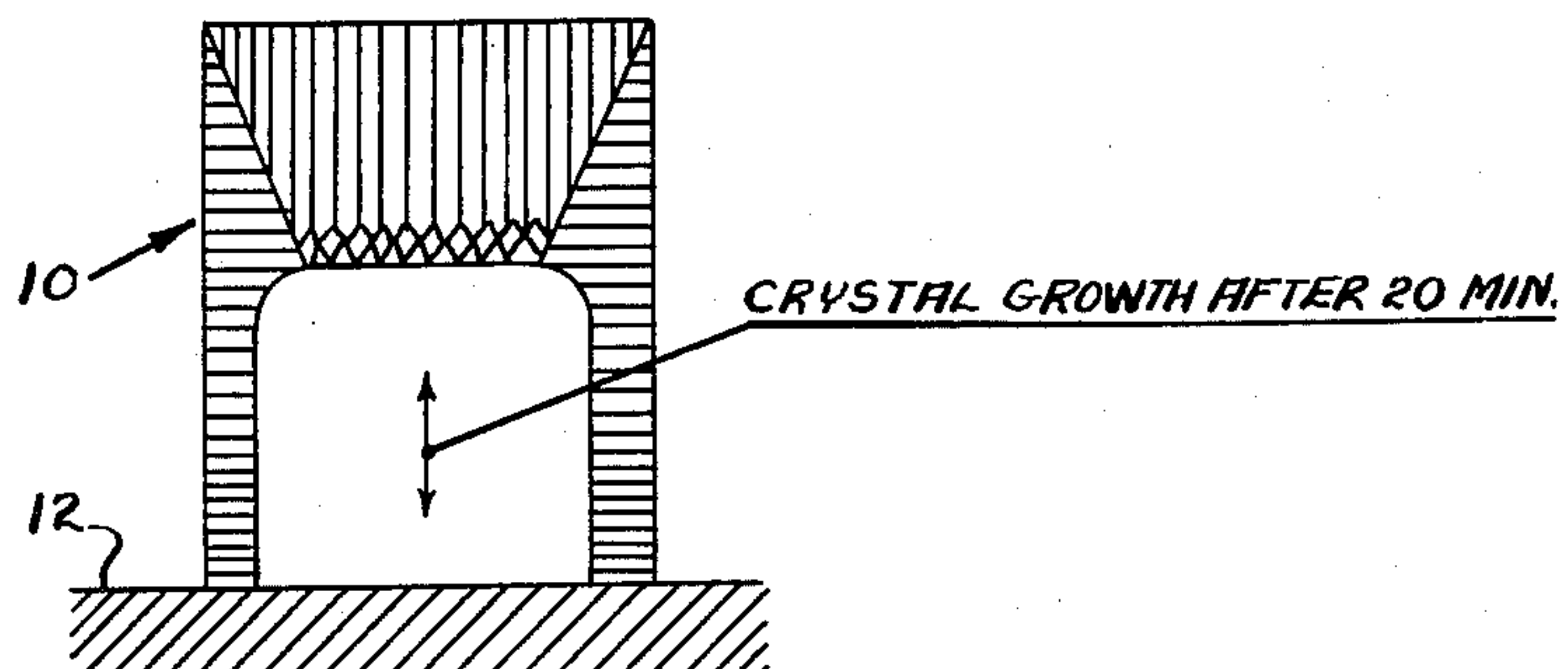
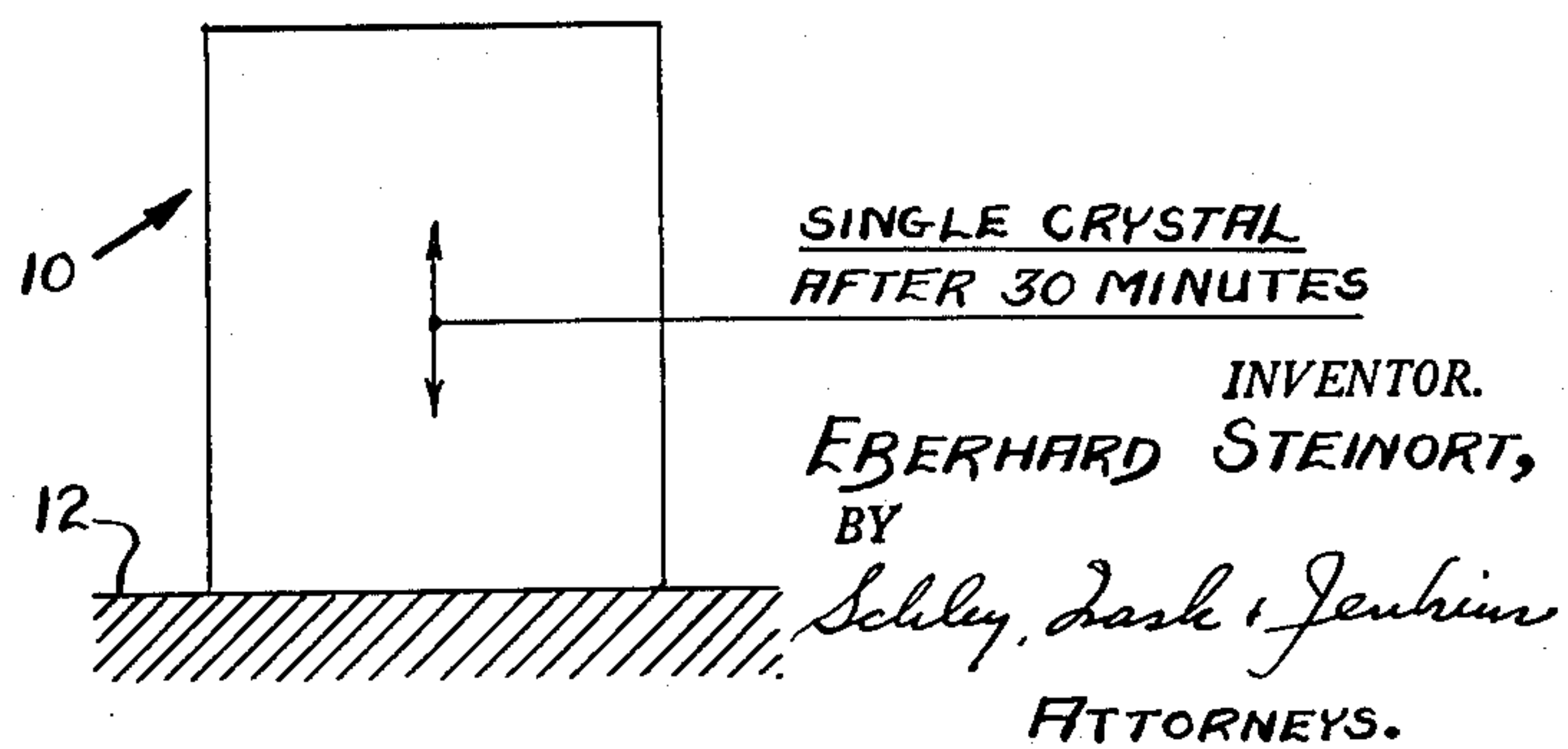


FIG. 5



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3,085,036

MONOCRYSTALLINE PERMANENT MAGNETS
AND METHOD OF MAKING THEM

Eberhard Steinort, Saronno, Italy, assignor to Centro
Magneti Permanenti, Milan, Italy, a corporation of
Italy

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This invention relates to permanent magnets, especially to iron-base anisotropic permanent magnets, more particularly to a method of obtaining monocrystalline structure therein, and to monocrystalline magnets so produced.

A series of high-strength permanent magnet alloys are known, as from the Mishima U.S. Patent Nos. 2,027,994 to 2,028,000, which may be referred to as iron-nickel-aluminum type alloys, since they are basically composed of iron and nickel and aluminum, with or without one or more other elements, such as cobalt, copper, and various addition elements. My invention is applicable to this iron-nickel-aluminum type permanent magnet alloy, especially as the same is represented by the known Alnico type alloys containing from 10 to 30 percent nickel, 6 to 14 percent aluminum, 5 to 42 percent cobalt, up to 8 percent copper, up to 10 percent titanium, and the balance substantially all iron, which balance may include up to 5 percent of one or more of various known addition elements, for example, silicon, zirconium, columbium (niobium), and the like, and which commonly includes small amounts of carbon, manganese, sulfur, and other elements as impurities. The invention preferably employs an Alnico alloy containing from 12 to 20 percent nickel, 6 to 11 percent aluminum, 16 to 30 percent cobalt, 2 to 6 percent copper, from a trace to 7 percent titanium, and the balance substantially all iron. By the term "up to" a stated percent, I mean a range from zero percent to the stated percent, inclusive.

It has long been known, as from the Jonas Patent No. 2,295,082 and the Ebeling Patent No. 2,578,407, that the inherently excellent permanent magnetic properties of Alnico alloys can be further enhanced unidirectionally by means of various pouring and heat-treating techniques. The magnetic anisotropy induced by such means is known as the direction of preferred orientation, and such direction of preferred orientation should have a predetermined relationship, desirably parallel, to the intended end-use direction of magnetization. As an example, magnets of an alloy of the typical analysis listed in line 1 of Table A below, if heat-treated to obtain the best isotropic properties, will attain the magnetic values tabulated in line 2 of such Table A. If magnets of the same alloy are given optimum directional hardening, as

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by optimum directional heat-treatment in a magnetic field and subsequent drawing, as in accordance with the Jonas patent, improved values in the preferred direction will be obtained as shown in line 3. Still further improvement can be achieved if the magnets are poured in such a manner that the crystallites grow with their (100) axis in the preferred direction, as in accordance with the Ebeling Patent No. 2,578,407, or the Koch et al. Patent No. 2,862,287. To accomplish this, the magnets must be subjected to a critical directional cooling treatment during solidification, followed by directional heat-treatment in a magnetic field and suitable draw. The properties listed in line 4 can be reached if complete orientation of the dendrites is achieved.

TABLE A

1. Composition (percentages)	Co 24.0; Ni 14.0; Al 8.0; Cu 3.0; C 0.02; Si 0.05; Mn 0.04; S 0.03; Fe rest		
	Gauss	Oersted	Gauss × Oersted
2. Best isotropic properties	Br=8,700 (rating 100%).	Hc=590 (rating 100%).	BH _{max} =2.14×10 ⁶ (rating 100%).
3. Typical anisotropic properties.	Br=12,900 (rating 148%).	Hc=650 (rating 110%).	BH _{max} =5.0×10 ⁶ (rating 234%).
4. Properties with complete crystal orientation and anisotropic heat-treat.	Br=13,300 (rating 153%).	Hc=800 (rating 136%).	BH _{max} =7.0×10 ⁶ (rating 327%).

Even greater magnetic properties can be obtained, ranging up to the theoretical maximum, if the magnet consists of a single crystal whose (100) axis lies parallel to the direction of magnetic orientation. Such single crystals have been successfully produced by growing the crystal through gradual withdrawal from the melt. This process is, however, very difficult and, while it represents a known scientific approach to single crystal formation, it is not suitable for manufacturing commercial magnets. Single crystals of the alloy listed above with the proper heat-treatment in the direction of the (100) axis can achieve the following properties:

Single-crystal magnet properties: Br=13,900 (rating 160%); Hc=860 (rating 146%); BH_{max}=10.2×10⁶ (rating 477%).

This data indicates that a monocrystalline magnet with optimum directional heat-treatment can show a 477% increase in energy values over the same magnet in polycrystalline isotropic state.

The principal object of this present invention is to provide a commercially practical technique for the development of monocrystalline magnets. The invention is based on the application of secondary recrystallization phenomena which, under proper conditions, result in such marked grain growth that in the end a single grain absorbs all other crystallites and the normal polycrystalline structure of a magnet casting is converted into a monocrystalline structure. It is a further object of the invention to provide a method by which to guide the grain growth in such a manner that the (100) axis of the monocrystalline structure bears a predetermined relationship to the desired preferred orientation.

Large crystal grain growth resulting from secondary recrystallization is known to occur in metals such as pure iron if two critical conditions are met, namely, (1) that the metal receive a critical degree of cold deformation to induce internal mechanical strains which set up large energy differentials between distorted crystal lattice and undistorted crystal lattice, and (2) heat treatment at a

temperature in a narrow critical transformation range. The attainment of the critical heat treatment temperature for the above-mentioned magnet alloys presents no great difficulty; but because of the inherent brittle nature of such magnet alloys, it has not been possible to create the necessary internal strains for secondary recrystallization of their normal polycrystalline structure by known coldworking methods.

By the present invention, I have found it possible, in magnet bodies of the alloys described, to produce by metallurgical means the critical strain conditions necessary for secondary recrystallization phenomena. I have found that by preliminary heat-treatment to produce gamma-phase precipitation in the otherwise alpha-phase micro-structure, and by care to retain such gamma-phase precipitation in the microstructure when the magnet alloy is subjected to recrystallization, the necessary internal strains are produced and are effective to induce development of monocrystalline structure in an originally polycrystalline magnet body.

While the gamma-phase and the resulting strains may be retained by careful and rapid handling of the castings between the two heat-treatment steps, the gamma-phase precipitation can be both accelerated and more-readily retained by introducing into the alloy one or more gamma-phase precipitants. There are elements whose presence has previously been considered deleterious in the heat treatment of anisotropic magnets. Surprisingly, however, they are highly advantageous here, and I have found that after they have served their advantageous purpose, it is possible to avoid their deleterious effect by rapid cooling through the critical temperature range.

I have further found that in the recrystallization heat treatment, the nucleation and development of monocrystalline structure can be guided by practical control of the heating.

The invention in its preferred form involves the following steps:

(1) Forming a magnet body, as by casting, in substantially the usual way, preferably with care to produce a nucleus of properly oriented crystallites at one end face of the magnet body and preferably with a small percentage of a gamma-phase precipitating and stabilizing element in the alloy composition.

(2) Subjecting the magnet body, having an essentially alpha-phase polycrystalline structure, to gamma-phase precipitating temperature, for a limited holding period to produce gamma-phase precipitation and resulting internal strains.

(3) Subjecting the internally-strained polycrystalline structure to a definite recrystallization temperature for a holding period sufficient to redissolve the gamma-phase and to permit large-crystal growth to go to completion in the magnet body.

(4) Establishing a directed temperature gradient in the magnet body during the recrystallization step to select the nucleus and guide the growth direction of the monocrystalline structure, to position its (100) axis in a predetermined relation to the preferred direction of orientation.

(5) Cooling the monocrystalline magnet body rapidly through the gamma-phase precipitation range to avoid gamma-phase contamination in the magnet structure.

(6) Directionally heat treating the magnet body by conventional steps to develop magnetic orientation.

The accompanying drawings illustrate the invention. In such drawings:

FIG. 1 is a diagram illustrating the phase changes of a typical Alnico alloy suitable for application of the invention;

FIG. 2 is a diagrammatic sectional view of a magnet body with a polycrystalline structure, illustrating the normal as-cast condition of a magnet casting; and

FIGS. 3, 4 and 5 are diagrammatic sections of the same body, showing monocrystalline growth in the pre-

ferred direction at successive stages of the recrystallization heat-treatment of the invention.

In the diagram of FIG. 1, the vertical dotted line represents an Alnico alloy of the composition indicated. The diagram shows that this alloy has a pure alpha-structure both above 1200° C. as well as in the range of 900–930° C. To obtain good magnetic properties, this alloy must be magnetically heat-treated, i.e., brought under the influence of the magnetic field while it is in the pure alpha-phase state. Any magnetic heat-treatment from temperatures between 930 and 1180° C. leads to poor results because the structure is not pure alpha-phase in this temperature range. At temperatures between 930° C. and about 1150° C. a second or gamma-phase is precipitated, first at the grain boundaries and then also within the crystals. From about 1175° C. to 1200° C. the gamma-phase again enters into solution. The presence of this gamma-phase in a magnet casting during heat-treatment in a magnetic field has a very marked deleterious effect on the formation of a preferred magnetic orientation. Even the presence of very small amounts of gamma-phase is enough to decrease the magnetic values to such a point that the magnets are useless. A 7% content of gamma-phase will lower energy values by about 25%.

Recent studies indicate that preferred magnetic orientation is the result of the directional precipitation of sub-microscopic particles of a second alpha-phase, referred to as "alpha-prime," at temperatures below 900° C. However, the presence of even minor quantities of the gamma-phase obstructs the proper establishment of a preferred magnetic orientation. I thus concluded that these gamma-phase precipitates can also cause major lattice distortions with associated large internal mechanical strains, and that these would serve to induce large-crystal growth under suitable recrystallization heat treatment.

Example 1

Normal polycrystalline magnet castings were cast in the conventional way from an alloy of the composition given in FIG. 1. These were then heat-treated at temperatures between 930 and 1150° C. in order to induce gamma-phase precipitation and thus create the associated lattice distortions and internal strains. According to the phase diagram for this alloy, the gamma-phase should redissolve above 1200° C. and the structure should revert to pure alpha-phase. However, a certain time interval is required for this solution process and it is therefore possible to raise the magnet casting to the recrystallization temperature range of 1220 to 1320° C., or preferably 1260 to 1310° C., before the solution process of the gamma-phase has gone to completion. Thus, the two requirements for large-grain growth, that is, (a) heat treatment at the critical recrystallization temperature and (b) the presence of internal strains in the crystal lattice, were both fulfilled, and large-crystal growth to a monocrystalline structure was attained.

In carrying out Example 1, it was generally necessary to raise the temperature of the magnet castings very rapidly from the range of alpha-gamma phase stability to the temperature of secondary recrystallization, in order to avoid complete redissolving of the gamma-phase before the critical temperature range for recrystallization was reached. A method was therefore sought to increase the stability of the gamma-phase at temperatures above 1200° C.

Certain elements, which may be referred to as gamma-phase precipitants, are known to widen the gamma-phase loop in the iron phase-diagram, and these include carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold.

Such elements have heretofore been considered deleterious in magnet alloys, and have been carefully avoided or held to low percentages in the alloy composition. I have found, however, that small quantities of these, when added to a magnet alloy composition, will ac-

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celerate the gamma-phase precipitation in the temperature range at which such precipitation occurs, as at temperatures ranging from 930 to 1150° C. Further, the presence of these gamma-phase precipitating elements makes the precipitate more stable at higher temperatures, for example, above 1200° C., so that the gamma-phase precipitate is more readily maintained in the transition to the recrystallization temperature and dissolves more slowly at that recrystallization temperature. With such a gamma-phase precipitate, the internal strains needed for large-grain crystal growth are more easily obtained and are retained for a longer period.

These same elements have previously been considered extremely deleterious in Alnico magnet alloys and extreme caution is commonly exercised to keep these impurities to a minimum. Thus carbon, for example, must normally be held below 0.035%, and preferably below 0.02%, and a carbon content of 0.07% would tend normally to produce very poor magnetic properties. I have discovered the surprising fact, however, that in the present process the addition of these gamma-phase precipitants which are deleterious in prior processes actually facilitates the performance of the process and favors the growth of monocrystalline structure in normally polycrystalline castings. Thus, the presence of these elements, previously considered harmful, actually makes possible a marked improvement in magnetic properties.

Example 2

An alloy composition similar to that indicated on FIG. 1, but with purposely high carbon content, in the range of 0.08% to 0.15%, was used to make conventional polycrystalline magnet castings. These castings were then heat-treated at a gamma-phase precipitating temperature, that is, in the temperature range from 930 to 1150° C., and preferably 1000 to 1050° C., for periods of 30 to 60 minutes to develop a considerable degree of gamma-phase precipitation and resultant internal stresses. The magnets were then rapidly raised to 1280° C. and after a holding period of 30 minutes at this temperature 80% of the magnets were found to have been converted to monocrystalline structure. A somewhat longer holding period and a somewhat higher temperature were needed to achieve the same results for the remaining 20% of the castings. After such heat treatment at the preferred recrystallization temperature range of 1250 to 1310° C. all traces of the gamma-phase precipitate had disappeared and the castings had again attained the desired pure alpha-phase structure conducive to the formation of preferred magnetic orientation by treatment in a magnetic field.

However, the castings must again traverse the two-phase alpha-gamma zone in cooling from the recrystallization temperature, and the presence of the gamma-phase precipitation contaminant will tend to produce and accelerate formation of gamma-phase precipitation at temperatures in this zone. Such precipitation can be avoided, however, by traversing the gamma-phase precipitation temperature range vary rapidly. With small castings, cooling in an air blast was sufficient to prevent gamma-phase precipitation. The cooled magnet castings so produced were of monocrystalline and uncontaminated alpha-phase structure.

To develop anisotropic magnetic properties, the monocrystalline castings were then heated to a maximum of 920° C. and cooled normally in a magnetic field. This was followed by a regular draw treatment at temperatures about 600° C. The following magnetic results were obtained in monocrystalline magnets produced in this manner from normal polycrystalline castings and oriented magnetically parallel to the (100) axis:

Alloy (percentages): Co 24; Ni 14; Al 8; Cu 3; the remainder Fe; with the addition of C 0.08.

Properties: Br=13,800—14,200 gauss; Hc=840—880 oersted; BH_{max}=9.2—10.6×10⁶.

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Example 3

A similar experiment was run with an alloy of the same composition as in Example 2, except that no carbon addition was made and, instead, 0.35% of manganese was added. The process was carried out in the same manner as with the high-carbon alloy, and magnets with monocrystalline structure were again produced, having the following magnetic properties:

Br=13,900—14,000 gauss; Hc=830—865 oersted; BH_{max}=9.8—10.1×10⁶.

Example 4

Additional experiments were conducted with alloys containing additions of other gamma-phase precipitation contaminants listed above, and it was found that such elements have the desired effect of producing conditions to facilitate and enhance the formation of monocrystalline structure in originally polycrystalline castings. My experiments thus indicate that the known class of elements which I use as gamma-phase precipitants are as a class suitable for use in my process.

In order to make best use of the magnetic properties of monocrystalline magnet castings produced as set forth above, it is essential that the (100) axis of the monocrystalline structure have a predetermined relationship, desirably parallel, to the direction of preferred magnetic orientation in the final magnet, and accordingly that the monocrystalline structure in the magnet casting be controlled to occur in such direction, for example, in the direction in which the magnet is to have preferential magnetic orientation. This may be done by providing a properly oriented crystal nucleus in the magnet body and causing monocrystal growth to progress from this nucleus. Experience has shown that even in unchilled normal sand castings, the crystallites lying closest to a surface are oriented perpendicular to such surface. FIG. 2 shows diagrammatically that the crystallites at the top and bottom faces of the rectangular casting 10 have their (100) axis oriented in the desired direction. Assuming that such magnet is to have a lengthwise preferred direction of orientation, crystal growth should be initiated in such a manner that these crystallites at the end faces provide the nucleus for grain growth, because the crystallites on the side walls are oriented at right angles to the desired direction while in the center of the casting there is a completely random arrangement of crystallites.

This selection of a nucleus at an end surface may be accomplished by preferentially heating one or both of the end surfaces during the recrystallization step. For example rectangular or cylindrical castings may be set upright on the hearth of an oven and heated predominantly through such hearth so that heat is principally supplied to the castings through their bottom ends. Thus, in FIG. 3, the magnet casting 10 is shown standing upright on a heating plate 12 which provides the source of heat during the recrystallization step. Other preferential heating methods may also be used, such as high frequency heating of the end surface, or even the application of a simple burner flame to the end surface.

As is indicated in FIGS. 3-5, when the heat is preferentially supplied to one end face of the casting, the large-crystal grain growth is nucleated by properly oriented crystallites at that face, and the monocrystalline structure grows progressively from this nucleus. Such monocrystal growth absorbs and converts to the monocrystalline structure not only the properly oriented crystallites, but the transversely and randomly oriented crystallites as well, until the entire casting becomes substantially a single crystal.

The specific amount of the gamma-phase precipitation element or contaminant used in a particular alloy will vary with processing conditions used in applying the process to the magnet castings made from such alloy. Indeed, it is possible with suitable processing conditions to omit any addition of a gamma-phase precipitant. But this re-

quires exceptional care, and I prefer to use such additions to facilitate practical operations. The amount of gamma-phase precipitation element added should be effective to produce during the precipitation heat treatment step a gamma-phase precipitate which will create the internal strains necessary for monocrystal growth during the recrystallization step. Excessive amounts should of course be avoided because these would tend to produce residual gamma-phase contamination in the desired pure alpha-phase structure of the final recrystallized monocrystalline magnet body. In practice, I desirably use the gamma-phase precipitants in amounts ranging preferably from 0.03% to 1.8%.

The specific temperatures and times used in the heat treatment steps will likewise vary with other conditions but will follow known metallurgical data. Thus, the gamma-phase precipitation step should be carried out at the known gamma-phase precipitation temperature for the type of alloy used, which in the case of the preferred Alnico alloys is in the range of about 930° to about 1170° C., and preferably from about 1000° to about 1050° C. The holding period at these temperatures is preferably from 30 to 60 minutes.

Similarly, the recrystallization step should be carried out at the known recrystallization temperatures for the type of alloy used, which in the case of the preferred Alnico alloys is in the range of about 1220° to about 1320° C., and preferably from about 1260° to about 1310° C. The holding period at these temperatures is preferably from 30 to 60 minutes.

As is shown by the foregoing examples, it is readily possible by this invention to attain magnetic properties and especially $BH_{(max)}$ values substantially beyond those previously obtainable. Thus, whereas practical prior methods gave magnets having $BH_{(max)}$ values not exceeding 8.0×10^6 , it is readily possible by the present invention to obtain $BH_{(max)}$ values ranging upward from 8.5×10^6 .

I claim as my invention:

1. The process of producing monocrystalline structure in a normally polycrystalline magnet body of iron-nickel-aluminum type permanent-magnet alloy, which comprises heat-treating the body at a gamma-phase precipitating temperature to induce gamma-phase precipitation therein, and subjecting the body to heat treatment, with gamma-phase precipitate present in the body in sufficient amount to produce critical strain therein, at a recrystallization temperature for a time sufficient to permit substantial monocrystal growth in the magnet body, and to redissolve the gamma-phase precipitate.

2. The process according to claim 1 with the addition of cooling the body from recrystallization temperature rapidly through the gamma-phase precipitation temperature zone to avoid gamma-phase precipitation in the treated magnet body.

3. The process according to claim 1 with the addition that in the recrystallization heat treatment heat is principally supplied to the magnet body at a predetermined portion thereof to establish a directed temperature gradient in the body for causing the monocrystalline growth to occur from a nucleus in said predetermined portion.

4. The process of producing a magnet body having substantial monocrystalline structure, which comprises forming a magnet alloy melt of the iron-nickel-aluminum type and including therein a gamma-phase precipitant, casting said melt into a polycrystalline magnet casting, heat treating the magnet casting at a gamma-phase precipitation temperature to induce gamma-phase precipitation in the casting, heat-treating the casting containing gamma-phase precipitate in sufficient amount to produce critical strain therein, at a recrystallization temperature for a time sufficient to permit substantial monocrystal growth in the magnet body, and to substantially redissolve the gamma-phase precipitate, and rapidly cooling the heat-treated

casting through the gamma-phase precipitation temperature zone to avoid substantial gamma-phase precipitation in the treated casting.

5. The process according to claim 4 with the addition that the magnet casting has a predetermined face from which heat is withdrawn during solidification of the magnet casting, and in the recrystallization heat treatment the heat is principally supplied to the casting through said predetermined face to establish a directed temperature gradient in the casting for causing monocrystalline growth to occur from a nucleus adjacent said face and in a direction normal to said face, whereby the monocrystalline structure is oriented with its (100) axis substantially normal to said face.

6. The process according to claim 4 in which the gamma-phase precipitant is a member of the class consisting of carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold.

7. The process according to claim 5 in which the gamma-phase precipitant is a member of the class consisting of carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold.

8. The process according to claim 5 with the addition that the magnet is magnetically hardened with its preferred direction parallel with the (100) axis of the monocrystalline structure.

9. The process of producing magnets having substantial monocrystalline structure from an alloy composed substantially of 10 to 30 percent nickel, 6 to 14 percent aluminum, 5 to 42 percent cobalt, up to 8 percent copper, up to 10 percent titanium, with the balance substantially all iron, which comprises casting a normally polycrystalline magnet casting from the alloy, heat treating the casting at a temperature substantially in the range of from 930° C. to 1175° C. to induce gamma-phase precipitation therein, and heat treating the casting containing gamma-phase precipitate in sufficient amount to produce critical strain therein, at a recrystallization temperature substantially in the range of 1220° C. to 1320° C. for a time sufficient to permit substantial monocrystal growth in the magnet body, and to redissolve the gamma-phase precipitate, and cooling the casting from the recrystallization temperature rapidly through the gamma-phase precipitation temperature range.

10. The process according to claim 9 with the addition of including in the alloy from 0.03 percent to 1.8 percent of a gamma-phase precipitant of the class consisting of carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold.

11. A permanent magnet composed of an iron-nickel-aluminum type permanent magnet alloy including at least 0.03 percent of a gamma-phase precipitant, said magnet consisting substantially of a monocrystalline structure of substantially pure alpha-phase (including alpha-prime phase) microstructure, and having a magnetically-hardened direction of preferred orientation substantially parallel with the (100) axis of the monocrystalline structure.

12. A permanent magnet of an alloy of the composition: 10 to 30 percent nickel, 6 to 14 percent aluminum, 5 to 42 percent cobalt, up to 8 percent copper, up to 10 percent titanium, and from 0.03 to 1.8 percent of a gamma-phase precipitant of the class consisting of carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold, with the balance substantially all iron, said magnet consisting substantially of a monocrystalline structure of substantially pure alpha-phase (including alpha-prime phase) microstructure.

13. A permanent magnet of an alloy of the composition: 12 to 20 percent nickel, 6 to 11 percent aluminum, 16 to 30 percent cobalt, 2 to 6 percent copper, up to 7 percent titanium, and from 0.03 to 1.8 percent of a gamma-

phase precipitant of the class consisting of carbon, nitrogen, manganese, ruthenium, rhodium, palladium, rhenium, osmium, iridium, platinum, and gold, with the balance substantially all iron, said magnet consisting substantially of a monocrystalline structure of substantially pure alpha-phase (including alpha-prime phase) microstructure, and having a magnetically hardened direction of preferred orientation substantially parallel with the (100) axis of the monocrystalline structure.

14. A permanent magnet composed of an iron-nickel-aluminum type permanent magnet alloy including at least 0.03 percent of a gamma-phase precipitant, said magnet consisting substantially of a monocrystalline structure of substantially pure alpha-phase (including alpha-prime phase) microstructure, and having a magnetically-hardened direction of preferred orientation substantially paral-

lel with the (100) axis of the monocrystalline structure, and said magnet having in said preferred direction a $BH_{(max)}$ value of at least 8.5×10^6 .

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