

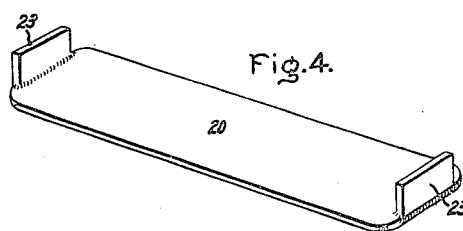
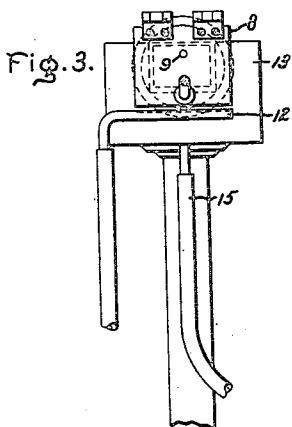
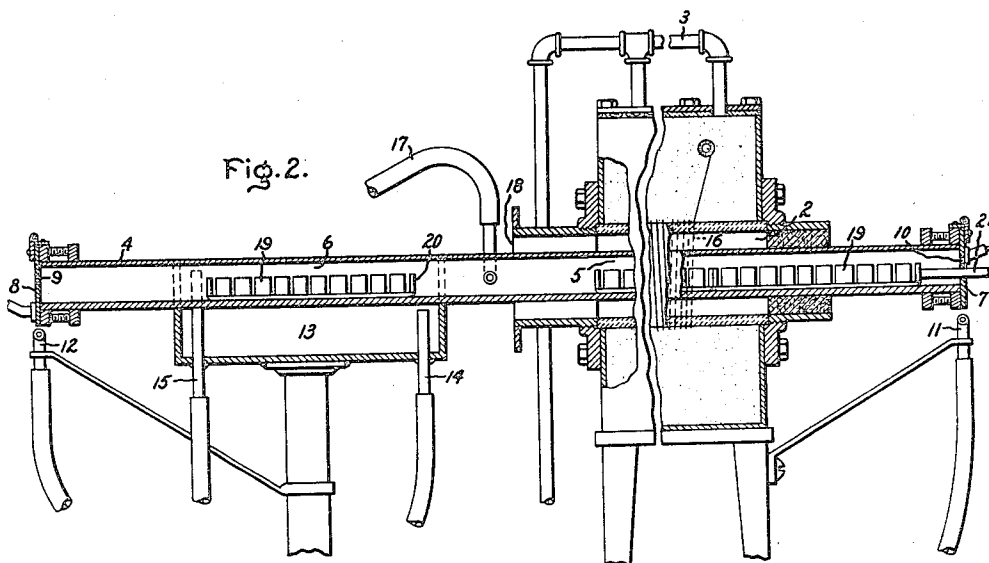
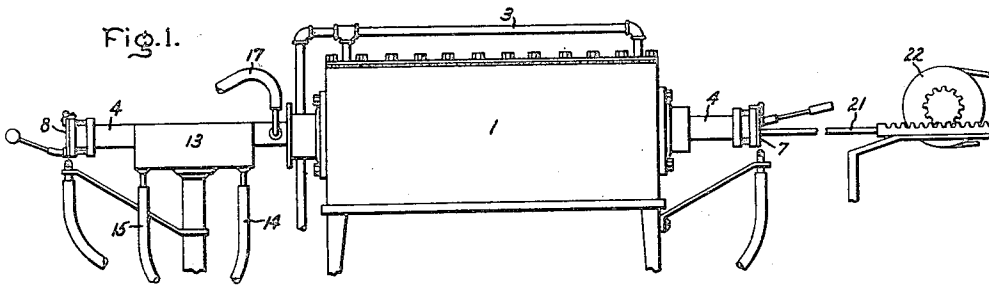
March 5, 1940.

G. H. HOWE

2,192,744

SINTERED PERMANENT MAGNET

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UNITED STATES PATENT OFFICE

2,192,744

SINTERED PERMANENT MAGNET

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Application May 24, 1939, Serial No. 275,551

2 Claims. (Cl. 75—22)

This application is a continuation in part of my copending application Serial No. 196,691, filed March 18, 1938, and entitled Sintered permanent magnet, the latter application being a division of my application Serial No. 164,354, filed September 17, 1937, and entitled Method of making a sintered alloy.

The present invention relates to sintered alloys containing a readily oxidizable element and more particularly to sintered permanent magnets of the type disclosed in Mishima Patents 2,027,994 to 2,028,000 inclusive, and in Ruder Patents 1,947,274 and 1,968,569.

Permanent magnet alloys of the type disclosed in the Mishima and Ruder patents contain iron, nickel and aluminum as the basic or essential ingredients. However, it is well known to include other elements such as cobalt, copper, silicon, titanium, chromium, molybdenum, tungsten and manganese in the alloys if desired and as indicated in the above patents. Preferred permanent magnet compositions are those containing about 6 to 15% aluminum, 17 to 35% nickel with the remainder iron; or 6 to 15% aluminum, 12 to 35% nickel, an appreciable quantity up to 18% cobalt with the remainder iron; or an alloy disclosed in Ruder application Serial No. 96,854, filed August 19, 1936, which may consist of about 14 to 25% nickel, about 8 to 13% aluminum, about 2 to 18% cobalt, about 2 to 16% copper with the remainder iron. In permanent magnets containing iron, nickel, aluminum, cobalt and copper, I prefer to employ a composition containing about 10% aluminum, 17% nickel, 12½% cobalt, 6% copper with the balance iron.

In the manufacture of alloys of the above type, it has been customary to melt the ingredients of the alloy and cast the molten metal in molds. After castings have been made they generally are normalized by heating at a temperature above 1000° C. but not materially higher than 1400° C. after which they are quenched and reheated between about 600° C. and 700° C. to effect ageing or precipitation. Magnets made in accordance with this process have highly desirable magnetic characteristics but are hard and brittle. Such magnets cannot be machined and usually require considerable finish grinding.

If a mixture of powdered iron, nickel, and aluminum comprising ingredients as disclosed in the Ruder and Mishima patents is pressed in a mold and then sintered, it will be found that even when the sintering is carried out in hydrogen which has been purified and dried by means

heretofore employed for that purpose the resulting product is a swollen mass lacking coalescence due to the very pronounced affinity of the finely divided aluminum particles for oxygen.

One of the objects of the present invention is to provide a hard, dense, sintered alloy containing a readily oxidizable ingredient such as aluminum. It is a further object of the invention to provide permanent magnets of the composition disclosed in the above noted Ruder and Mishima patents which will require a minimum of finish grinding, which shall be fine grained, uniform in character and have a high tensile and transverse strength as well as highly desirable permanent magnet characteristics. Other objects will appear hereinafter.

The novel features which are characteristic of my invention will be set forth with particularity in the appended claims. The invention itself, however, will best be understood by reference to the following specification when considered in connection with the accompanying drawing in which Fig. 1 is a view in elevation of a hydrogen furnace which may be employed in carrying my invention into effect; Fig. 2 is a longitudinal sectional view, broken away and on an enlarged scale, of the apparatus disclosed in Fig. 1; Fig. 3 is an end view of the apparatus shown in Fig. 2, while Fig. 4 is a perspective view of a boat employed in the operation of the furnace and adapted to hold the material to be sintered.

In manufacturing sintered magnet alloys containing iron, nickel and aluminum as essential ingredients, the process which I employ may or may not be continuous. In both processes, however, oxidation of the aluminum content of the alloy may be avoided by the use of a foundation alloy. The foundation alloy preferably is a very brittle iron-aluminum alloy which consists of about 50% aluminum and 50% iron and contains all the aluminum present in the final alloy. In making the foundation alloy the necessary quantity of iron, which may comprise several pieces of ordinary melting stock, is heated in a high frequency induction or other suitable furnace to an elevated temperature, for example, a temperature in the neighborhood of 1000° C. This temperature is several hundred degrees below the melting point of iron but is sufficiently high to permit alloyage of aluminum with iron. Aluminum, which likewise may comprise several pieces of ordinary melting stock, is added to the iron to form an alloy consisting substantially of 50% iron and 50% aluminum. The molten alloy is poured into a graphite or other suitable

mold to solidify. The alloy thus produced is very brittle and may be crushed easily, it also remains stable; that is, it does not disintegrate physically when exposed to atmospheric conditions.

5 In fabricating the magnetic alloy the foundation alloy is crushed to the desired extent and added to the other finely divided ingredients present in the magnetic alloy, for example, iron and nickel, or iron, nickel and cobalt, etc., to provide the desired alloy composition. The finely divided materials are mixed, usually in a ball mill, for about five hours and then pressed into a desired shape in a steel mold. The pressed mixture may then be placed in a closed tube having a small outlet opening at one end. The tube is supplied with pure dry hydrogen or other reducing gas, positioned in a hydrogen furnace, and sintered at a temperature above 1000° and preferably at about 1400° C. The time required to complete the sintering action may vary from about one-half to five hours depending upon the load and size of the pieces to be sintered.

After the material has been sintered into a hard, dense mass, it preferably is normalized by heating for about one hour at a temperature above 1000° C. but not materially higher than 1400° C. and preferably at a temperature of about 1050° to about 1100° C. No advantage is obtained by employing a normalizing temperature materially higher than about 1100° C. Any fired furnace may be employed in the normalizing operation since the sintered alloy does not tend to become appreciably oxidized. The alloy may be cooled from the normalizing temperature in still air although, under certain circumstances, the cooling may be accelerated by means of an air blast or slowed down by cooling a plurality of parts in contact with one another. Also parts of small cross sections that would cool too fast in still air can be retarded to a proper rate of cooling by introduction into an air furnace running at about 500 to 600° C.

The copending application of William E. Ruder, Serial No. 758,441, filed December 20, 1934, discloses that for the production of alloys having the best permanent magnet qualities, the rate employed in cooling the magnet material from the sintering or normalizing temperature to about 500° C. will vary with the composition and the size of the magnet alloy pieces; also that the magnetic properties of the finished alloy such as residual and coercive force may be varied by varying the cooling rate. For example, alloys consisting of iron, nickel and aluminum should be cooled from the normalizing temperature to about 500° C. at about 400 to 600° C. per minute and preferably at about 500° C. per minute, while alloys consisting of iron, nickel, aluminum and cobalt should be cooled at a slower rate, for example 175 to 325° C. per minute, and preferably at about 250° C. per minute. The cross section of the magnetic material determines to some extent the cooling rate to be employed. For example, a bar consisting of iron, nickel, aluminum and cobalt about one-quarter inch square preferably should be cooled in still air to obtain the best magnetic qualities, while bars of the same composition and about one-half inch square should be cooled in moving air. If bars of the same composition are about one inch square and over they should be cooled in an air blast or in an air blast moistened with a cooling medium.

The permanent magnet properties of the alloy vary with the rate of cooling from the normalizing temperature. For example, an iron, nickel,

aluminum alloy one-quarter inch by one-half inch in cross section cooled in an air blast at the rate of about 600° C. per minute has a residual of about 6900 and a coercive force of about 445. The same cooling rate applied to an iron, nickel, aluminum, cobalt alloy of the same cross section gives a residual of 8100 but a low coercive force of about 315. The same iron, nickel, aluminum, cobalt alloy of the same dimensions when cooled at a slower rate, as in still air, has a residual of 7800 and a coercive force of about 360. With a further decrease in the cooling rate, for example with two bars in contact with one another, a residual of 7400 and a coercive force of 435 may be obtained. By selecting a proper cooling rate, it is possible to secure a wide range of residual and coercive force, and if residual is more important than coercive force in an iron, nickel, aluminum, cobalt alloy, it should be cooled more rapidly than would be the case if coercive force were the more important.

If desired the alloy may be cooled at the desired rate directly after the sintering operation and the normalizing treatment omitted without impairment of the magnetic properties of the alloy. Such a process however is not as satisfactory economically as the process employing the normalizing step. For example, in cooling the alloy directly from the sintering temperature the container with the sintered material therein may be removed from the furnace and the sintered material taken out of the container while the container is at an elevated temperature. Under such circumstances the interior of the container will become oxidized and the oxide coating must be removed before the tube is again used for the sintering operation. If the container and load therein are removed from the furnace and allowed to cool to room temperature the rate of cooling may be too slow due to the bulk of the container and the load therein. These difficulties are avoided when the normalizing step is employed since there is no danger of oxidizing the sintered alloy. It is preferable therefore always to normalize the alloy unless the continuous process hereinafter disclosed is employed.

If the alloy, after sintering or normalizing, is cooled in air at the proper rate for that alloy the optimum atomic arrangement for best permanent magnet properties is obtained so that reheating of the material at temperatures between about 500° C. and 700° C. at the end of cooling period to effect ageing or precipitation may be omitted if desired.

Although I have referred to cooling in air after the sintering or normalizing treatment, my invention is not limited to cooling in any particular atmosphere. If desired, the cooling action may be carried out in substantially any gaseous medium without adversely affecting the magnetic or physical properties of the material, for example in the cooling chamber of the ordinary hydrogen furnace. Where I employ the expression "cooling in air" in the specification or claims, I have reference to a type of cooling rather than to cooling in a certain gaseous medium.

The sintered alloy containing iron, nickel, and aluminum as basic ingredients may be machined. However, in general, it will be found easier to carry out any desired machining operation either on the material as pressed and before sintering, or on the pressed and partly sintered material. In the latter case the material which has been mixed and pressed, as heretofore pointed out, is heated at a temperature of about 600° C. for

about one hour in a hydrogen or other suitable reducing atmosphere to effect partial sintering. It is then cooled rapidly, preferably in the cooling chamber of the furnace, to prevent oxidation of the iron, nickel and aluminum.

In the partial sintering operation, it is not necessary to employ a pure dry hydrogen atmosphere. Ordinary line hydrogen, which contains some moisture, may be employed since the ferro-aluminum foundation alloy employed in making the magnetic alloy is not oxidized in such an atmosphere at 600° C. Material which has been partially sintered may be machined readily. Moreover, the partial sintering action does not adversely affect the magnetic properties of the finished magnets. Material which has been partially sintered and then machined should, of course, be sintered thereafter at a temperature above 1000° C. and preferably at about 1400° C. and then cooled at the proper rate, or reheated to effect precipitation, as hereinbefore indicated.

In quantity production of permanent magnet alloys of the Ruder and Mishima type, I prefer to employ a continuous process in which sintering and precipitation may be effected without any reheating. In carrying out my improved process, I may employ a modified form of hydrogen furnace 1. The furnace comprises the usual heating chamber 2, supplied with hydrogen gas through the pipe 3. An elongated rectangular shaped steel tube 4, which extends through and beyond each end of the heating chamber of furnace 1, comprises a heating or sintering chamber 5 coextensive with the heating chamber 2, and a cooling chamber 6 which forms an extension of the sintering chamber 5. The inlet and outlet ends of tube 4 are provided with doors or closure members 7 and 8 respectively. Outlet door 8 is provided with an opening 9 which in the present case is about .040" in diameter, while the inlet door 7 is provided with an opening 10 about .052" in diameter. Slotted pipes 11 and 12, positioned beneath these doors, supply a curtain of hydrogen over the ends of the tube 4 and prevent access of air, particularly by convection, to the interior of the tube when either door is opened. The portion of the tube 4 comprising the cooling chamber 6 is cooled by a water jacket 13, having an inlet pipe 14 and an outlet pipe 15 for the circulation of a cooling medium therethrough.

In operation the tube 4 is heated to a temperature above 1000° C. and preferably at about 1400° C. by means of the usual heater coil 16. Hydrogen, which preferably has been purified and dried to remove moisture therefrom, is supplied to the tube at a point between the heating and cooling zones through a pipe 17. As the hydrogen enters tube 4 it divides. One part flows towards the outlet opening 9, while the remainder and greater portion flows towards and out of the larger opening 10 in the closure member 7. Ordinary line hydrogen may be supplied to the heating zone 2 of the furnace 1 through pipe 3. This hydrogen is burned as it emerges from the opening 18, while the hydrogen in tube 4 is burned as it emerges from openings 9 and 10. The pieces of material 19 to be sintered are positioned on metal boats 20 which are pushed slowly through tube 4 by means of a rod 21 operated by a driving device 22.

That portion of the hydrogen in tube 4 which flows towards the opening 10 comes in contact with the sintered pieces emerging from the hottest zone in the furnace. At this temperature,

about 1400° C., the purified hydrogen is further purified by the removal of substantially all traces of oxygen therefrom. No reaction to form water takes place, however, as the surface of the sintered material, in this case, permanent magnet material containing as essential ingredients, iron, nickel, and aluminum, is practically inert to reduction after oxidation, due to the aluminum content of the alloy. As the purified hydrogen reaches the point where the pressed material 19 enters the heating zone of the tube 2, it attains an unusually high degree of purity and protects the pressed material against oxidation and also reduces any trace of oxide on the pressed pieces 19.

In a 4' furnace of the type disclosed and having outlets as designated, the magnetic properties of sintered alloy are progressively improved as the hydrogen flow is increased from about 4 to 10 cubic feet per hour. Although freshly hydrogen cleaned, powdered materials are employed in fabricating the alloys, some degree of oxidation of the powdered materials occurs during mixing and pressing. However, since the relatively large hydrogen discharge opening is at the inlet end of the tube 4, the hydrogen flow is in the proper direction to maintain an extremely high degree of purity at the inlet point of tube 4 where the demand is most critical.

The time required for a boat 20 to pass through the heating zone in the tube 4 usually varies from about one-half to two hours depending upon the size of the pieces to be sintered. The temperature of the hot zone grades off from about 1400° C. at the center to about 1050° C. to 1150° C. at the end adjoining the cooling chamber. As a boat approaches the end of the hot zone 5 the outlet door 8 is opened and the operator inserts a rod and pulls the boat quickly into the cooling chamber 6, where the sintered material is permitted to cool below 600° C. The cooling period, usually varies from about 10 to about 30 minutes according to the composition, size of pieces 19, and magnetic properties desired. The cooling rate may be controlled by varying the rate of flow of the cooling medium in water jacket 13.

The material carried on the fore-end of the first boat load which is passed through the sintering chamber may under some conditions be oxidized. To avoid any spoilage of good material, it is advisable to push through the tube 4, a preliminary load of scrap material which will serve to clean up the hydrogen in preparation for succeeding loads.

The atmospheric conditions in the hot section of the sintering section of tube 4 are ideal for causing sticking of the boats to the tube 4 owing to the reducing action of the hydrogen and the high temperature employed. To avoid such action a composite type of boat 20 may be employed. In constructing the boats, iron powder is mixed with a smaller quantity of aluminum oxide powder, for example about 75% iron and about 25% aluminum oxide and spread into a relatively thin even layer having the surface dimensions desired for the finished part. A second and thicker layer of powdered iron is then superimposed on the first mentioned layer and the two layers pressed together in a mold and then sintered for about two hours at about 1450° C. Two or more metal pieces thus fabricated may be readily welded together, owing to the presence of the iron layer, to provide boats of any desired width. End pieces 23 having the oxide-iron mixture on the outside are welded to the base portion of the boat. A boat may be pressed

entirely of the 25 Al_2O_3 -75 Fe mixture but such material cannot be welded and unless a large enough mold is available to press a complete boat it is necessary to use pieces of composite construction having weldable material on one side. Another advantage in using a composite boat is that any deformation of the boat due to alternate heating and cooling may be removed by cold pressing without danger of breakage or oxidation.

I prefer to employ a mixture of about 75% iron and 25% aluminum oxide in the construction of the boat 20 but these percentages are not critical and may be varied to a considerable extent. Furthermore, I may, if desired, employ in the construction of the boats a mixture of aluminum oxide or any other inert oxide with metals other than iron. For example, if unusually high temperatures are to be employed in the sintering operation, metals such as tungsten and the like may be employed to replace the iron in whole or in part.

Although I prefer to make sintered material in accordance with the process hereinbefore set forth in which a foundation alloy of 50% iron and 50% aluminum is first produced and thereafter pulverized and mixed with the desired quantity of iron and nickel or other powdered ingredients of the alloy and then pressed into shape, it is possible by the use of certain precautionary measures to avoid the formation of a foundation alloy of iron and aluminum and to make the finished product by mixing powdered iron, nickel and aluminum in the desired proportions. These precautionary measures comprise the use of very pure ingredients, particularly pure iron; the use of a very fine and dense aluminum; the proper control of gas flow; and the use of hydrogen having a degree of purity substantially equal to that of the hydrogen flowing through the hot zone of the tube 4 in the above continuous process. The product obtained by mixing and pressing powdered iron, nickel, and aluminum, or other similar mixtures containing aluminum, and thereafter sintering in a hydrogen atmosphere is substantially as good magnetically and in appearance as that obtained when a foundation alloy of iron and aluminum is employed in fabricating the alloy.

The finished sintered alloy has permanent magnet properties equal to those of an alloy of the same composition made by casting and heat treating. In addition, the sintered alloy is fine grained and has tensile and transverse strengths which are respectively ten and five times greater than the tensile and transverse strengths of cast alloys of the same composition. The sintered alloy requires a minimum of grinding which may

be carried out without any danger of cracking the alloy. Furthermore, if desired, solid or hollow inserts may be made in the pressed material. Under such circumstances however, the holes drilled in the pressed material should be slightly larger than the inserts to thereby prevent cracking or distortion when the material is sintered. The sintered product made by the continuous process has the additional advantage of having a very bright finish.

In fabricating the sintered magnetic alloy, the mixed ingredients usually are subjected to a pressure of about 10 to 30 tons per square inch. Material which has been subjected to such pressure shrinks about 6 to 14% during the sintering operation, the greater shrinkage occurring with material pressed at the lower pressures. Notwithstanding such shrinkage, the sintered alloy can be made to very close tolerances, i. e., plus or minus five mils. Sintered magnets have an additional advantage in economy of material due to the fact that there is no waste material such as the gates and risers which are formed during the process of manufacturing cast materials.

Although the alloy material preferably is shaped by pressure in a steel mold, it also may be formed in some cases by extruding the mixed ingredients with a binder material of which starch is an example. The extruded material may then be heat treated as hereinbefore set forth.

While I prefer to employ a foundation alloy consisting of about 50% iron and about 50% aluminum, other percentages of iron and aluminum may be employed in the fabrication of the foundation alloy. Also, the foundation alloy may be formed by alloying the aluminum with either cobalt or nickel, or with iron and cobalt in various percentages. However, although various foundation alloys containing the aluminum may be employed, the alloy consisting of 50% iron and 50% aluminum is, in general, the most satisfactory owing to the low temperature at which it can be manufactured and the ease with which it may be crushed.

What I claim as new and desire to secure by Letters Patent of the United States, is:

1. A sintered, machinable, permanent magnet containing 14% to 25% nickel, 8% to 13% aluminum, 2% to 18% cobalt, 2% to 16% copper with the remainder substantially all iron, said magnet being characterized by its high transverse strength as compared with the transverse strength of cast material of the same composition.

2. A sintered permanent magnet containing about 10% aluminum, 17% nickel, 12½% cobalt, 6% copper and balance iron.

GOODWIN H. HOWE.