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## MAGNETIC ALLOY PARTICLE COMPOSITIONS AND METHOD OF MANUFACTURE

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8 Claims

### ABSTRACT OF THE DISCLOSURE

This invention relates to the preparation of finely  
divided particles and especially to the preparation of high  
coercivity, finely divided magnetic alloy particles by re-  
duction of salts of cobalt, and mixtures of salts of cobalt  
and iron, mixtures of salts of cobalt and nickel, in a bath  
containing hypophosphite and amine borane anion re-  
ducing agents. Sodium hypophosphite and dimethyl amine  
borane or any soluble salts which provide hypophosphite  
and amine borane anions in solution are reacted with  
soluble metal salts of cobalt alone, or cobalt with iron,  
or nickel dissolved in the bath. Precipitation of finely di-  
vided metal-phosphorus-boron alloy particles is brought  
about by the reduction of metal cations by the hypo-  
phosphite and amine borane anion reducing agents. After  
separating and drying, the precipitate is found to consist  
of non-pyrophoric magnetic alloy particles, including  
about 0.1–5% phosphorus and about 0.1–1.5% boron,  
which are generally spherical in shape and which vary  
in size from about 0.01 micron to 3.0 microns in diameter.

This is a division of application Ser. No. 812,433, filed  
Apr. 1, 1969, now abandoned.

### BACKGROUND OF THE INVENTION

#### Field of the invention

This invention relates to magnetic alloy compositions  
and to a novel method for preparing finely divided mag-  
netic alloy particles. Such particles are suitable for use in  
magnetic recording media, permanent magnets, magnetic  
cores, and in magnetically responsive fluid suspensions,  
such as magnetic or electrostrictive clutch couplings or  
the like.

#### Description of the prior art

In the prior art, magnetic particles of the free metal,  
alloy and oxide type, have been prepared in numerous  
ways. In the common type of preparation, cobalt, iron,  
and nickel compounds are prepared, often by chemical  
precipitation, and then decomposed, oxidized, and/or re-  
duced to produce either oxide, metal, or alloy magnetic  
particles. In another type of preparation, solutions of  
cobalt, iron, or nickel salts are subjected to reduction at  
the cathode of an electrolytic cell to produce continuous  
magnetic films or particles. In yet another technique,  
solutions of cobalt, iron, and nickel salts are subjected  
to chemical reduction by the action of a reducing agent  
on the metal cations. In the prior art, such chemical or  
electroless reduction procedures have most often been  
carried out to produce continuous films or coatings. In  
such electroless plating procedures, reducing agents have  
commonly been of the hypophosphite, boron-nitrogen,  
borohydride, or organic formate type. It has been ob-  
served that in such electroless film plating procedures the  
plating bath is sometimes subjected to catastrophic de-  
composition, whereby a large portion of the metal cation  
content of the solution is vigorously and quickly reduced.  
The resulting deposited material is normally a mixture

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of film and particles covering a wide range of sizes and  
shapes. It has been determined that such unwanted cata-  
strophic decomposition during film plating is usually  
brought about by a combination of excessive heating of  
the electroless solution, a change in pH, the build up of  
nucleating material, such as insoluble salts, or the addi-  
tion of catalytic material to the bath. Since the material  
plated in an electroless bath is itself autocatalytic to the  
decomposition reaction, once uncontrolled decomposition  
begins, it increases in an avalanching manner, so that  
plate-out of the bath is accomplished in a very short  
time.

As has been already noted, electroless plating baths  
have been most often used in the prior art to produce  
continuous films. Development of related technology has  
been heavily aimed at achieving means to avoid cata-  
strophic decomposition. In the few instances where elec-  
troless baths have been used to intentionally produce  
particles, finely divided particles having uniform size and  
good magnetic characteristics have been produced only  
by initiating the decomposition reaction with catalytic  
metals or their salts, while utilizing temperature, pH, and  
concentration parameters to vary the physical properties  
of the particles. The catalytic material most often used  
for initiating controlled chemical reduction of magnetic  
metal salts to form particles has been palladium and its  
salts. In view of the high cost and limited availability  
of palladium and its salts, it is desirable to have other  
techniques for producing uniform, finely divided magnetic  
particles by chemical reduction. The present invention  
provides a highly effective technique for producing such  
finely divided magnetic particles without utilizing cata-  
lytic materials to initiate or control the reaction. As a  
result of the technique employed, unique magnetic alloys  
are also provided.

### SUMMARY OF THE INVENTION

It is an object of the present invention to provide new  
and improved techniques for manufacturing finely di-  
vided magnetic alloy compositions.

Another object of this invention is to provide a unique  
metal-phosphorus-boron alloy composition in finely di-  
vided form having magnetic properties suitable for use  
in magnetic recording media, permanent magnets, mag-  
netic cores, and in magnetically responsive fluid suspen-  
sions.

The present invention provides a new finely divided,  
non-pyrophoric, ferromagnetic composition in the form  
of microscopic particles consisting essentially by weight  
of 0.1–5% phosphorus, 0.1–1.5% boron, 0.8–10% oxy-  
gen, and the balance cobalt, cobalt-nickel, or cobalt-iron.

The present invention also relates to a method of mak-  
ing finely divided magnetic alloy particles by dissolving a  
metal salt of cobalt, or mixtures of cobalt and iron, or  
cobalt and nickel salts in a bath, preferably aqueous, and  
reducing the metal salts with sodium hypophosphite and  
dimethylamine borane or other sources of hypophosphite  
and amine borane anions which dissolve in the bath,  
thereby precipitating alloy particles by chemical oxida-  
tion reduction to produce metal-phosphorusboron par-  
ticles of spherical structure in a narrow range of particle  
sizes varying between about 0.01 to 3.0 microns.

In preparing the reaction mixture, any soluble cobalt,  
iron and cobalt, or nickel and cobalt salts may be dis-  
solved in a bath with any soluble source of hypophosphite  
anion and heated. A separate solution of amine borane  
anion is prepared. Upon mixing these separate solutions,  
a clear solution is initially formed. Reduction and pre-  
cipitation are effected spontaneously after a short time,  
with or without continued heating. In an alternative  
technique for producing alloy particles, a heated solution

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of metal salts, including any soluble source of amine borane, may have added thereto a solution containing sodium hypophosphite or any soluble hypophosphite salt dissolved therein. Yet another technique for producing alloy particles is the preparation of a solution of hypophosphite and amine borane anions to which a solution of soluble cobalt, iron, or nickel salt is added. Precipitated magnetic particles are separated by filtering, decanting, centrifuging, magnetic separation, or any other suitable means.

Spherical cobalt-phosphorus-boron, cobalt-iron-phosphorus-boron, or cobalt-nickel-phosphorus-boron alloy particles are formed by these reactions.

Alloy particles produced in accordance with this invention display intrinsic coercivities up to 600 oersteds and more. The saturation magnetization per gram,  $\sigma_s$ , ranges up to 161 electromagnetic units per gram, and the squareness ratio  $M_r/M_s$  is in the range of about 0.16 to 0.46. They are in the form of finely divided spherical particles about 0.01 to 3 microns in diameter, with the vast majority between 0.01 to 3 microns in diameter, with the vast majority between 0.01 and 1.0 micron in diameter. After formation in solution, the particles tend to form both agglomerates and chains.

The foregoing and other objects, features, and advantages of the invention will be apparent from the following more particular description of preferred embodiments of the invention.

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the following examples, all solutions were prepared with distilled water and reagent grade chemicals. Unless otherwise clearly indicated, the total volume of the reaction mixture was approximately one liter. In order to bring the solutions together rapidly and completely agitation via a magnetic stirring bar was employed. Particles produced by the method of the present invention were separated from the reaction mixture, usually magnetically, and washed with water and acetone. The particles were then dried, usually under non-oxidizing conditions. While precautions were taken to avoid exposing the particles to oxygen, prior to and during drying, the resulting particles exhibited from about 0.8 to 10% oxygen content, by weight. In most instances the oxygen content was less than 2% by weight of the alloy and was limited almost entirely to the skin or shell of the particles.

Powder samples of the alloys produced by the present invention were measured with a vibrating sample magnetometer, VSM, to determine their magnetic properties. Determination of the chemical content of the alloy particles was obtained by both X-ray fluorescence and neutron activation. Particle size and shape was determined from electron micrographs of the particles.

While the products of the present invention consist predominantly of cobalt, nickel, iron, and their alloys, there is associated therewith small, but significant quantities of phosphorus, boron, and oxygen, as indicated by analysis. It would appear that during the course of reduction of the metal cations to metal, a small amount of the phosphorus in the hypophosphite anion and the boron in the amine borane anion is oxidized to the neutral states. The resulting phosphorus and boron formed thereby, is co-precipitated with the reduced metal to form an alloy. It further appears that during the washing and drying steps of the method, some small degree of oxidation of the surfaces of the particles occurs with the result that the final product contains oxygen.

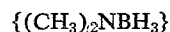
All alloy compositions in the examples are given in weight percent.

#### EXAMPLE I

An aqueous solution containing 10 g. cobalt sulfate ( $\text{CoSO}_4 \cdot 2\text{H}_2\text{O}$ ), 10 g. sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) and 20 g. sodium hypophosphite ( $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ ) in 800

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ml. of water was prepared and heated to 95° C. A separate solution of 10 g. dimethylamine borane



in 60 ml. water was prepared. The dimethylamine borane solution and 125 ml. of 29% ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) were then poured into the cobalt bath with magnetic stirring. A clear solution, without any precipitate or noticeable reaction was formed. After approximately three minutes, with continued heating and stirring, a vigorous reaction took place and a black, finely divided precipitate was formed. This precipitate was washed thoroughly with water and then with acetone, and dried in the absence of air. The resulting particles were packed in a glass cylinder for measurement of magnetic properties by the VSM. The saturation magnetization per gram or sigma value was 114 emu/g. at 4000 oersteds, and the intrinsic coercive force was 437 oersteds. Electron micrographs of the powder indicated that it consisted of spherical particles, 0.01 to 1 micron in diameter. Analysis indicated that the particles consisted essentially by weight of 1.5% phosphorus, 0.1% boron, less than 5% oxygen, the oxygen being limited almost entirely to the surface of the particles, and the balance cobalt.

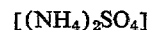
A second preparation of this product was carried out in precisely the same manner, and the resulting particles exhibited a sigma value of 115 emu./g. and a coercivity of 490 oersteds. The particles consisted of 1% phosphorus, 0.1% boron, less than 5% oxygen, and the balance cobalt.

The preparation of particles was repeated once more, as described above, with the exception that the temperature of the bath was held at 90° C. Particles began forming in the bath approximately 120 to 140 seconds after the mixture of the dimethylamine borane solution and the cobalt bath and continued to form for 8 minutes thereafter. After particle formation had ceased and the particles were removed from the solution, it was noted that the supernatant liquid was colorless, and without the pink coloration normally attributed to cobalt salts. Spectral analysis was performed on the depleted solution. No absorbance peaks were evident for the divalent cobalt amine complex, the trivalent cobalt amine complex, or the divalent cobalt citate complex. As these complexes are known to be quite stable, evidence of their absence in the solution, as well as the colorless nature of the solution was taken as a strong indication that substantially all of the cobalt cations had been removed from solution by the reaction of this invention. Analysis of the particles formed in the latter experiment indicated a sigma value of 84 emu./g., and a coercivity of 425 oersteds. The particles contained about 96.6% cobalt, 2% phosphorus, 0.2% boron, and 1.2% oxygen. They were spherical and ranged in size from 0.1 to 3 microns in diameter.

#### EXAMPLE II

In view of the outstanding cobalt yield indicated by the previous experiments, it was decided to prepare cobalt particles by the classical method of palladium seeding, and then add amine borane to the remaining cobalt cations and hypophosphite anion in what would normally be considered a spent bath by the prior art.

To a one liter bath containing 35 g.  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , 35 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ , 66 g. ammonium sulfate



20 g.  $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$  and 100 ml. of 29%  $\text{NH}_4\text{OH}$ , and heated to 88° C. in a resin kettle, was added approximately 0.1 g.  $\text{PdCl}_2$ . An instantaneous and vigorous exothermic reaction took place, causing a finely divided black material to be precipitated in the bath. The reaction was allowed to proceed to completion while maintaining the temperature of the bath at 88° C. As a matter of interest, the powder formed was analyzed and found to

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be a cobalt-phosphorus alloy containing about 0.6% phosphorus and having a sigma value of 99 emu/g. and an intrinsic coercivity of 486 oersteds.

After all traces of the magnetic material produced by palladium seeding were removed from the bath, the supernatant liquid was poured into a two-liter beaker and once more heated to 88° C. To this spent bath was then added 2 g. of  $(\text{CH}_3)_2\text{NBH}_3$  as well as an additional 50 ml. of 29%  $\text{NH}_4\text{OH}$  to assure basic solution. Upon addition of the dimethylamine borane, a turbulent reaction once more took place with the formation of additional finely divided black particles. The particles formed in this latter reaction were collected magnetically, washed with water and acetone and dried at 60° C. in a vacuum oven. They were found to be cobalt-phosphorus-boron alloy, containing 0.5% phosphorus and 0.2% boron, and having a sigma value of 106 emu/g. and an intrinsic coercivity of 622 oersteds.

#### EXAMPLE III

In order to determine what effect the source of hydroxide ions had, if any, on the reaction, sodium hydroxide was substituted for ammonium hydroxide in the reaction.

A one liter bath containing 35 g.  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , 35 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ , and 20 g.  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$  was heated to 65° C. and brought to a volume of one liter by the addition of 200 ml. of 1 N NaOH and 75 ml. of a solution of 10 g.  $(\text{CH}_3)_2\text{NBH}_3$ . The latter two solutions had been heated to 65° C. prior to adding them to the cobalt solution. A blue flocculate, believed to be cobalt hydroxide, formed in the bath, but began to dissolve after approximately 4 minutes. As the flocculate dissolved, a black precipitate formed in the bath, with this latter reaction becoming more vigorous as it proceeded. The resulting product was found to have a sigma value of 118 emu/g. and an intrinsic coercivity of 75 oersteds. It consisted of 0.5% phosphorus, and 0.5% boron, with the balance cobalt.

#### EXAMPLE IV

The formation of cobalt-nickel alloy by the method of the present invention is of interest.

To 800 ml. of hot, distilled water was added 8 g.  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , 2 g. nickel sulfate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), 10 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$  and 20 g.  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ . To this bath was added a 90° C. solution of 10 g.  $(\text{CH}_3)_2\text{NBH}_3$  in 50 ml. of water. The bath temperature was maintained at 92° C. for 4 minutes without the formation of any precipitate. At this point 15 to 20 ml. of 29%  $\text{NH}_4\text{OH}$  was added to the solution. An immediate and violent reaction took place causing the formation of finely divided black particles and much heat. The reaction proceeded to completion quite rapidly with apparent good efficiency. The resulting spherical particles were about 0.01 to about 0.1 micron in diameter and exhibited a sigma value of 50 emu/g. and an intrinsic coercivity of 249 oersteds as measured on the VSM. The particles were determined to contain a ratio of cobalt to nickel of 2.15:1 as well as approximately 1% by weight of oxygen; the oxygen being concentrated almost entirely in the shell of the particles. Exclusive of oxygen, the alloy particles consisted of 66.5% cobalt, 31% nickel, 2% phosphorus, and 0.5% boron, by weight.

#### EXAMPLE V

The formation of iron-cobalt alloys by the method of the present invention is also of interest.

To avoid the formation of interfering iron oxides or hydroxides during the reaction, 800 ml. of water was purged of oxygen by the process of bubbling nitrogen through the water for an extended period of time. To 800 ml. of this purged water was added 4 g.  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , 75

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6 g. ferrous sulfate ( $\text{FeSO}_4$ ), 10 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ , and 20 g.  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ , without reaction. The bath was heated to 95° C. at which time 125 ml. of 29%  $\text{NH}_4\text{OH}$  and 10 g.  $(\text{CH}_3)_2\text{NBH}_3$  were added to the bath. The temperature of the bath was maintained at 95° C. for 5 minutes without any reaction occurring. At this time an additional 125 ml. of 29%  $\text{NH}_4\text{OH}$  was added to the bath along with an additional 10 g. of dimethylamine borane. A mild reaction was noted in the bath which continued for about 20 minutes with the resulting formation of finely divided black particles. The particles were recovered from the bath with a magnet, washed with water and acetone and dried. By electron microscopy it was determined that the particles thus formed were spherical and had diameters on the order of 0.01 to 0.1 micron. Examination of a powder sample by the VSM indicated a sigma value of 161 emu/g. and an intrinsic coercivity of 103 oersteds. Exclusive of oxygen, the alloy was determined to contain 70.9% cobalt, 28% iron, 1% phosphorus and 0.1% boron, by weight.

#### EXAMPLE VI

A nickel alloy containing 5% phosphorus and 1.5% boron was prepared by dissolving 11 g.  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , 10 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ , and 20 g.  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$  in 800 ml. of hot distilled water. To this solution was added a hot solution containing 10 g.  $(\text{CH}_3)_2\text{NBH}_3$ . The entire bath was maintained at 95° C. In less than 60 seconds a vigorous reaction began which resulted in the formation of finely divided black spherical particles which were determined to have a diameter of about 0.1 micron. X-ray analysis indicated a large percentage of nickel oxide throughout the particles, with oxygen constituting about 10% by weight of the particles formed.

#### EXAMPLE VII

The formation of particles of non-ferromagnetic metal alloys by the method of the present invention is of interest. Preparation of copper alloys by the method of this invention was successfully carried out.

To 800 ml. of hot, distilled water was added 9 g. copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), 10 g.  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ , and 20 g.  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ . To this bath was added a hot solution containing 10 g.  $(\text{CH}_3)_2\text{NBH}_3$ , and the entire mixture was maintained at 80° C. Almost immediately a vigorous reaction began which resulted in the formation of finely divided particles. The resulting copper alloy particles included about 0.1% boron and 0.1% phosphorus. A large amount of oxygen also combined with the particles and CuO was detectable. These particles are suitable for use as catalysts in organic reactions and may be blended with binder material in the formation of conductive matrices.

Proportions of the reactants in the foregoing examples can be varied considerably. Concentrations may range up to saturation. However, solutions of lower concentration have been found to be suitable.

The process of this invention is normally carried out under atmospheric conditions. However, moderate variations in pressure, for example, from 0.5 to 5 atmospheres may sometimes be desirable.

While a convenient method for carrying out the process of this invention is to place solutions of salt in a suitable container, such as glass, resin, or stainless steel, the invention may easily be modified for continuous operation. Reactants may be introduced into a reaction vessel or tube in appropriately proportioned quantities, and the reaction mixture, including the reaction products, continuously withdrawn. With this latter type of operation, much larger quantities of reactants can be efficiently and conveniently processed.

Any soluble salt of cobalt, iron, or nickel may be used, and the halides, nitrates, sulfates, and acetates are representative salts which are readily available and have been

used with both good results. Soluble salts of other metals which are reducible to metal in solution may also be utilized in accordance with the teachings of this invention.

For reasons of economy and availability, dimethyl amine borane and sodium hypophosphate are the preferred sources of amine borane and hypophosphite anions. However, other soluble amine boranes and hypophosphites may be used. All of the alkali metal hypophosphites are suitable sources of hypophosphite anion. Other sources of amine borane include, for example, ammonia borane, monomethylamino borane, ethylamine borane, tertiary propylamine borane, and isopropylamine borane. Other boron-nitrogen reducing agents may be substituted for amine borane materials. These include borozanes, in which the amine boranes are encompassed, borozenes, borazines, and borazoles.

While water is a convenient medium for carrying out the process of this invention, other media, including organic liquids, and especially water-miscible organic liquids can be used.

The use of buffering materials, complexing materials, and pH controls constituents in the reaction bath is a matter of technical choice. As is well known, these materials, and the techniques of using them, control the availability of various ions as well as the formation of interfering oxides and hydroxides in the bath.

During the reduction precipitation step, which follows the combination of hypophosphite and amine borane reducing agent, and metal salt in the bath, it may be advantageous to employ an ultrasonic field which aids in forming alloys having a very fine and uniform particle size range, which, in turn, leads to superior magnetic results.

The ultrasonic field may be generated by commercially available devices which vibrate a blade at a high frequency, or by piezoelectric crystal transducers (e.g., quartz, barium titanate, and the like) which convert electric energy into ultrasonic waves between 10 kc. p.s. and 1 mc. p.s.; or by other transducers which are described in the literature and known in the art. Low intensities of the order of 0.1-0.7 watt per square centimeter of ultrasonic energy are generally adequate to disperse the precipitate and prevent particle agglomeration by vibrational motion in the bath.

An external magnetic field effecting the reaction mixture during the formation of the precipitate can be used to enhance the character of the particles formed, but it is not an essential feature of this invention. Fields of A.C. or D.C. magnetization of as much as 1000 oersteds, and more, can be used to advantage.

Uses for the materials produced in the foregoing example are well known. The ferromagnetic alloy particles produced by the foregoing examples may be coated with non-magnetic organic film-forming materials to inhibit agglomeration. These coating materials may be organic polymers or non-magnetic fillers which have known utility in the preparation of magnetic recording media and magnetic responsive fluids, such as are used in the electromagnetic clutch of Rabinow, U.S. Pat. 2,575,360, or the electrostrictive fluid compositions of the type shown in Winslow, U.S. Pats. 2,417,850 or 2,886,150.

Typical, but not limiting, binders for preparing various recording media including ferromagnetic particles produced in accordance with this invention are polyesters, cellulose esters and ethers, vinyl chloride, vinyl acetate, acrylate and styrene polymers and co-polymers, polyurethanes, polyamides, aromatic polycarbonates and polyphenyl ethers.

A wide variety of solvents may be used for forming a dispersion of the fine ferromagnetic particles and binders. Organic solvents, such as ethyl, butyl, and amyl acetate, isopropyl alcohol, dioxane, acetone, methylisobutyl ketone, cyclohexanone, and toluene are useful for this purpose. The particle-binder dispersion may be applied to a suitable substrate by roller coating, gravure coating, knife coating, extrusion, or spraying of the mixture onto

the backing or by other known methods. The specific choice of non-magnetic substrate binder, solvent, or method of application of the magnetic composition to the support will vary with the properties desired and the specific form of the magnetic recording medium being produced.

In preparing recording media, the magnetic particles usually comprise about 40-90% by weight of the solids in the film layer applied to the substrate. The substrate is usually a flexible resin, such as polyester or cellulose acetate material, although other flexible materials as well as rigid base materials are more suitable for some uses.

In preparing magnetic cores and permanent magnets, the products of the examples are mixed with non-magnetic plastic or filler in an amount of about 33-50% by volume of the finished magnetic metal; the particles aligned in a magnetic field; and the mixture pressed into a firm magnet structure. Alignment of the particles may be accomplished in an externally applied DC magnetic field of about 4000 gauss, or more, and fields up to 28000 gauss may be used. Pressure may vary widely in forming the magnet. Pressures up to 100,000 p.s.i. have been used commercially.

Modification of cobalt salts with iron, as taught in Example V, is useful for producing particles from which magnets or cores can be made, since the magnetization of an iron-cobalt alloy particle is dependent on (a) the ratio of the iron to cobalt, and (b) the unit particle size (see the Japanese Journal of Applied Physics, vol. 6, No. 9, pp. 1096-1100, September 1967).

While the invention has been particularly shown and described with reference to preferred embodiments thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of the invention.

What is claimed is:

1. A method for preparing alloy particles by reduction comprising: reacting a solution consisting essentially of hypophosphite and amine borane anion reducing agents, and metal cations which are reducible to metal in solution by hypophosphite and amine borane anion reducing agents.

2. A method as claimed in claim 1, wherein the alloy particles produced are collected, washed, and dried.

3. A method as claimed in claim 2, wherein the particles are dried in the absence of oxygen.

4. A method as claimed in claim 1, wherein the alloy particles prepared are spherical finely divided ferromagnetic alloys, and the reducible metal cations are selected from the group consisting of cobalt cations, and mixtures of cobalt and iron cations, and mixtures of cobalt and nickel cations.

5. A method as claimed in claim 4, wherein the ferromagnetic alloy particles produced are collected, washed, and dried.

6. A method as claimed in claim 5, wherein the particles are dried in the absence of oxygen.

7. A method as claimed in claim 4, wherein the source of hypophosphite anion is an alkali metal hypophosphite salt, and the source of amine borane is dimethyl amine borane.

8. A method for preparing spherical finely divided ferromagnetic alloys of cobalt having a diameter of about 0.01 to 3 microns by reduction comprising:

preparing a bath consisting essentially of hypophosphite anion reducing agent and reducible metal cations selected from the group consisting of cobalt cations and mixtures of cobalt and iron cations and mixtures of cobalt and nickel cations;

preparing a solution including amine borane anion reducing agent;

mixing the amine borane anion solution with the hypophosphite anion-metal cation bath; and

heating the mixture for a time sufficient to form finely divided cobalt alloys in solution.

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