

Dec. 26, 1944.

C. C. NEIGHBORS

2,365,720

HIGH FREQUENCY CORE MATERIAL AND CORE

Filed Feb. 9, 1940

3 Sheets-Sheet 1

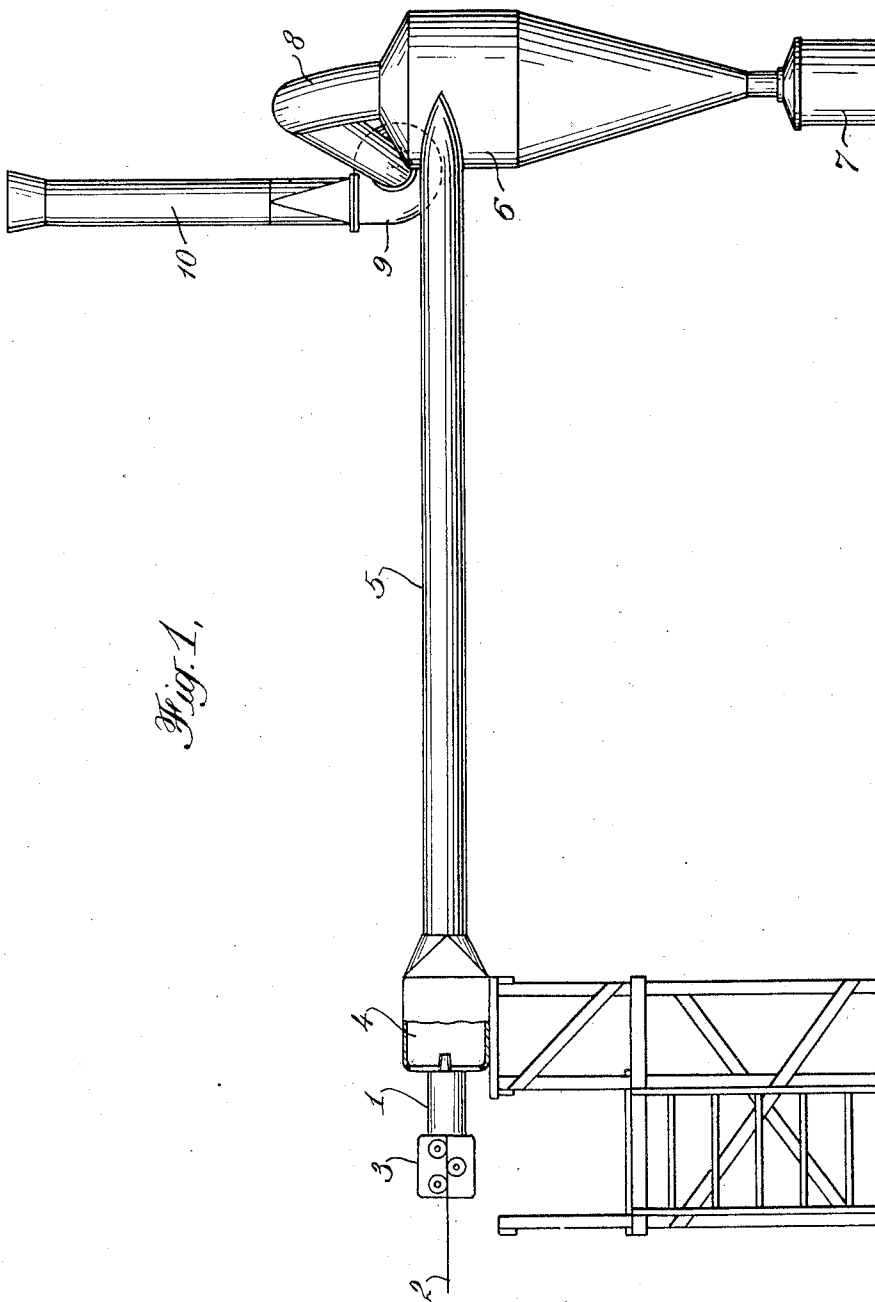


Fig. 1,

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3 Sheets-Sheet 2

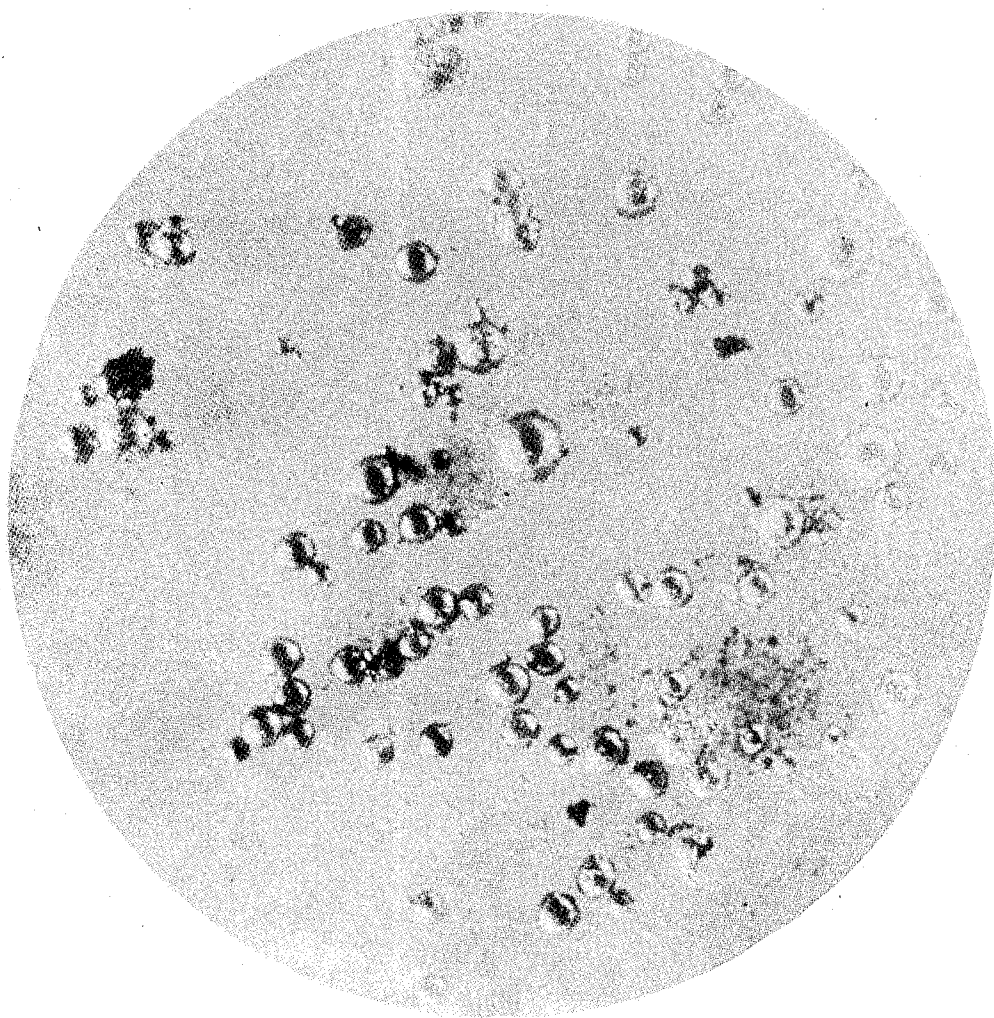


Fig. 2,

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3 Sheets-Sheet 3

Fig. 3,



Fig. 4,

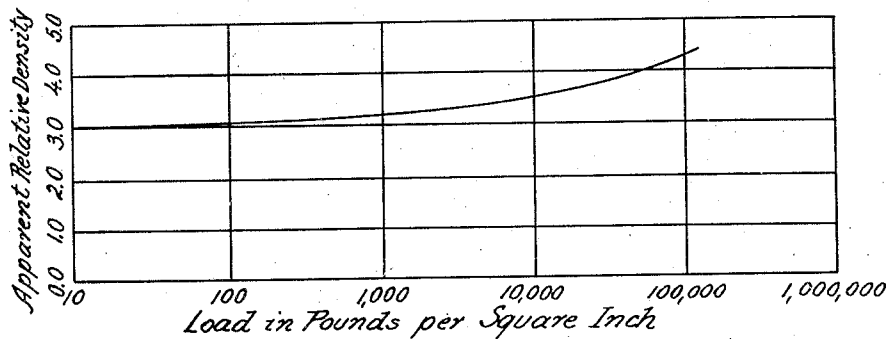
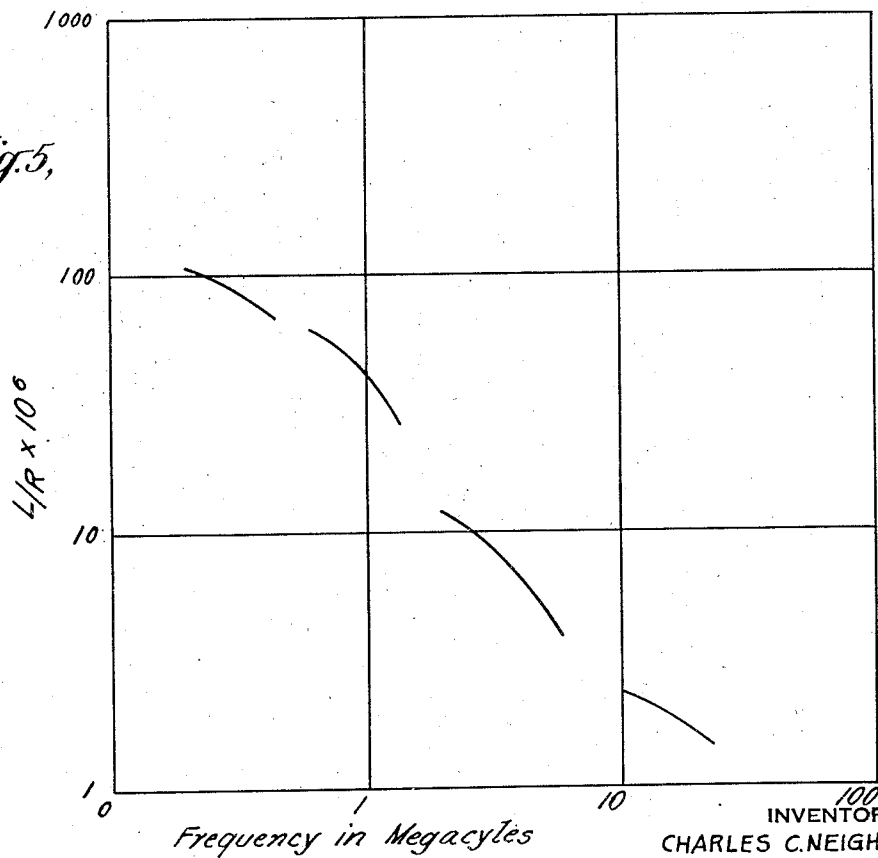


Fig. 5,



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UNITED STATES PATENT OFFICE

2,365,720

HIGH FREQUENCY CORE MATERIAL AND CORE

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Application February 9, 1940, Serial No. 318,114

7 Claims. (Cl. 175—21)

This invention relates to a finely-divided substantially metal-free ferromagnetic mass-powder product, the particles of which, as they appear under a microscope, are of generally rounded form. The invention has to do particularly with a magnetic-oxide-of-iron product having certain novel and useful electrical and physical properties and with a process of controllably producing the improved product with those properties.

As produced in accordance with the invention, products thereof when formed into appropriate shapes, preferably with a binder and with compression, provide highly useful so-called "high-frequency" cores which are comparatively economical to produce and which may be employed successfully in inductors adapted to be operated at high and ultra-high frequencies, for example up to and beyond 150 megacycles.

I am aware of prior proposals and uses of finely divided ferromagnetic essentially "metallic" particle mass-powder products, the particles of which are of generally rounded or spherical form. For example, the product of United States Patent No. 1,695,041 is essentially one having spherical metallic particles which may or may not be provided with an insulating oxide film or coat by heating the particles as produced or after production, in an oxidizing atmosphere. United States Patent No. 1,840,286 likewise provides metallic iron particles from iron carbonyl which as produced are generally of spherical form. In United States Patent No. 2,038,251 the particles as ultimately formed are metallic and generally spherical in form, and like those of United States Patent No. 1,695,041, may or may not be provided with an insulating oxide film or coat.

I am also aware of the prior proposals and uses of finely divided ferromagnetic iron-oxide or magnetite substantially metal-free particle powder products, the particles of which as they occur in nature or as produced or ground are generally irregularly shaped or formed, usually angular. These irregularly-shaped-particle powder products are readily distinguished from the powder product contemplated herein, as will be further explained in the description to follow.

I believe I am the first to conceive and controllably produce a substantially metal-free ferromagnetic-oxide-particle mass-powder product which is particularly adapted for use at extremely high and ultra-high frequencies. My invention provides a process for the manufacture of this improved product which requires equipment of only moderate cost, which may be operated to produce the product continuously as distinguished from batch operation, and which utilizes readily available materials and apparatus.

My invention includes the process of producing the highly pure ferromagnetic powder, as well

as the mass-powder product itself as a composition of matter, and compressed comminuted cores or similar bodies made with the powder as a principal ingredient as articles of manufacture.

5 In the accompanying illustrative drawings, one figure of which reproduced from a photograph,

Fig. 1 is an elevation, partly in section, of one suitable form of apparatus for carrying out the process in accordance with the invention;

10 Fig. 2 is a photomicrograph of a typical sample of the powder as produced by the apparatus of Fig. 1;

Fig. 3 is a perspective drawing of one form of compressed comminuted core which may readily be produced with the powder of Fig. 2 and which has important commercial application;

Fig. 4 is a graph showing the apparent relative density of a sample of my improved material under varying pressures; and

20 Fig. 5 is a graph showing the variation of the inductance-to-resistance ratio with frequency for standard test coils with cores made from a sample of the improved material according to the invention.

It will be readily understood that the improved ferromagnetic powder in accordance with my invention may be molded with any suitable binding agent in innumerable forms, depending upon the design and the requirements of the particular application, and that I show in Fig. 3 one such form merely as illustrative of a typical compressed core.

My improved ferromagnetic mass-powder product consists substantially wholly of magnetic oxide. My process for producing this powder, in its essential aspects, consists in producing minute masses of metal-containing material at elevated temperature and in introducing them into an oxygen-containing atmosphere and maintaining them in this atmosphere and out of contact with each other for a time adequate to allow them to combine with the oxygen. In carrying out the process, the material is first converted by heat to the liquid state, and the material is then separated into minute masses which are then subjected to the oxygen-containing atmosphere while still at a temperature adequate to assure oxidation in the desired degree. The reaction between the metal and the oxygen generates heat which tends to maintain the masses at elevated temperature, the heat thus generated being sufficient to facilitate the completion of the oxidation, after which the masses cool and solidify, and become entirely stable. As will be explained later in connection with the chemical analysis of an illustrative sample of my mass-powder product, the process is such that in producing the oxide-of-iron product, the oxidation ceases when a magnetic oxide is produced.

Any suitable iron-containing material may be

employed, and any suitable method of converting it to the molten state may be used. The material is conveniently separated into minute masses by the use of an air jet of suitable velocity, which, in itself provides an oxygen-containing atmosphere in which the minute masses may be supported out of contact with one another, except for accidental collisions, while oxidation proceeds. If the air jet is directed into a closed duct, the masses are carried through the duct by the air stream thus created and may be collected as cooled oxide particles at the end of the duct. Additional air may be provided in the duct to supplement the air provided by the jet.

A convenient source of heat for converting the material to the molten form is an oxy-acetylene flame. The air jet for separating the material into minute masses may be introduced directly into the flame. A suitable burner for carrying out this arrangement is shown in United States Patent No. 1,695,041. Alternatively an electric arc may be employed to liquefy the material, and the air jet may be blown through the arc to form the minute masses. A suitable apparatus for employing such an arc and air jet is shown in United States Patent No. 2,157,498. Either of these devices may readily be arranged with a suitable duct and with suitable additional air supplies if necessary, in order to produce oxidation of the masses, in accordance with the invention, as will be described in more detail later. It should be noted that the devices of the two patents above mentioned are intended to produce metallic coatings, but that they may be arranged to produce oxide particles in accordance with the invention by employing the additional apparatus and the additional process steps herein disclosed.

Referring now to Fig. 1 of the drawings, I prefer to carry out my process by employing an oxy-acetylene burner 1 and by using a steel wire 2 as the iron-containing material. The burner is supplied with oxygen and acetylene. Adjacent the burner there is an air turbine 3 with suitable reduction gears and wheels to feed the wire 2 into the flame. The air turbine 3 is supplied with compressed air, which is exhausted through the flame and projects the material in the form of minute masses into the chamber 4 the rear wall of which is open and thence into duct 5, at the far end of which the oxide particles formed during transit through the duct are collected in dust collector 6, and fall into can 7. At the top of dust collector 6, a pipe 8 connects to exhaust fan 9 and thence to stack 10. By means of exhaust fan 9 a large amount of additional air may be drawn into the system through the open rear wall of chamber 4.

In operating the arrangement shown in Fig. 1, I prefer to employ a steel wire of No. 11 B. and S. gauge, having a carbon content of 0.4 percent, but numerous other iron, and steel wires will be found to result in a satisfactory product. I prefer to adjust the flame to consume about 1 cubic foot of oxygen and of acetylene per minute, and the air jet through the flame to deliver about 50 cubic feet of air per minute. The wire is preferably fed into the flame at the rate of about 4 feet per minute. The exhaust fan 9 is preferably adjusted to create an additional air stream of about 3500 cubic feet per minute. Under these conditions the time of transit of the minute masses and their conversion into oxide particles is less than 1 second. I have found that for most satisfactory results the temperature in the duct

5 should be maintained relatively low. This temperature may be regulated by the amount of air drawn through the duct by the exhaust fan 9. In the arrangement shown in Fig. 1, the chamber 4 may suitably be about 2 feet on a side and the duct 5 about 1 foot in diameter and 18 feet long. A satisfactory dust collector may be 4 feet in diameter at its largest cross-section and 8 feet high. It will be apparent that the entire apparatus may alternatively be arranged in a vertical manner. The proportions, quantities and materials stated are illustrative only, and may be varied to suit different cases.

In accordance with the invention the metal is raised to a temperature sufficient to make it fluid and in a condition to be blown into the duct in the form of a fine spray, in which condition by contact with the air in the duct, oxidation begins. Oxidation of the molten particles of the spray is an exothermic reaction producing heat which tends to at least maintain the temperature of the particles until their oxidation is completed, after which the particles rapidly cool, the temperature of the material as collected in can 7 being rarely in excess of 100° F.

An important and highly advantageous attribute of the mass-powder product thus produced, and one which I regard as a principal reason for its unique and highly advantageous properties, is that although the particles are oxide particles, they are nevertheless spherical in form. The spherical form is assumed after the material is torn away from the heated end of the wire by the stream of gas, and is probably due principally to the surface tension of the liquid material. The particles retain this spherical form as they proceed through the duct and as oxidation progresses. The particles are very minute, as will be described in more detail later, and thus have a relatively very large ratio of surface area to volume, and this, I believe, assists materially in the oxidizing process.

In a sample of the powder produced in accordance with the preferred form of the process above described, the average diameter of the oxide particles was well below 20 microns. Using standard wire cloth screens, a sieve analysis to determine the particle-size distribution of this sample showed percentages by weight as follows:

	Per cent by weight	Computed per cent of total number of particles
Retained on a 100-mesh screen.....	4.25	(1)
Through 100-mesh, retained on 200-mesh....	20.70	0.84
Through 200-mesh, retained on 300-mesh....	16.60	4.06
Through 300-mesh, retained on 400-mesh....	3.90	2.43
Through 400-mesh.....	54.55	92.90

¹ Neglected.

The percents of the total number of particles are computed from the percents by weight and from the volume of a particle having a diameter midway between the screen openings. The largest particles in the above sample, of which there were only a very few, measured under the microscope, showed an average diameter of about 168 microns. The smallest particles, of which there were a very large number, measured by the same method, showed an average diameter somewhat over one micron. While this sample may be regarded as typical for the conditions under which it was produced, it will be understood that with variations in the conditions of the process, the

average, maximum and minimum particle sizes produced in accordance with the invention, as well as the particle-size distribution, may vary considerably.

Upon analysis as shown below, samples of my improved material made from the above mentioned steel wire, are found to consist principally of iron and oxygen substantially in the proportions corresponding to the ferrous-ferric oxide, Fe_3O_4 . It is to be noted that ferric oxide, Fe_2O_3 , contains more oxygen than ferrous-ferric oxide, Fe_3O_4 , in the ratio of 9 to 8, that is, three molecules of the ferric oxide, Fe_2O_3 , contain six atoms of iron and nine atoms of oxygen, whereas two molecules of ferrous-ferric oxide, Fe_3O_4 , contain six atoms of iron but only eight atoms of oxygen. Complete oxidation in the chemical sense, therefore, would result in the production of ferric oxide, Fe_2O_3 . It is apparent, however, that my process as applied to the production of an oxide-of-iron product, produces only sufficient oxidation to result in a stable powder containing iron and oxygen in the proportions corresponding to the ferrous-ferric oxide, Fe_3O_4 , and that the oxidation achieved by the process is automatically arrested and completed with the production of this product.

The amount of free iron in the above referred to samples of my improved product is quite small, but uncombined iron may be present in some cases to a larger but not detrimental extent. Manganese is also present unless manganese-free iron is employed in the process of manufacture. With commercial steel wire as suggested above, manganese may be present in the mass-powder product to the extent of about four-tenths of one percent. The carbon present in the steel wire largely disappears in the process, and in the final product is only present to the extent of from two to three one-hundredths of one percent.

In the preparation of many magnetic materials by other processes, additional steps of treatment are used in order to reduce impurities to a minimum. A feature of my process and of the mass-powder product resulting therefrom is that impurities are present in remarkably small percentages, so that the employment of any additional purifying process steps becomes unnecessary.

I give below an analysis of a typical sample of my improved material made from steel wire, and of a sample of commercial powdered natural magnetite, it being understood that the percentages shown will vary somewhat from sample to sample:

	Improved material according to the invention	Commercial powdered natural magnetite
	Percent	Percent
Total iron.....	71.43	68.87
Metallic iron (free).....	0.07	0.02
Oxygen.....	27.81	26.05
Carbon.....	0.02	0.09
Silica.....	0.40	1.73
Sulphur.....	0.005	0.024
Phosphorus.....	0.007	0.180
Manganese.....	0.406	0.102
Alumina.....	0.09	0.76
Titania.....	0.006	0.55
Calcium oxide.....	None	0.73
Magnesium oxide.....	None	0.24

Figure 2 is a photomicrograph of a typical field of particles from a random sample of the improved material. In taking this photograph,

compound illumination was employed in order to duplicate, as nearly as possible, the appearance of the particles when viewed through the microscope by eye. The basic magnification was 250 diameters. The particles are black or nearly black in color and possess considerable luster, which renders them difficult to photograph.

It will be seen that substantially all of the particles are spherical in form, being true spheres as nearly as can be judged from the photograph. It will also be noted that they vary greatly in size, and that the very small particles are very numerous. It will also be noted that many of the small particles are on the surfaces of the larger particles, so that in many cases the surface of a large particle is quite well covered with small particles. This is a distinguishing feature of my improved material, which is exhibited, at least to a noticeable degree, by the powder as produced.

Study of Figure 2 will reveal that there are a relatively very small number of particles of irregular (non-spherical) shape. I am unable to state what causes these irregular particles, but they appear to be incompletely formed spheres and many if not all of them are found to be hollow, with wall thicknesses varying over wide limits. The possibility that even the perfectly formed spheres are hollow is indicated by the relatively low immersion density of the powder, which in a measured sample was 4.66, as compared with the density of 5.18 of natural magnetite. Many of the larger particles may easily be fractured by squeezing them between two microscope slide glasses, and these fractured particles in many cases are also found to be hollow.

By means of a hardened steel cell and two hardened steel plungers arranged to enter the cell from top and bottom respectively, it is possible to determine the apparent relative density of any powder at various pressures, suitable gauges being provided for indicating the spacing between the parallel ends of the plungers. A carefully weighed charge of the material under test is placed in the cell and the upper plunger replaced. The initial apparent relative density of the material as computed from the gauge readings and the weight of the sample, corresponds to the very small pressure due to the weight of the upper plunger and its supporting member. The fixture may then be placed in a compression testing machine and measured loads applied, note being made of the decreased spacing between the plungers. From these readings, the apparent relative density at each load may be computed.

It will be understood that the initial relative density thus determined is the density of a solid of the computed volume, that is to say, it neglects the fact that there are voids in the sample. Subsequent readings, however, indicate accurately the extent to which it is possible to close these voids and to cause them to be filled with the smaller particles of the material.

The results of such compression tests are most easily analyzed by plotting the load in pounds per square inch against the apparent relative density of the charge, on semi-logarithmic paper. Figure 4 shows such a plot for a sample of the material in accordance with my invention. It will be noted from the curve of this figure that the sample of my improved material tested had an initial relative density of 2.99 and a density of 4.43 under a pressure of 125,000 pounds per square inch and that it was relatively resistant to compression. After compression, the material

according to my invention was still in powdered form.

My improved magnetic material may be prepared for molding into cores by first classifying the material to secure a desired range of particle sizes and then mixing it with a synthetic resin, as for example Bakelite, preferably in powdered form, which acts as a binder when the particles are compressed into a core. The amount of binder will be from 1 to 4 percent of the weight of ferromagnetic powder in the usual case, but may be more than 4 percent in special cases. In accordance with a preferred process, powdered Bakelite, consisting of particles sifted through a 200-mesh screen, is intimately mixed with the magnetic particles and enough ethyl alcohol or other suitable solvent is added to thoroughly wet the mixture with a slight excess. The solvent is then evaporated to dryness with constant stirring. It will be understood that Bakelite varnish may equally well be employed. The particles are then passed through a fine screen (60 to 100 mesh), any lumps being pulverized. The material is then ready to mold. The magnetic material thus produced may be either hot or cold molded into cores of any desired shape. If hot molded with Bakelite, the mold is preferably maintained at a temperature of approximately 205 degrees F. Cores produced by either hot or cold molding are preferably cured at 300 degrees F. for three or more hours, until the Bakelite is completely polymerized, or at any temperature and for any period which will accomplish this.

The molding pressure and the properties of the finished core depend upon its particular shape and size. In the case of a tubular core as shown in Fig. 3, having a diameter of three-eighths of an inch and a length of one-half inch, a pressure of 23 to 27 tons per square inch may be employed. The specific gravity of such a core will be approximately 3.9.

The resistivity of material compressed with 3 percent of Bakelite binder, computed from the measured direct-current resistance between suitable electrodes pressed against opposite faces of a sample of suitable shape, is of the order of from 15,000 to 16,000 ohm-centimeters.

The apparent permeability of a compressed comminuted ferromagnetic material may be determined from measurements on a toroidal core of rectangular cross-section in which the difference between the external and internal diameters is small compared with the external diameter, and in which the axial length is about one-third of the external diameter, using a single-layer winding having enough uniformly spaced turns to substantially cover the surface of the core. The inductance of the winding and core combination is preferably measured on a radio-frequency bridge, at a standard test frequency which for convenience may be taken as 1,000 kilocycles. From this inductance value, the dimensions of the winding and core, and the number of turns in the winding, the apparent permeability μ may be computed using the following formula:

$$\mu = \frac{85.5L}{\pi^2 s \log d_2/d_1} - \left(\frac{S \log D_2/D_1}{s \log d_2/d_1} - 1 \right)$$

where

L=measured inductance in microhenries

s=axial length of the core in inches

S=axial length of the winding in inches

D₁=inside diameter of the winding in inches

D₂=outside diameter of the winding in inches

d₁=inside diameter of the core in inches

d₂=outside diameter of the core in inches

n=number of turns.

This formula is based upon the ratio of the inductance of the winding and core, as measured, to that of the winding alone.

The apparent permeability μ is a multiplying factor representing the increase in the inductance of the winding due to the presence of the core. By constructing an identical winding on a core of insulating material, and by measuring the high-frequency resistance of the iron-core winding and the "air-core" winding at the test frequency, and taking the ratio of these two resistances, a similar factor for the resistance increase due to the core may be determined. I prefer to call this quantity the "resistancy" of the core material, and to represent it by the Greek letter (ρ). Material made from particles of my improved powder which would pass through a 400-mesh screen, measured at 1000 kilocycles in the manner described above, showed an apparent permeability of 6.4 and a resistancy of 4.2.

It will be understood that variations in particle size, kind and amount of binder, and compression pressure will produce corresponding variations in both apparent permeability and resistancy, and that while the apparent permeability is reasonably constant up to some very high limiting frequency, the resistancy varies rapidly with frequency.

Reference is now made to Figure 5, which shows the inductance-to-resistance ratio, L/R, plotted against frequency for a set of standard test coils, using with each coil the same 1/2-inch long by 3/8-inch diameter plug core made of my improved material. It is to be remembered that this ratio depends not only upon the core material and its apparent permeability and resistancy but also upon the size and shape of the core, and the size and shape of the winding, and its relation to the core. The coils used in these measurements were each wound on an insulating tube having an inside diameter of .386 of an inch and a wall thickness of .015 of an inch, the coil used between 10 and 24 megacycles being a solenoid having its turns spaced twice the wire diameter, and the other three coils being universal wound. Four different coils were used. The coil used between 200 and 500 kilocycles had 247 turns of 7 strand #41 enamel litz wire, single silk covered, the coil used between 500 and 1500 kilocycles had 97 turns of 20 strand #44 enamel litz wire, single silk covered, the coil used between 2 and 6 megacycles had 22 turns of 30 strand #46 enamel litz wire, double silk covered, and the coil used between 10 and 24 megacycles had 7 turns of #22 copper plain enamel wire.

With facilities thus far available, it is difficult to make accurate measurements at ultra-high frequencies much above 75 megacycles. Experiments have shown, however, that my improved material continues to be useful as the frequency is increased above that value, so that even at 150 megacycles and above, my improved material may advantageously be employed to tune a resonant circuit over a range of frequencies. This is not true of powdered magnetite or other known powdered magnetic materials, which lose their favorable characteristics at frequencies below 75 megacycles.

I am aware that United States Patent No. 1,695,041 discloses the use of a Schoop's pistol for

preparing a ferro-magnetic powder. It directs, however, that the particles as formed by the pistol are to be immediately chilled, for example by projecting them directly into a tank of water, as shown in the patent drawings. In such a process there is little opportunity for the particles to be converted even partially into oxide, and in fact the patent states that the particles are metallic. I have prepared a powder in accordance with the instructions given in the said patent, and, as expected and as stated in said patent, the particles upon analysis are found to be substantially pure metallic iron. I have constructed cores from this material, and I find that it is markedly inferior to my improved material, even at frequencies as low as 100,000 cycles, and that it is increasingly inferior as the frequency is increased.

While magnetic oxide of iron is probably the most readily produced magnetic oxide, other ferromagnetic oxides, for example of other metals in the iron group and of various alloy metals, can be produced. Therefore, while I have described my process and the product resulting therefrom by reference to magnetic oxide of iron, the possibility of substituting other metals or alloys must be kept in mind. It may be, in fact, that ferromagnetic mass-powder products can be produced in accordance with my process from materials containing only a relatively small amount of iron or no iron at all, which will be comparable in properties with that produced from materials consisting substantially entirely of iron. When in the claims, therefore, I refer to a ferromagnetic oxide mass-powder product, the term "ferromagnetic" is not to be taken as limited to iron-containing products, but is to be understood in its commonly accepted sense, that is, as referring to products which exhibit magnetic properties like those exhibited by iron.

Careful examination of numerous samples of my improved mass-powder product under the microscope indicates that while the particles are characteristically of ball-shaped or spherical form, numerous and various irregular shapes occur as well as particles which appear to be the result of fracture of originally generally rounded particles. It is to be understood, therefore, that when in the claims I describe the particles as of generally rounded form, I refer to their characteristic shape without excluding irregular shapes and fractured portions such as are observable in Fig. 2.

By the term "metal-free" as used in the specification and claims to in part define the product of the invention, I mean that the product is substantially free from metal in the uncombined metallic state.

While I have shown my invention in the particular embodiment above described, it will be understood that I do not limit myself thereto, as I may employ equivalents thereof without departing from the scope of the appended claims.

Having thus described my invention, what I claim is:

1. A compressed ferromagnetic core consisting principally of minute particles of magnetic oxide

of iron in substantially spherical form produced by liquefying iron at an elevated temperature, picking it up in minute masses in a stream of gas and introducing said masses into a stream of oxidizing gas for a sufficient time to permit them to oxidize.

2. A compressed ferromagnetic core consisting principally of minute particles of magnetic oxide of iron in substantially spherical form produced by melting iron, picking it up in minute masses in a stream of gas of approximately the same temperature as the molten iron and introducing said masses into a stream of an oxidizing gas of a temperature below that of the molten iron and keeping said masses suspended in said oxidizing gas out of contact with each other until they are cool.

3. A compressed ferromagnetic core for use with inductance coils operating at high frequencies and having its magnetic component consisting principally of a synthetically produced ferromagnetic powder of minute particles of magnetic oxide of iron whereof substantially all of said oxide particles are of substantially spherical form imparting to said core at the ultra-high frequencies a substantially improved L/R producing characteristic.

4. A compressed ferromagnetic core for use with inductance coils operating at high frequencies and having its magnetic component consisting principally of a synthetically produced ferromagnetic powder of minute particles of magnetic oxide of iron whereof substantially all of said oxide particles are of substantially spherical form imparting to said core at the ultra-high frequencies a substantially improved L/R producing characteristic, said powder having an apparent density of approximately 4.6.

5. A compressed ferromagnetic core for use with inductance coils operating at high frequencies and having its magnetic component consisting principally of a synthetically produced ferromagnetic powder of minute particles of magnetic oxide of iron whereof substantially all of said oxide particles are of substantially spherical form imparting to said core at the ultra-high frequencies a substantially improved L/R producing characteristic, and are of such sizes that not less than 50% by weight will pass through a screen having 400 meshes to the inch.

6. A compressed ferromagnetic core for use with inductance coils operating at high frequencies and having its magnetic component consisting principally of a synthetically produced ferromagnetic powder of minute particles of metal-free magnetic oxide of iron whereof substantially all of said oxide particles are of substantially spherical form imparting to said core at the ultra-high frequencies a substantially improved L/R producing characteristic.

7. A compressed ferromagnetic core having its magnetic component consisting principally of synthetically produced ferromagnetic minute particles of magnetic oxide of iron in substantially spherical form.

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