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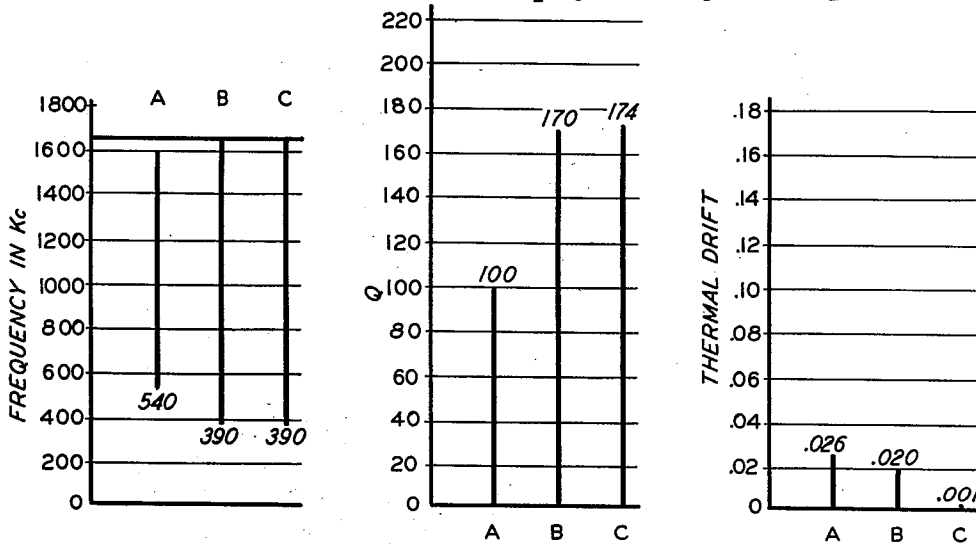
G. BERGE

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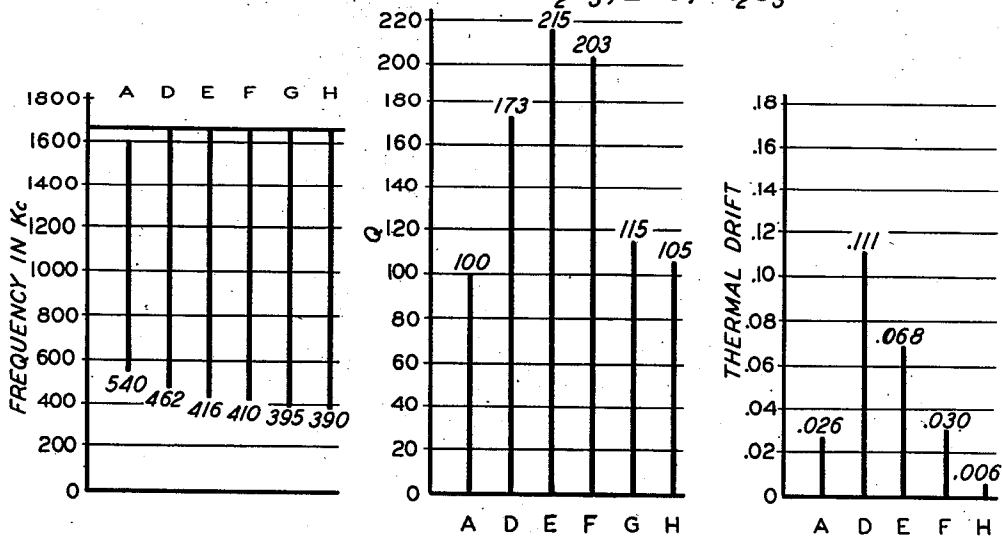
REACTION PRODUCT OF A MIXED FERRITE AND LEAD TITANATE

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SYSTEM BASED ON Fe_2O_3, ZnO, MgO, MnO_2



SYSTEM BASED ON Fe_2O_3, ZnO, Ni_2O_3



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REACTION PRODUCT OF A MIXED FERRITE
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This invention relates to magnetic cores which form a part of the coils and transformers of radio-frequency circuits. It relates, more particularly, to the composition of and the method for making a magnetic core by which many desirable and improved characteristics are imparted to radio-frequency circuits.

It is a matter of common knowledge to those skilled in the art that selectivity, that is, the ability to select one desired frequency and discriminate against others, is greatly influenced by the shape of the resonance curve or sharpness of the radio circuit. A sharp circuit gives good selectivity and sharpness, in turn, is regulated by the Q (quality factor) of the circuit. At frequencies within the normal broadcast band, the Q factor of the circuit is greatly influenced by the quality and characteristics of the magnetic core in the coil. It is a desideratum, therefore, to provide a magnetic core which introduces a high Q factor, especially one that causes the circuit to exceed the value of 100 characteristic of present radio circuits.

Selectivity is but one of the important characteristics of a radio-frequency circuit which is subject to influence by the magnetic core. There are many others. For example, the magnetic core plays an important role in the determination of the tuning range of the radio circuit. The present range of most radio receivers lies between 540 and 1600 kc. and, of course, it is desirable to broaden the tuning range as much as possible without harmfully affecting other characteristics of the radio circuit.

Still another important consideration in radio circuits is the characteristic of frequency stability, that is, the ability to hold a set or tuned frequency for an extended time. Invariably it is desirable to maintain the resonant frequency as constant as possible. One of the principal factors that causes changes of frequency is the influence of temperature. Temperature changes cause expansions or contractions of the various elements of the radio circuit and correspondingly cause changes in capacitances and inductances and the frequency will change accordingly. Frequency changes caused by temperature change are referred to as thermal drift. Temperature changes are unavoidable, but it is considered possible by proper selection of materials and by proper arrangement of elements to reduce the thermal drift to a minimum. Here too the magnetic core plays an important part.

It is an object of this invention to provide a new and improved magnetic core composition and

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a method for producing the same. The composition of my invention is particularly adapted for the production of magnetic cores having greatly improved operating characteristics in radio-frequency circuits; that overcomes many of the shortcomings of magnetic cores now in use in such circuits; that introduces into the circuit a Q value which is much higher than the value obtained with core compositions now in use with the result that improved selectivity is secured; that extends the tuning range of the radio circuit; that is an outstanding improvement in markedly decreasing the thermal drift and teaches a concept by which thermal drift may be correspondingly lowered in other magnetic core compositions; that is relatively easy and economically manufactured of low cost and available materials, and which forms a core of predetermined shape having sufficient strength and durability to withstand the forces incident to normal handling and use.

Another object is to provide a new and improved magnetic core and a method for producing the same which, amongst other things, embodies the properties described above and which is fabricated of finely divided materials temporarily bonded with a heat destructible adhesive and finally bonded to predetermined shape to produce a microporous product in the manner corresponding to the principles of powder metallurgy, the finely divided particles remaining substantially in their original form to provide for desirable characteristics with respect to eddy current losses.

Briefly described, my invention contemplates the reaction at elevated temperatures of the oxides of various metals, including zinc, iron, and one or more of the oxides of the metals selected from the group consisting of magnesium, manganese, cobalt and nickel. For instance, my invention may be practiced by the use of a ternary system based upon the use of iron oxide, zinc oxide and nickel oxide or iron oxide, zinc oxide and cobalt oxide. The system may be quaternary based upon iron oxide, zinc oxide, magnesium oxide and manganese oxide, or it may be a five component system based upon iron oxide, zinc oxide, magnesium oxide, manganese oxide and nickel or cobalt oxide. The oxides are usually reacted in finely divided form and in intimate mixture. Originally the mixture has very little, if any, magnetic properties, but upon reaction at the desired thermal conditions the reaction product is highly magnetic and can be used advantageously in the fabrication of mag-

netic cores having characteristics in radio-frequency circuits which were heretofore unobtainable with commercial core compositions.

I have found further that by the addition of titanium oxide or titanium and lead oxides to the above compositions, the characteristics of the magnetic core can be further improved. That is, additions of limited amounts of titanium and lead oxides, which preferably is added in combined form as lead metatitanate (PbTiO_3), hereinafter referred to as titanate, further improves the turning range and the Q value of the circuit and reduces the thermal drift characteristics of the core by outstanding proportions. The added ingredients give considerable strength to the molded core product to the extent that they are better able to withstand the forces incident to normal handling and use.

In practicing my invention, I prefer to consider the invention as involving two distinct concepts. The first relates entirely with the production of the basic magnetic materials for magnetic cores, including reaction of the oxides of iron, zinc, and one or more of the oxides of the metals magnesium, manganese, nickel and cobalt, to produce a product having high magnetic properties. The second concept is concerned entirely with the fabrication of the magnetic core from the basic materials alone or in admixture with the titanium and lead oxides. Although greatest benefit is derived by the addition of the latter oxides to the basic reaction product, I have found that many of the desirable improved characteristics may be derived also by the addition of these titanium and lead oxides to the original basic ingredients prior to their thermal reaction.

In the original mixture, the materials may be present in the ratio of 60 to 75 parts of iron oxide (calculated as Fe_2O_3), 10 to 25 parts by weight of zinc oxide (calculated as ZnO), and 10 to 25 parts of one or combinations of the other oxides which may be either nickel oxide (Ni_2O_3), cobalt oxide (Co_2O_3), manganese oxide (MnO_2), or magnesium oxide (MgO). The oxides in finely divided or powder form are intimately mixed and heated or fired together at a temperature in the range of about 2200 to 2500° F. for an extended time. Ordinarily firing is continued for two or more hours, and I have obtained excellent results by reacting the mixture of oxides for three hours at 2300 to 2400° F. The original mixture before firing has very little, if any, magnetic characteristics, but after the described thermal treatment the product is highly magnetic. The reaction product is cooled down and then reduced into finely divided or powder form for subsequent use as a basic material in the fabrication of magnetic cores.

In the second phase, the finely divided magnetic material, that is, the reaction product, is mixed with a binder which serves to impart temporary mechanical strength immediately to a formed mass of the reaction product enabling the substances to be molded to desired shapes in the usual manner. The molded composition is then heat treated at about 1900 to 2200° F. for about an hour or more. Excellent cores result from the heat treatments of about one and one-half hours at 2050° F., or one hour at 2200° F., after which the heat treated core is cooled to room conditions. I prefer to cool the heat treated product rather slowly for best results, for example, I prefer to cool the core from a temperature of 2050 or 2200° F. at the rate of about

100° F. per hour. My invention, however, should not be limited to these conditions as higher or lower temperatures may be used to advantage, and correspondingly longer or shorter times in heat treatment may be used because heat treatment, cooling, and cooling rate is greatly influenced by the mass of material involved.

When an organic binder is used originally to secure the particles in molded form, the binding property generally is of temporary character which holds the particles in the desired arrangement until the heat treatment is effective to cause bonding such as is experienced in powder metallurgy. The organic binder will for the most part be burned off at the temperature of heat treatment and very little effect will be had thereof as a binder or as an insulator in the final product. Suitable organic binders may be based on liquid or solvent solutions of various adhesive resins such as the phenol-aldehydes, urea or melamine-aldehydes, vinyl polymers and copolymers, polyacrylates, rubber based materials, polystyrene and the like. The amount of binder generally is influenced by the constituents of which it is formed, the particle size and character of the base materials, and the pressures under which the core is molded. I prefer to use the least amount of binder necessary. I have successfully molded cores with as little as 2 per cent binder. However, 3 per cent binder gives excellent results and not infrequently as much as 6 or 8 per cent have been used. In any event, the final product is of a microporous nature, in which the mixed heat treated constituents for the most part are in their original form.

It is in this latter stage of the process that I prefer to add the oxides of titanium or the oxides or titanium and lead. I have secured best results when these latter oxides are added in combined form, such as titanate. The use of titanate as a constituent with my basic materials, comprises a cardinal feature of my invention. By the use of titanate cores are produced which favorably influence the radio circuit with respect to Q values and tuning range. By the additions of titanate, the thermal drift of the core is reduced by outstanding proportions. The additions of the oxides of titanium and lead appear greatly to improve the strength of the molded product subjected to a heat treatment. To the best of our knowledge, the additives become binding agents for the matrix of base materials because the coherence and the strength of the core increases according to the amount of titanate used.

By way of illustration but not by way of limitation, the following examples illustrate the manner for practicing my invention.

Example 1 (composition B in the graphs)

A mixture constituted of 69 parts iron oxide (Fe_2O_3), 17 parts zinc oxide (ZnO), 10 parts manganese oxide (MnO_2), and four parts magnesium oxide (MgO) in finely divided or powder form, is fired at about 2300 to 2400° F. for about three hours. The original mixture is relatively non-magnetic. The cooled reaction product, however, has highly magnetic characteristics. The cooled mass is friable and is reduced to finely divided form to comprise a base material which may be used as is for magnetic core formation.

To the granular reaction product described, there is admixed sufficient liquid binder, such as 2-3 per cent by weight of a phenolic resin advanced to an intermediate stage of polymeric

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growth, a soluble stage, and the mass is then formed in the usual manner, such as by molding to the desired core shape. Thereafter, the molded mass is heat treated at about 2050° F. for one to two hours and then cooled down slowly in steps of 100 degrees per hour until a temperature of 500 or 600° F. is reached. The resultant product is a molded core having the improved characteristics shown by the letter B in the accompanying graphs. It will be noted that the tuning band is extended to include 390 and 1650 kc. as compared to the limitation to 540 and 1600 kc. of prior materials. A very marked and beneficial improvement is had in the Q value, which with core composition formed in my first example is in the range of 170 as compared to 100 of the prior art. To a lesser extent the thermal drift is helpfully lowered.

Example 2 (composition C in the graphs)

To 98 parts of the reaction product of Example 1, 2 parts by weight of titanate (PbTiO_3) was added prior to or in conjunction with the admixture of the liquid binder. The required amount of material is molded under 1000 to 2000 pounds per square inch pressure to core shape which is then heat treated at about 2000° F. for one and one-half hours. Upon slowly cooling the heat treated core, it was found that the strength characteristic of the molded product was unexpectedly improved. More important, however, was the notable improvement in thermal drift. By the addition of 2 per cent titanate at this stage of the process, the thermal drift was lowered from an acceptable value of 0.020 to an astonishingly low value of 0.001. Instead of carrying out the heat treatment at 2000° F. it may be carried out for one hour at 2200° F. Further improvement is also secured in the Q value which rises to 174.

Example 3 (composition D in the graphs)

I form a basic 3 phase system by combining in intimate mixture of 66 parts by weight iron oxide (Fe_2O_3), 17 parts by weight zinc oxide (ZnO) and 17 parts by weight nickel oxide (Ni_2O_3). The ingredients may be fired for two hours at about 2500° F. during which a reaction takes place that converts these materials in combination from their original non-magnetic properties to a highly magnetic substance. The cooled and friable mass comprising the reaction product is reduced in finely divided form to comprise a material suitable for molding magnetic cores.

Magnetic core formation is effected by the addition thereto of about 2-3 per cent solution of the reaction product of the urea-formaldehyde in suitable solvent and the subsequent molding under 500-4000 pounds per square inch to desired core shape. The molded composition is heat treated for one hour at 2200° F. and then slowly cooled at about 100 degrees per hour to room temperature. The nickel oxide in the above formulation may be partially or wholly replaced by cobalt oxide or the oxides of manganese and magnesium. Instead of 3 per cent urea-formaldehyde solution, other binders in greater or lesser proportion may be used.

Example 4 (composition E in the graphs)

To the base composition or finely divided reaction product of Example 3, there is added about 2 per cent titanate and the core formation is effected in the manner described above by the addition of a temporary resinous binder and mold-

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ing in the usual manner. The molding may be heat treated for about two hours at 1900° F. and slowly cooled to produce a very desirable magnetic core. By reference to the graphs it will be evident that the addition of titanate was instrumental in extending the tuning range, increasing the Q value of the circuit to above 200, and noticeably reducing the thermal drift from that originally secured by the base ingredients.

Example 5 (composition F in the graphs)

To 96 parts of the reaction product of Example 3, there is added 4 parts by weight of titanate, and from this composition a core is formed in the usual manner of binder incorporation followed by the heat treatment at about 2100° F. for two hours. The increased amount of titanate causes very little change in the tuning band range or the Q value of the circuit which exceed present standards by a wide margin. The increased amount of titanate lowers by outstanding proportions the thermal drift of the resulting magnetic core and its presence is felt in the resulting increased strength of the core.

Example 6 (composition G in the graphs)

To 94 parts of the reaction product of Example 3, there is added 6 parts by weight of titanate. The composition is carried through the steps of binder incorporation, molding, heat treatment and cooling in the manner described in connection with Examples 4 and 5. Although the Q value of the circuit is lowered by the presence of the increased amount of titanate, it is still above the present value of 100. Important is the drastic reduction in thermal drift as shown in the graphical illustrations.

Example 7 (composition H in the graphs)

To 92 parts of the reaction product of Example 3, there is added 8 parts by weight of titanate. The composition is admixed with suitable organic binder and heat treated, then molded to form, at about 2200° F. for one and one-half hours. The greater proportion of titanate markedly improves the physical strength of the heat treated core. This may be caused by the greater amount of what may be a more fusible material which functions as a binding agent or it may be the result of better densification resulting in the molding step. The resulting product introduces a Q value of 105 into the circuit which is still above present standards. The frequency range is extended to 390 to 1650 kc. and the thermal drift is further reduced by an appreciable amount. From these results there is reason to believe that considerable benefit would be derived from the use of increased amounts of titanate, that is about 10 or 12 per cent. Obviously, the thermal drift, which is a very important characteristic in radio and high frequency circuits, is favorably influenced by its presence.

In making these tests on the core, a standard coil was used having a progressive universal winding of 492 turns 7-44, S. S. E. wire in one and three-eighths inch space on a tubing having 0.221 outside diameter.

Referring now to the graphs, a composition theoretically possessing the acceptable characteristics of magnetic cores in present use is designated by the letter A. Letters B, C, D, E, F, G and H correspond to the compositions of Examples 1 to 7 respectively. By comparing the graphical illustrations, the significance and the

importance of my invention will become immediately apparent.

For example, compared to standard values, the composition B of Example 1 extends the tuning range by 200 kc., there is a marked improvement in the Q values of the circuit, and the thermal drift is benefitted. The addition of titanate (C) is outstanding for its unprecedented and unexpected improvement which it imparts to the thermal drift characteristics.

Turning now to the compositions based on the system zinc oxide, iron oxide and nickel oxide, marked improvement is shown in the influence on the Q factor of the circuit by the care of composition D (Example 3). By the additions of titanate in increasing amounts noticeable reduction in thermal drift is secured and the tuning range is steadily extended. Vast improvements in the Q value of the circuit is shown when up to 6 per cent titanate is used and the values are still above normal when 8 per cent is used.

The thermal drift continues to decrease with the addition of more than 6 per cent titanate and by extrapolation should almost approach zero value at about 10 or 12 per cent. Where low thermal drift is important, lower Q values can be tolerated and therefore it is expected that even greater proportions of lead and titanium oxides might be used.

It will be apparent from the foregoing description that I have produced a new and improved core composition and materials for producing the same, having many properties and characteristics which exceed normal expectations without harmfully affecting other characteristics desired in a suitable magnetic core. My invention also illustrates a new concept for manufacturing magnetic cores for radio circuits out of finely dividing the metal oxides which are thermally reacted to produce a complex compound which for the present is unknown, the compound possesses the improved characteristics described. It illustrates a process by which magnetic cores may be economically formed of selected metal oxides converted by heat treatment to magnetic characteristics and the production of a microporous body having adequate physical strength to resist the forces incident to normal handling and use and in which eddy currents are held to a minimum. It sets forth the novel use of titanium oxides or titanium and lead oxides in proportions of about 12 per cent or less by weight to impart noticeable improvements in Q value of the circuit tuning range and outstanding reduction in thermal drift. It will be understood that the values in thermal drift secured by composition D and E may be compensated to the extent that the core introducing excellent Q value to the circuit may be used to advantage in radio circuits.

While the ultimate object of this invention is the production of a magnetic core, it will be readily understood that the fired base composition is likewise an article of commerce and comprises subject matter of this invention. It will be also understood that the titanium oxide or combined titanium and lead oxides may be originally incorporated with the base ingredients to be fired, and that numerous other changes in amounts and types of materials as well as conditions of treatment may be made without departing from the spirit of the invention especially as defined in the following claims.

I claim:

1. A magnetic core formed of the thermal reaction product of lead metatitanate with the

thermal reaction product of iron oxide calculated as Fe_2O_3 , zinc oxide calculated as ZnO and the oxide of a metal selected from the group consisting of magnesium, manganese, nickel and cobalt and mixtures thereof calculated as MgO , MnO , Ni_2O_3 and Co_2O_3 respectively.

2. A magnetic core formed of the reaction product of up to 12 percent by weight lead metatitanate with the thermal reaction product of iron oxide calculated as Fe_2O_3 , zinc oxide calculated as ZnO and the oxide of a metal selected from the group consisting of magnesium, manganese, nickel and cobalt and mixtures thereof calculated as MgO , MnO , Ni_2O_3 , and Co_2O_3 respectively.

3. A magnetic core formed of the thermal reaction product of a mixture of metal oxides in particle form present in the ratio of 60-75 percent by weight iron oxide calculated as Fe_2O_3 , 10-25 percent by weight zinc oxide calculated as ZnO , and 10-25 percent by weight of the oxide of a metal selected from the group consisting of magnesium, manganese, nickel and cobalt and mixtures thereof calculated as MgO , MnO , Ni_2O_3 and Co_2O_3 respectively, and up to 12 percent by weight of the oxides of lead and titanium calculated as PbTiO_3 .

4. A magnetic core formed of the product of reaction at a temperature within the range of 1900-2200° F. between lead metatitanate in amounts up to 12 percent by weight with the product of reaction at a temperature range within 2200-2500° F. of iron oxide calculated as Fe_2O_3 , zinc oxide calculated as ZnO and the oxide of a metal selected from the group consisting of magnesium, manganese, cobalt, nickel and mixtures thereof calculated as MgO , MnO , Co_2O_3 and Ni_2O_3 respectively.

5. A magnetic core material formed of the product of reaction at a temperature within the range of 1900-2200° F. between the oxides of lead and titanium calculated as lead metatitanate with the product of the reaction at a temperature within the range of 2200-2500° F. between metal oxides present in the ratio of 60-75 percent by weight iron oxide calculated as Fe_2O_3 , 10-25 percent by weight zinc oxide calculated as ZnO , and 10-25 percent by weight of the oxides selected from the metals consisting of magnesium, manganese, nickel, cobalt and mixtures thereof calculated as MgO , MnO , Ni_2O_3 and Co_2O_3 respectively.

6. In the method of preparing materials for magnetic cores, the steps of firing a mixture of iron oxide calculated as Fe_2O_3 , zinc oxide calculated as ZnO , and the oxides of a metal selected from the group consisting of magnesium, manganese, cobalt, nickel and mixtures thereof calculated as MgO , MnO , Co_2O_3 and Ni_2O_3 respectively at a temperature within the range of 2000-2500° F. for more than 2 hours and thermally reacting the reaction product in finely divided form with lead metatitanate at a temperature within the range of 1900-2200° F. for more than 1 hour.

7. In the method of preparing materials for magnetic cores, the steps of firing a mixture of iron oxide calculated as Fe_2O_3 , zinc oxide calculated as ZnO , and the oxides of a metal selected from the group consisting of magnesium, manganese, cobalt, nickel and mixtures thereof calculated as MgO , MnO , Co_2O_3 and Ni_2O_3 respectively at a temperature within the range of 2000-2500° F. for more than 2 hours and thermally reacting the reaction product in finely divided

form with lead metatitanate in amounts ranging up to 12 percent by weight of the composition at a temperature within the range of 1900–2200° F. for more than one hour and then cooling the reaction mixture gradually to a temperature of 600° F. and then to room temperature.

8. In the method of preparing a magnetic core, the steps of reacting a mixture of finely divided metallic oxides present in the ratio of 60–75 percent by weight iron oxide calculated as Fe_2O_3 , 10–25 percent by weight zinc oxide calculated as ZnO , and 10–25 percent by weight of the oxides of a metal selected from the group consisting of manganese, magnesium, nickel and cobalt calculated as MnO , MgO , Ni_2O_3 and Co_2O_3 respectively at temperatures in the range of 2200–2500° F. for more than 2 hours, subdividing the reaction product, reacting the subdivided reaction product with up to 8 percent by weight of lead and titanium oxides calculated as PbTiO_3 in combination with sufficient resinous binder temporarily to hold the particles in predetermined shape until the particles become thoroughly bonded at reaction temperature of 1900–2200° F., and then cooling the molded core gradually to room conditions.

9. In the method of preparing a magnetic core, the steps of reacting a mixture of finely divided metallic oxides present in the ratio of 60–75 percent by weight iron oxide calculated as Fe_2O_3 , 10–25 percent by weight zinc oxide calculated as ZnO , and 10–25 percent by weight of nickel oxide calculated as Ni_2O_3 at temperatures in the range of 2200–2500° F. for more than 2 hours, subdividing the reaction product, reacting the subdivided reaction product with up to 8 percent by weight of lead and titanium oxides calculated as PbTiO_3 in combination with sufficient resinous binder temporarily to hold the particles in predetermined shape until the particles become thoroughly bonded at reaction temperature of 1900–2200° F., and then cooling the molded core gradually to room conditions.

10. In the method of preparing a magnetic core, the steps of reacting a mixture of finely divided metallic oxides present in the ratio of 60–75 percent by weight iron oxide calculated as Fe_2O_3 , 10–25 percent by weight zinc oxide calculated as ZnO , and 10–25 percent by weight cobalt oxide calculated as Co_2O_3 at temperatures in the range of 2200–2500° F. for more than 2 hours, subdividing the reaction product, reacting the subdivided reaction product with up to 8 percent by weight of lead and titanium oxides calculated as PbTiO_3 in combination with sufficient resinous binder temporarily to hold the particles in predetermined shape until the particles become thoroughly bonded at reaction temperature of 1900–2100° F., and then cooling the molded core gradually to room conditions.

11. In the method of preparing a magnetic core,

the steps of reacting a mixture of finely divided metallic oxides present in the ratio of 60–75 percent by weight iron oxide calculated as Fe_2O_3 , 10–25 percent by weight zinc oxide calculated as ZnO , and 10–25 percent of a mixture of manganese oxide calculated as MnO and magnesium oxide calculated as MgO at temperatures in the range of 2200–2500° F. for more than 2 hours, subdividing the reaction product, reacting the subdivided reaction product with up to 8 percent by weight of lead and titanium oxides calculated as PbTiO_3 in combination with sufficient resinous binder temporarily to hold the particles in predetermined shape until the particles become thoroughly bonded at reaction temperature of 1900–2200° F., and then cooling the molded core gradually to room conditions.

12. In the method of manufacturing a bonded magnetic core, the steps of thermally reacting a mixture of finely divided metallic oxides present in the ratio of 60–75 percent by weight iron oxide calculated as Fe_2O_3 , 10–25 percent by weight zinc oxide, calculated as ZnO , 10–25 percent by weight of the oxide of a metal selected from the group consisting of magnesium, manganese, cobalt and nickel and mixtures thereof calculated as MgO , MnO , Co_2O_3 and Ni_2O_3 respectively, for at least two hours at a temperature in the range of 2200–2500° F., subdividing the reaction product to particle form, thermally reacting a mixture of the subdivided reaction product with up to 8 percent by weight lead metatitanate after forming the particles to predetermined shape with less than 8 percent by weight organic resinous binder which functions as a temporary binder until an interbonded relation is established by thermal reaction for over one hour at a temperature within the range of 1900–2200° F., and then cooling the product at the rate of about 100° F. per hour until a temperature of 600° F. is reached, and then cooling to room temperature.

13. A magnetic core prepared by the method of claim 7.

14. A magnetic core prepared by the method of claim 12.

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