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P. ROBINSON

3,065,181

INDUCTOR MATERIALS

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Fig. 1

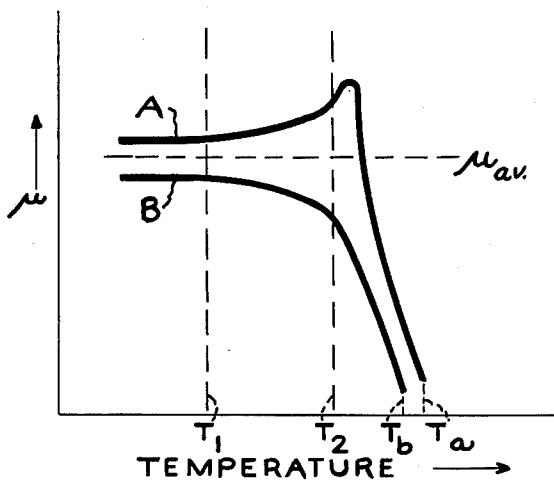


Fig. 3

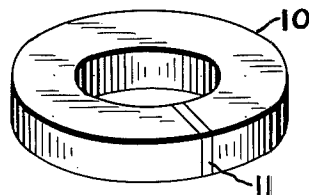


Fig. 4

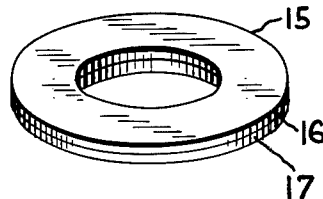
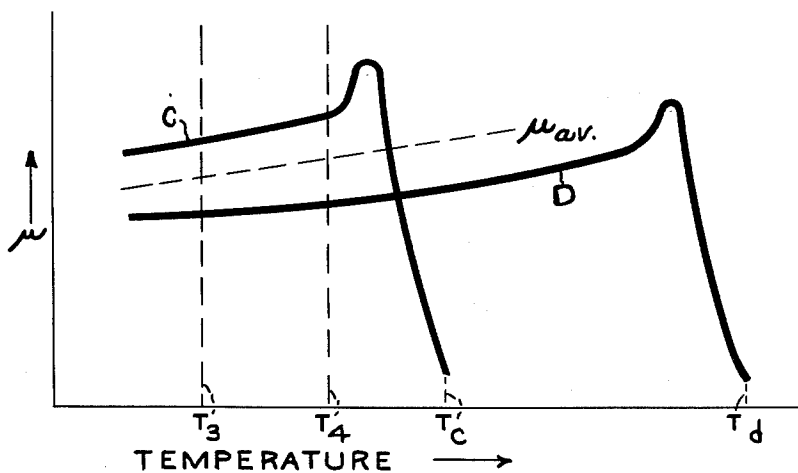


Fig. 2



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3,065,181

INDUCTOR MATERIALS

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1 Claim. (Cl. 252-62.5)

This invention relates to inductor materials and cores having exceptional temperature stability, and more particularly to ferrite materials and ferrite cores having a controlled temperature coefficient of permeability over the operating temperature range. This application is a continuation in part of my copending application, Serial No. 293,606, filed June 14, 1952, now abandoned.

It is known that the inductance of a coil wound on ferrite cores, such as toroidal or other closed magnetic path cores, varies with the permeability of the core material. This in turn varies according to the temperature of the core. In order to overcome difficulties resulting from the temperature coefficient of permeability varying through the range of temperatures in which a ferrite core is used, it has been the practice to introduce an air-gap into the magnetic path, causing both the permeability of the core material and its temperature coefficient to decrease. The effective permeability is not altered as much by this expedient as the latter temperature coefficient, and is not lowered to an impractical value by virtue of the high inherent initial permeability present with ferromagnetic ferrite materials.

The introduction of air gaps into ferrite cores creates a number of mechanical and electrical disadvantages, other than loss of permeability. It is difficult to create gaps of uniform dimension and perfectly parallel side walls. When the gaps are not uniform, there is a non-uniformity of the magnetic flux between the exposed ends of the core and a concentration of flux at the portions of the walls which are closest to one another, thereby causing increased hysteresis losses. Also the magnetic strength of a core is lowered to a substantial extent by an airgap.

Prior attempts at providing a control over the temperature coefficient of permeability have included controlling the time and temperature of sintering of a homogeneous ferrite. This has resulted in an unsatisfactory product in that control was evident only in a body having a low (200-500) initial permeability.

Another attempt in the prior art has been the use of a binder to hold two different magnetic materials together. This has produced a product having very poor magnetic properties, in that the particles are spaced from one another by too great a distance.

Still another attempt has been to utilize a solvent such as silica or lead oxide to provide a glossy intercrystalline phase. However, this attempt has not been satisfactory, in that if enough solvent is mixed with the ferrite to provide any control, the magnetic properties will be diminished to the point of non-utility by the presence of the large non-magnetic phase.

It is an object of the present invention to overcome the foregoing and related disadvantages present in ferromagnetic ferrite cores utilizing an air gap, and in the prior art attempts to eliminate the air gap. A further object of the invention is to produce ferrite core materials having controlled predictable temperature coefficients of permeability over a given range of operating temperatures and at the same time maintain excellent magnetic properties. Other objects of the invention, as well as the advantages of it, will be apparent from the following description and claims as well as the accompanying drawing wherein:

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FIGURE 1 shows the temperature permeability curves for two ferrites used in a single core of the invention;

FIGURE 2 shows the same curves for two other individual separate ferrite materials;

FIGURE 3 pictures a toroidal core utilizing a ferrite gap in place of an air gap; and,

FIGURE 4 illustrates a composite core composed of laminations of two separate ferrite materials.

The above objects are attained in the production of a core having a controlled temperature coefficient of permeability over a predetermined operating range of temperatures by utilizing together at least two different ferrite materials having different Curie temperatures and/or temperature-permeability curves, so as to produce a composite body in which the two ferrites retain their identity and in which the body has a permeability which is either constant over a predetermined range of temperatures, or varies in a predictable manner throughout this range.

In a more particular degree, the objects of this invention are attained by uniting two nickel-zinc or cobalt-zinc ferrites which have incomplete spinel formations in the presence of a flux in such a manner that grain growth is inhibited and the two ferrites retain their individual identity.

The double ferrites produced in accordance with this invention may be utilized to produce cores which fall within three general embodiments. The first and preferred embodiment of my invention is a complete toroid of uniformly heterogeneous double ferrite. However, my invention may also be practiced in a core of two segments. One segment, the larger of the two, may be composed of a double ferrite of one composition, while the other segment may be of another double ferrite or a single ferrite. A third possible embodiment is concerned with cores made in layers. Each layer may be composed of one of my double ferrites, or one layer only may be a double ferrite.

Before considering the invention further, it is necessary to discuss slightly the ferrites and their composition and behavior. In general, the term ferrite defines a crystalline material which is the reaction product of a metallic oxide and ferric oxide having the formula $MO \cdot Fe_2O_3$ wherein M stands for a bivalent metal. These ferrites assume two different crystalline structures. Those assuming the so-called inverse spinel configuration are ferromagnetic in nature. Among them are nickel, cobalt, copper, manganese, lead, barium, and strontium ferrites. The ferrites which assume the normal spinel structure such as zinc, cadmium, calcium, and tin ferrites are non-magnetic in nature. For a further discussion of subject matter, the reader is referred to pages 244-249 of Bozorth's treatise on Ferro-magnetism.

For the purposes of this invention the process for making ferrites may be considered to involve two steps, i.e., the formation of the spinel and the process of grain growth. The formation of a spinel has been shown by X-ray diffraction to reach a definite equilibrium at a definite temperature. For example in nickel-zinc or cobalt-zinc ferrites the spinel formation is roughly 30 to 40% complete at 1100° C. and 95% complete at 1400° C. The process of grain growth may be determined by particle size, pressure of molding, time and temperature of sintering, and the presence or absence of an inhibitor. This invention may be practiced with any typical grain size of nickel, or cobalt, and zinc ferrites fired initially at 1400° C. to form the spinel.

For the purpose of this specification, the term "Curie point" is to be considered as designating the point at which a ferromagnetic material becomes paramagnetic in nature. For convenience, this term is often taken as designating the temperature at which the permeability of

a ferrite is approximately one-tenth of its maximum value. The Curie points in the ferrites which are commercially sold are considerably lower than those points in the pure ferrite compounds. These commercial mixtures can be designated with accuracy as homogeneous mixed crystal materials. In effect, this designation means that they are homogeneous and that no second phase can be determined by normal microscopic examination.

In general the uniformly heterogeneous double ferrite of my invention is obtained by separately producing two or more nickel-zinc or cobalt-zinc ferrites by conventional milling and sintering procedures known to the art. The ferrites should fit the basic formulation of one mol of divalent metal oxide to one mol of ferric oxide. Specific formulations are utilized to produce ferrite material having different initial permeability and Curie points. Broadly speaking the higher the zinc content, the higher the initial permeability and the lower the Curie point. These fully sintered ferrites are then ground according to conventional ball-milling techniques. Portions of two or more distinct ferrites are then combined and blended in a ball-mill compressed into the form of a toroid and are fired at a temperature such that porosity of 30-40% is achieved. This porosity is in effect only another method of expressing the extent of completeness of the spinel formation. These porous toroids are then impregnated with a flux which acts as a grain growth inhibitor and are refired at a temperature approaching or slightly more than the eutectic between the flux and the ferrite. This produces a core of ferrite material in which the distinct ferrites which make up the core have retained their identity and yet one in which the porosity has been reduced to such an extent that the magnetic properties closely approach those initial ferrites making up the core. In the second firing, that is the firing together of the two distinct ferrites, care must be taken that the reaction is not allowed to continue to a point where the two ingredients diffuse into a crystalline structure and thereby form a homogeneous mass. It is the essence of this invention that the ferrites retain their identity and while the final porosity is such that the crystals are in intimate contact they retain their distinct crystal structure. The manner of cooling following this firing can be carefully controlled in the manner known to the prior art so as to prevent or regulate any second phase formation.

Specific examples of this invention may be best understood by reference to the drawings. Two nickel-zinc ferrites having different initial permeabilities and different Curie temperatures are utilized to provide the double ferrite of my invention. The ferrite of curve A in FIGURE 1 may be a 20:30:50 nickel:zinc:ferrite produced by starting with 20 mol percent of nickel oxide, 30 mol percent of zinc oxide and 50 mol percent ferric oxide blended and ball-milled for 8 hours. The powder may be pressed at roughly 15,000 lbs. per square inch and sintered for at least one hour and not more than six hours in air at 1400° C. The ferrite of curve B may be produced in substantially identical manner with a ferrite of curve A with the exception that the zinc percent is increased to 34 mol percent and the nickel percent correspondingly reduced to 16 mol percent. These ferrites have Curie temperatures of T_a and T_b , respectively, and are ground and mixed in equal proportions and are pressed together to form the toroid of the final product. Throughout the area between the two curves within the boundary temperatures T_1 and T_2 , the permeability of the final core is substantially constant, inasmuch as the average permeability μ_{av} remains unchanged at all temperatures throughout this range. This composite ferrite is then sintered in a relatively low temperature of 1200° C. until 30% porosity exists. This porous body is then impregnated with a flux in liquid form. This flux can be bismuth nitrate dissolved in nitric acid or it can be ethyl silicate or it can be copper acetate or in fact any similar material which decomposes at a relatively low tem-

perature to provide an oxide which in turn can act as a flux. For the purposes of this example, bismuth nitrate dissolved in nitric acid was used as the flux. The porous bodies were then resintered at the temperature of the eutectic between the ferrite and the flux or to slightly exceed the eutectic temperature which in the case of this example is roughly 1300° C. The flux reacts at the grain boundaries of the ferrite to reduce the melting point locally and to cause the ferrite to shrink without seriously affecting the bulk properties of the ferrite. At the same time the sintering temperature is so low that appreciable diffusion of ions making up the ferrite will not take place.

In FIGURE 2 the permeability temperature curves for two other ferrites C and D, which are present in equal proportions in a product such as is described above, are shown. The ferrite of curve C is a 25:25:50 cobalt:zinc:ferrite sintered at 1450° C. to give a 95% complete spinel formation and a definite grain size. The ferrite of curve D is a 20:30:50 cobalt:zinc:ferrite also sintered at 1450° C. These ferrites have Curie temperatures T_c and T_d , respectively. Throughout the temperature range T_3 - T_4 , the average permeability μ_{av} of the composite core increases at a constant slope. Ferrites C and D are ground milled and pressed together at 15,000 lbs. per square inch and are fired at 1250° C. to provide a 30% porosity. These porous bodies are filled with a flux, ethyl silicate and resintered at 1320° C. to produce the final body which is a uniformly heterogeneous double ferrite. Cores having such predictable temperature coefficients of permeability can be used to advantage in many applications.

The second embodiment differs from the first in that the reaction of the component materials need not be carried out as indicated above. During the pressing of one of the double ferrites, a small segment of a second ferrite either double or single can be introduced into a toroid by purely physical means to replace an air gap which is normally machined in such a toroid following its firing. The size of the gap, of course, varies with the various design factors involved. In general, in the three inch toroid a gap of about a quarter inch filled with a second ferrite component is satisfactory. Such an arrangement shown in FIGURE 3 wherein there is illustrated a ferromagnetic ferrite core 10 having a second ferrite segment 11 in place of an air gap.

The third embodiment of this invention relates to ferrites in which two or more separate ferromagnetic ferrite bodies at least one of which is one of my double ferrites, are placed in physical contact with one another during the formation of a "laminated" core. To cut the hysteresis losses in such cores, the individual components are preferably made to extremely close tolerances, and in some cases they are even lapped so as to produce absolutely flat surfaces which fit together. This embodiment of the invention is pictured in FIGURE 4 wherein there is shown a ferrite core 15 composed of two separate flat rings 16 and 17 which fit as closely as possible against one another. It is to be realized that the third embodiment of the invention is not limited to toroids or to cores employing bodies having flat, adjacent surfaces. Thus, with the invention it is possible to form cores having one ferrite completely surrounding the outside of a first ferromagnetic ferrite. Such cores can take the shape of one toroid completely surrounding a first toroid or a ferrite rod completely covered with a second rod. A multiplicity of layers can be used in all of the modifications of this embodiment of the invention.

Inasmuch as the production of the individual ferrites is well-known, it is not considered necessary to discuss them in detail at this point. Reference is made to the above cited Bozorth text, to the Snoek Patents 2,452,529, 2,452,530, and 2,452,531, and to the monograph "Recent Developments in Ferromagnetic Materials" by Snoek.

It will be realized that the specific proportions of ferrites employed in any core of any of the embodiments

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set forth will vary with a number of separate factors such as the composition of the individual ferrite components, the reaction conditions, and the like. Those skilled in the art are able to calculate these proportions for any given application with a minimum of difficulty. Frequently, it is preferable to utilize one homogeneous mixed crystal ferrite having a Curie temperature above room temperature with a second similar ferrite having a Curie temperature at room temperature or lower in forming the cores of this invention having substantially constant permeabilities. It is contemplated that the broad teachings of the invention will be extended to a number of ferromagnetic compositions besides the ferrites such as the ferromagnetic composition FeMgAlO_4 , and $\text{FeMg}_{1.5}\text{Ti}_{0.5}\text{O}_4$, and lanthanum and strontium magnetites.

As many apparently widely different embodiments of my invention may be made without departing from the spirit and scope hereof, it is to be understood that my invention is not limited to the specific embodiments hereof except as defined in the appended claim.

I claim:

The method of producing a body of uniformly heterogeneous ferromagnetic conductor material of a composite ferrite having a controlled temperature coefficient of permeability over a predetermined temperature range comprising the steps of mixing and blending together particles of two distinct nickel-zinc ferrites of different initial permeabilities and different Curie temperatures with different temperature coefficient curves over a predetermined temperature range in a formulation of nickel oxide in a range of 16 to 20 mol percent, zinc oxide in a range of 30 to 34 mol percent and 50 mol percent of ferric oxide, said two distinct nickel-zinc ferrites being mixed and blended in such proportion that the mixture is more uniform, compressing said mixture into a body, sintering the composite ferrites of said body at a relatively low temperature of about 1200°C ., producing an incomplete spinel formation of the composite ferrites by said sintering and thereby reducing the porosity of said body to 30-40%, impregnating the sintered, partially porous, incomplete spinel ferrite body with a grain growth inhibitor flux selected from the group consisting of a bismuth nitrate dissolved in nitric acid, ethyl silicate, and copper acetate; said flux reacting at the grain boundaries

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of the ferrites to reduce the melting point locally, subsequently firing the body at a temperature of about 1300°C . and exceeding the eutectic between the flux and the composite ferrites and less than the sintering temperature of the composite ferrites, said subsequent firing of said composite ferrites taking place with inhibited grain growth, whereby the ferrites retain their identity and the body has a substantially constant permeability over a predetermined range of temperatures.

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