July 7, 1964 B. C. COAD 3,140,172

PRODUCTION OF ALLOY MATERIALS

Filed June 30, 1961

FIG. 1.

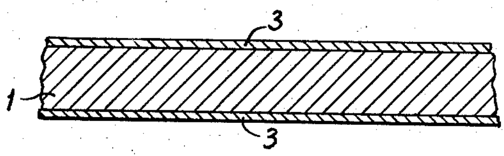


FIG. 2.

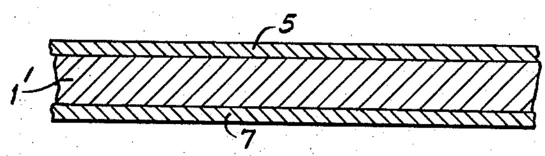


FIG. 3.

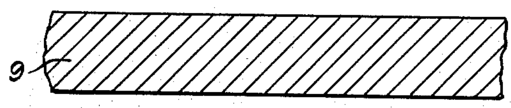


FIG. 4.

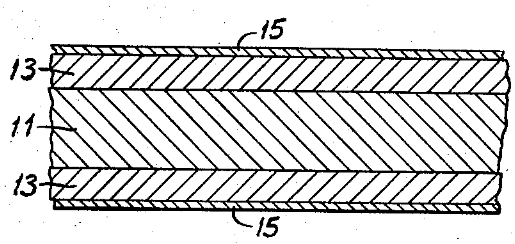


FIG. 5.

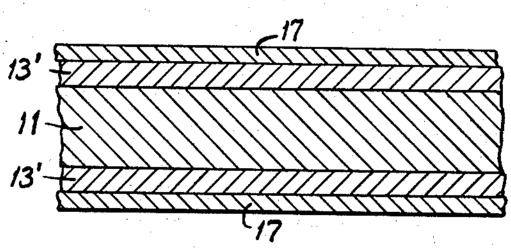
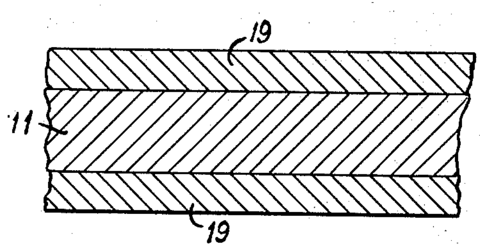


FIG. 6.



Brian C. Coad,
Inventor.
Koenig, Pope, Senniger and Powers,
Attorneys.

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PRODUCTION OF ALLOY MATERIALS

Brian C. Coad, Attleboro, Mass., assignor to Texas Instruments Incorporated, Dallas, Tex., a corporation of Delaware

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This invention relates to an improved method for the production of alloy materials, and to the fabrication of improved products made therefrom, and more particularly to alloys of iron and aluminum.

Among the several objects of the invention may be noted the provision of an improved, simpler and less costly method for obtaining alloys, particularly those such as iron and aluminum, which may be quite brittle because, for example, of a high aluminum content, the compositions of which by means of the invention are more precisely controllable than heretofore; the provision of such a method which avoids need for complex melting techniques; the provision of a method for obtaining products incorporating iron-aluminum alloys in bonded form which avoids former difficulties encountered in attempts to cold-work such iron-aluminum brittle alloys in the required bonding operations; and the provision of iron-aluminum alloy products which have desirable physical characteristics as required for various uses. Other objects and features will be in part apparent and in part pointed out hereinafter.

The invention accordingly comprises the elements and combinations of elements, steps and sequence of steps, features of construction and manipulation, and arrangements of parts which will be exemplified in the products and methods hereinafter described, and the scope of which will be indicated in the following claims.

In the accompanying drawings, in which several of various possible embodiments of the invention are illustrated,

FIG. 1 is a diagrammatic cross section of a typical bonded composite sheet of aluminum and iron useful for carrying out the invention;

FIG. 2 is a view similar to FIG. 1, illustrating a conversion of the FIG. 1 sheet after a first heating step;

FIG. 3 is a view similar to FIGS. 1 and 2, showing a final conversion of the FIG. 2 sheet into a finished product after a second heating step;

FIG. 4 is a diagrammatic sectional view of a starting composite sheet for producing magnetizable elements such as used for transformers and the like;

FIG. 5 is a view similar to FIG. 4, illustrating a conversion of the FIG. 4 sheet after a first heating step; and

FIG. 6 is a view similar to FIGS. 4 and 5, showing a final conversion of the FIG. 5 sheet into a finished product after a second heating step.

Corresponding reference characters indicate corresponding parts throughout the several views of the drawings.

While the invention has certain characteristics broadly applicable to various metals, it is particularly useful in the field of iron-aluminum alloys. Iron-aluminum alloy materials are characterized by good oxidation resistance, high electrical resistance and low density, as compared with most other iron alloys. They also have good so-called soft magnetic properties, meaning that they are easily magnetized under low coercive forces and that they spontaneously and rapidly demagnetize themselves. In the past, however, problems have been encountered in making such alloys by conventional melting techniques because of the disparity in the melting temperatures of the components. This made it difficult to achieve precise control of the compositions. Another difficulty manifested itself with such alloys when they contained substantial amounts of aluminum in that then these alloys became more difficult to cold-work in view of their increased brittleness. According to the present invention, solid-state type alloying techniques are employed in a manner to obtain closer control of compositions and so as to avoid the above-mentioned cold-working difficulties in making up certain types of products. This is in addition to the ability to obtain at lower cost a more closely controlled composition of the alloy.

Briefly, in its iron-aluminum alloy application, the present invention in one form is carried out by bonding one or more sheets of aluminum to an iron base sheet, then reducing the resulting composite material thus obtained to finished size by rolling or otherwise, after which the composite is heat-treated a first time to produce intermetallic compounds of iron and aluminum on the iron base. Thereafter, the composite is heat-treated a second time so as to bring about an alloying of the intermetallic compound with the iron, thus producing a solid solution of aluminum and iron. This constitutes the desired alloy. The invention may be carried out to produce the desired alloy in single-sheet form or to produce it in attachment with other sheets, such as magnetic alloy sheets used for transformer cores or the like, in which cases a composite material results, the brittle iron-aluminum components requiring no complex or cumbersome working to get them bonded to the core sheets. The invention may also be carried out in steps employing powder forms of certain components, as will be described in more detail below.

Referring now more particularly to FIG. 1 of the drawings, there is shown at numeral **1** an iron base sheet which may be composed of a low carbon steel, for example, 1010 steel. To this has been metallurgically bonded two cladding sheets **3** of aluminum. Appropriate cladding means and methods are, for example but without limitation, disclosed in United States Patents 2,691,815 and 2,753,623. Solid-phase bonding, such as shown in these patents, is employed in carrying out the invention because no extraneous material is involved at the bond, other than intermetallic compounds of the metals which are preliminarily bonded, such metals being those which ultimately make up the desired alloy. The bonded sheet is rolled down to a total thickness of, for example, .005 inch, which is to say that the sheet may have been rolled down from a previously bonded iron-aluminum combination of greater thicknesses of its various layers. The thickness ratio of aluminum-iron-aluminum as rolled down is, for example, 10–80–10. This provides approximately 8% of aluminum by weight in the composite sheet shown in FIG. 1, according to this example.

The .005 inch composite sheet example thus described and illustrated in FIG. 1 is subjected to a first heating step by heating in air for an hour or so to a temperature in the range from 1,000° F. to 1,500° F. Since the melting point of aluminum is approximately 1,200° F., this temperature range covers cases in which the aluminum does not melt during the first heating step, and cases wherein it does so. It has been found that even when the temperature of the first heating step exceeds the melting point of aluminum that little or no difficulty is encountered with run-off of the aluminum during this first heating step. While I do not wish to be bound by any theories, it is believed that this phenomenon may at least in part be due to the thinness of the aluminum layer, which permits surface tension to preponderate over any tendency to run-off, and due in part to the presence of an oxide film on the aluminum cladding which also helps to prevent run-off.

FIG. 2 shows the result of the first heating step above described which is the conversion of the aluminum layers **3** of FIG. 1 into layers **5** and **7**, shown in FIG. 2. These are constituted by intermetallic compounds of iron and aluminum, such as, for example, Fe_3Al_5 and others. In FIG. 2 the base is numbered **1'**.

Next a diffusion or homogenizing step is employed wherein the product illustrated in FIG. 2 is transferred to a controlled-atmosphere furnace having, for example, an inert or nonoxidizing atmosphere such as argon to prevent further oxidation such as may have occurred in the first heating step. This furnace is operated at or above the melting point of the intermetallic compounds constituting the cladding sheets **5** and **7** of FIG. 2. Thus, the approximate melting point of the iron-aluminum intermetallic compound, for example, Fe_3Al_5, is 2,120° F. Therefore, a satisfactory operating temperature for the controlled-atmosphere furnace is 2,300° F. This is below the melting point of iron, which melts at approximately 2,800° F. Again, the layers of the intermetallic compounds do not tend to run off. Heating is continued at the selected temperature of 2,300° F., for example, for approximately four hours. The exact maximum temperature that may be carried in the controlled-atmosphere furnace depends somewhat upon the percentage of the ratio of aluminum to iron used, because the melting point of iron-aluminum alloy produced, varies somewhat with the percentage of aluminum therein. It is also intended not to exceed at any stage the melting point of the iron base sheet **1** or **1'**. Thus the desirable temperature range of operation of this furnace may be between 2,120° F. and the melting point of iron, as influenced by the amount of aluminum that it may have in it as this heating step proceeds. However, 2,300° F. is a satisfactory temperature. After the four-hour 2,300° F. heat treatment, the layers **1**, **5** and **7** of FIG. 2 become converted to a homogeneous solid solution alloy of iron and aluminum, being a true alloy of these components. This is illustrated at numeral **9** in FIG. 3.

An advantage of this method of making iron-aluminum alloys lies in the fact that an unalloyed composite such as shown in FIG. 1 can be annealed and readily formed, since no substantial brittle stage has been reached which would complicate such working or render the material unworkable. This is subsequently alloyed as described in connection with FIGS. 2 and 3 by the simple heat treatments described herein. This postpones the brittle condition until any desired working has been completed. Iron-aluminum alloys as made by the former all-liquid-phase method reach the brittle condition before working is possible, so that any working step required becomes costly and difficult and in some cases impossible. Former methods also have resulted in alloys and problems generally characterized by lack of homogeneity in the alloy, and by poor ductility and formability, all of which problems are obviated or at least minimized by means of the present invention.

The iron-aluminum ratio selected for the clad material of FIG. 1 is such as to obtain the percentage of aluminum desired in the finished alloy. In some instances the first heating step described in connection with converting from the FIG. 1 formation to the FIG. 2 formation may be omitted; but it is preferred that it be employed because it is believed that greater uniformity of composition and cross section may be achieved with the two-step process.

It should be understood that both the first and second heating steps described above may, in some cases, conveniently be carried out in a controlled, non-oxidizing atmosphere-furnace or in the air, if desired.

Other alternative examples of the above method are as follows:

As first and second alternatives, 1010 steel may be clad with 2S aluminum in 14–72–14 and 17–66–17 thickness ratios, to produce approximately 12% aluminum and 15% aluminum by weight of iron-aluminum alloys, respectively. The clad composites have been rolled to

.005 inch at the time that said ratios are obtained. The composites are then heated while loosely coiled for one hour at 1,500° F. in air to form iron-aluminum compounds including Fe_3Al_5; then four hours at 2,300° F. in argon, to produce the 12% and 15% alloys by diffusion, as above set forth.

As a third alternative, a starting layer may be employed consisting of bonded 2S aluminum, 310 stainless steel and 2S aluminum in a 5–90–5 thickness ratio, having a total thickness of .005 inch. This is first heated for one-half hour at 1,200° F. to form the desired intermetallic compounds; then heated for diffusion for two hours at 2,300° F. to make the alloy. In this case, both heating steps are carried out in air. The resulting aluminum content of the alloy is approximately 3.5%.

In FIGS. 4–6 is illustrated an application of the invention to the production of magnetic or magnetizable core, armature, field and like members for transformers, motors, generators and the like. In FIG. 4, numeral **11** illustrates a base sheet of, for example, any conventional magnetic alloy such as is now used for making transformer cores. An example of such an alloy is silicon iron. Clad to the magnetic alloy sheet **11** are sheets **13** of iron, and clad to these iron sheets are sheets **15** of aluminum. The iron of sheets **13** may be in the form of a low carbon steel such as 1010 steel and preferably in the form of substantially pure iron.

The thickness of the magnetic alloy base material **11** is arbitrary. Assume it to be 100 units. Then the ratio of thicknesses of the layers **15**, **13**, **11**, **13**, **15** may for example be 10–40–100–40–10. The over-all thickness of the FIG. 4 assembly will be that ordinarily desired for use of the final product such as, for example, a sheet useful as transformer core material. This thickness may for example be from .012 inch for medium size transformers.

The bonded assembly thus formed (FIG. 4) is first heated in air for one hour to a temperature in the range of from 1,000° F. to 1,500° F. This produces an intermediate material illustrated in FIG. 5, wherein the magnetic alloy base **11** carries on it layers of iron **13'**, and on these layers **13'**, layers **17** of intermetallic compounds such as Fe_3Al_5. Then the product of FIG. 5 is subjected to the second heat treatment in a protective or nonoxidizing atmosphere at 2,300° F. for approximately four hours, with the result shown in FIG. 6, wherein the magnetic alloy core material **11** is clad with layers **19** of iron-aluminum alloy. The FIG. 6 material then consists of the usual magnetic core material for transformers, faced with a bonded high-resistance iron-aluminum alloy **19**. The interleaved iron-aluminum alloy sheets, having a high resistance, reduce eddy-current losses in the electrical devices employing them in laminated form. It should be understood that the temperature and time for the second heating step are so selected in accordance with the thickness of the iron layers **13'**, **13'** so as to substantially avoid diffusion or penetration of the aluminum into the silicon iron layer **11**.

The invention may also be carried out with finely divided or comminuted starting materials instead of the solid sheet materials as in FIGS. 1 and 4. Thus, for example, finely divided aluminum and finely divided iron in a 50–50 ratio by weight are mixed thoroughly. The degree of comminution of the components is not critical. The particles may range in size from small pellets, chips or the like, to powder. The mixed components are then compacted into appropriate form by rolling, pressing or the like. Next the compacted form is heat-treated for example for one hour at 1,300° F. in an atmosphere, for example, of cracked ammonia, to sinter and produce solidified intermetallic compounds. The resulting intermediate product includes, for example, Fe_3Al_5. The compacted intermediate product is broken up by ball milling, again to form comminuted particles, which at this time becomes a finely divided form of the intermetallic compound. This is hereinafter referred to as powder A.

Next, finely divided iron is mixed with, for example, 24% by weight of powder A, to provide, if so desired, a final iron-aluminum alloy containing approximately 12% of aluminum by weight. A final iron-aluminum alloy having approximately 16% aluminum by weight would be obtained by mixing powdered iron with 32% by weight of powder A. In either case, the mixture of iron and powder A is compacted by rolling, pressing or the like to form a sheet, strip, pellet, shape or part of final desired configuration which is thereafter heat-treated for example for three hours at 2,250° F. This results in a homogeneous iron-aluminum alloy containing approximately 12% or 16% aluminum, as the case may be. It will be understood that, by employing powder proportions other than those described above, other proportions of aluminum may be obtained in the final iron-aluminum alloy.

If desired, one of the mixtures of finely divided iron and 24% (for example) by weight of powder A may, during compaction, be rolled onto and bonded to a suitable base material such as the magnetic alloy sheet above mentioned. Then upon applying the alloying heating step to the compacted mixture of finely divided iron and powder A as rolled on the base material, the mixture will be converted into the final iron-aluminum alloy, the alloy bonded to the base material.

In view of the above, it will be seen that the several objects of the invention are achieved and other advantageous results attained.

As various changes could be made in the above products and methods without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

I claim:

1. The method of producing an alloy, comprising solid-phase bonding and rolling to a small thickness, on the order of a few thousandths of an inch, layers of all of the constituents required in individual thicknesses adapted to supply the correct proportions thereof in the desired alloy, heating the bonded layers to form an intermediate multi-layer sheet in which substantially all of one constituent and some of the other are converted to at least one intermetallic compound in at least one bonded layer, some of the constituent material remaining as another bonded layer, and then heating the layers in the multi-layer state at a temperature adapted to convert them from their multi-layer state to the desired alloy in a single-layer state.

2. The method according to claim 1, wherein one of said layers comprises aluminum and the other comprises iron, the total thickness of the aluminum and iron layers being selected to be on the order of a few thousandths of an inch.

3. The method of producing magnetizable core material comprising cladding by solid-phase bonding on a magnetizable alloy sheet at least two sheets having multi-layer constituents of different metals, said sheets being also solid-phase bonded, each of the constituent layers being of small thicknesses on the order of a few thousandths of an inch and in a ratio adapted to supply the correct proportions thereof in desired alloy cladding, heating the bonded layers to form an intermediate multi-layer sheet in which substantially all of one constituent and some of the other are converted to at least one intermetallic compound, and then heating all of the layers to convert them to the desired alloy.

4. The method of producing magnetic core material comprising solid-phase bonding to a first sheet of a magnetic alloy second and third sheets of solid-phase bonded layers of aluminum and iron, said second and third layers being of a total thickness on the order of a few thousandths of an inch, the respective thicknesses of said second and third layers adapted to supply the correct proportions thereof in a desired alloy facing on the first layer, heating the three bonded layers to form an intermediate multi-ply sheet in which the first layer is preserved and substantially all of the third layer and some of the second layer are converted to at least one intermetallic compound, and then heating all of the layers at a temperature sufficient to convert the second and third layers to form the desired alloy in unit-layer form on the first layer.

5. The method of producing magnetizable clad core material in sheet form comprising cladding by solid-phase bonding on each of opposite sides of a magnetizable alloy sheet one double-layer sheet, each sheet being composed of different constituent metals, each of said sheets being of small thickness on the order of a few thousandths of an inch, the component layers of each sheet being in thicknesses adapted to supply the correct proportions thereof in desired alloy cladding, heating all of the bonded layers to form an intermediate multi-layer sheet on each side of the magnetizable alloy sheet in which substantially all of one constituent and some of the other are converted to at least one intermetallic compound, some of the constituent material remaining as another layer, and then heating all of the layers to convert them to the desired alloy cladding on each side of the core material.

6. The method of producing an alloy comprising mixing portions of different finely divided particles of first and second metals, pressing the mixed portions to compact them, heat-treating said compacted portions at a sintering temperature adapted to form a solid including in its constituent materials an intermetallic compound, finely dividing said sintered solid into finely divided material, mixing the last-named finely divided material with an additional amount of one of said finely divided metals to form a mixture of particles to supply all of the constituents required in a proper proportion of the desired alloy, pressing the particles of said last-named mixture by rolling them against a sheet of metallic base material, and heat-treating said sheet of base material with the mixture pressed thereon to bond said mixture to the base material and to convert the mixture into the desired alloy.

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