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**FERRITIC ALLOYS AND METHODS OF MAKING AND FABRICATING SAME**

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The present invention relates to a novel group of high temperature and magnetic ferritic alloys and to methods of making same.

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

Most conventional high temperature alloys contain large percentages of such strategic materials as nickel, chromium and cobalt. Accordingly, the principal object of the present invention is to provide high temperature aluminum-iron base alloys which are free of strategic materials.

Certain aluminum-iron alloys containing a minimum of about 10% Al have previously been described as possessing excellent resistance to oxidation when exposed to high temperatures. These alloys, however, have suffered from the fact that they are weak in structure and lack high temperature strength. Additionally, they do not possess sufficient malleability to permit them to be formed into any desired shape.

For example, considerable difficulty has been encountered in processing Al-Fe base alloys of more than 10% Al content into satisfactory cold-rolled sheets. Even with the inclusion of a strengthening additive, such as molybdenum, it has not hitherto been possible to produce usable and workable sheet materials due to the inherently brittle nature of the Al-Fe base alloys of at least 10% Al content.

In view of the above-mentioned difficulties, another object of the invention is to provide Al-Fe base alloys containing more than 10% Al which possess exceptionally high strength, as well as oxidation resistance, at elevated temperatures.

A further object of the invention is to provide Al-Fe base alloys possessing sufficient malleability to be shaped, as, for example, by rolling or extrusion.

An additional object is to provide Al-Fe base alloys of at least 10% Al possessing magnetic properties, substantially better oxidation resistance and tensile strength at high temperatures than most ferritic-type material currently being used in high temperature applications and other highly desirable characteristics such as a high degree of hardness, wear resistance and electrical resistivity.

Still another object of the invention is the provision of novel methods for producing the high strength, oxidation resistant alloys of the type referred to above, as well as methods for fabricating same into desired shape.

A more specific object is to provide methods for cold and/or hot rolling the ferritic alloys of the invention into physically sound and otherwise highly desirable sheet materials.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the

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invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

Broadly stated, the foregoing objects are accomplished by means of a process including the following steps: (1) producing a melt consisting essentially of 10 to 18% aluminum, 1 to 5% of a strengthening additive, particularly molybdenum, and the remainder iron; (2) casting said melt into the desired shape; (3) solidifying the same to produce a fine-equiaxed cast grain structure; (4) annealing the thus-solidified product to eliminate any internal stresses resulting from the solidification; and (5) slowly cooling the annealed product to a temperature below which the formation of internal stresses is substantially eliminated. Thereafter, the product is subject to further treatment, e. g., hot and/or cold rolling, as described in detail below, depending upon the nature of the final article desired.

The process, as briefly set forth above, is hereinafter described with particular reference to the production of sheet materials from the resulting alloys by subsequent rolling operations, since this is one of the most important aspects of the invention. However, it will be appreciated that the invention is not limited to the production of such sheet materials. For instance, the alloy may be cast into its final form in step (3) supra. Alternatively, the product obtained from steps (1) to (5) may be extruded into shapes and drawn into wire instead of being rolled into sheet material.

Regardless of final form, the alloys of the invention are characterized by their unusually high strength at elevated temperatures. For example, stress-rupture tests on 0.020" cold-rolled sheet material, prepared according to the invention, gave the following results using a preferred alloy composition of 15–16% Al, about 3% Mo and the rest essentially Fe.

TABLE 1

*Stress-rupture properties*

| Test Temperature | Stress (p. s. i.) | Stress Life (hours) | Percent Elongation $\frac{1}{4}$ Gage Length | Remarks                                               |
|------------------|-------------------|---------------------|----------------------------------------------|-------------------------------------------------------|
| 1,100° F.-----   | 30,000            | 374                 | 20.4                                         | Broke in grip.                                        |
| 1,100° F.-----   | 25,000            | 670                 | 33.3                                         |                                                       |
| 1,100° F.-----   | 20,000            | 1,656               | 62.5                                         |                                                       |
| 1,100° F.-----   | 15,000            | 2,010               | 10.4                                         |                                                       |
| 1,100° F.-----   | 10,000            | 2,010               | 2                                            | Tests stopped without breakage.                       |
| 1,200° F.-----   | 30,000            | 16.6                | 12.5                                         |                                                       |
| 1,200° F.-----   | 25,000            | 47.6                | 22.9                                         |                                                       |
| 1,200° F.-----   | 20,000            | 72.9                | 37.5                                         |                                                       |
| 1,200° F.-----   | 15,000            | 250                 | 54.3                                         |                                                       |
| 1,200° F.-----   | 10,000            | 796                 | 233                                          | Test allowed to run without load for about 100 hours. |

The success of the invention is due to several factors. First, it is essential that the casting be solidified at a sufficiently slow rate, or in some equivalent way, to form a fine-equiaxed cast grain structure, i. e., an equiaxed structure of minimum grain size. Otherwise, i. e., if a large columnar cast grain structure is obtained, the resulting alloy does not possess maximum tensile strength and during rolling or other fabrication, hot tearing or cracking will occur. These disadvantages do not occur when the cast product has a fine-equiaxed cast grain structure.

With respect to this phase of the invention, it should be noted that while the molybdenum or other strengthening additive improves the high temperature tensile strength of the present alloys, it does not have any effect on the nature of the grain structure. That is, the molybdenum does not function as a grain refiner, the desired grain refinement being obtained instead through regulat-

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ing the rate of solidification, as discussed in more detail below.

It is also essential to the success of the invention that the cast product be subjected while red hot to an annealing operation followed by slow cooling. These steps eliminate internal stresses which occur during solidification and cause cracking of the casting when it is machined, rolled or otherwise fabricated.

The elimination of internal stresses, as described above, can be carried out in several ways. One especially desirable technique comprises placing the red hot casting, just as soon as it is solid enough to be moved, in a furnace kept at a temperature of 600–1100° C., preferably 1050° C. The duration of this treatment will necessarily vary due to other operating conditions, e. g., the size of the casting and furnace temperature. However, it is only essential that the casting be heated at least sufficiently long to be uniformly heated throughout to the annealing temperature to eliminate any internal stresses caused by the comparatively rapid chilling of the alloy as it solidified. As an illustration, it can be stated that annealing for two hours at 1050° C. gives excellent results. It should, however, be appreciated that longer or shorter periods of time may also be used satisfactorily.

After the annealing operation, the casting must be slowly cooled, e. g., furnace cooled, to room temperature (20–25° C.) or some other temperature (such as 100–200° C.) where thermal stresses induced by more rapid cooling rates would be negligible. However, until such relatively low temperature is reached, cooling of the casting must be effected slowly so as to avoid setting up any new stresses. A specific cooling rate of 30° C./hour has been found desirable. However, the optimum rate in any given case will depend upon the various other operating conditions and can be readily determined.

As an alternative to the use of a separate furnace for the purpose of carrying out the annealing and subsequent slow-cooling operations, the alloy may be cast in a sand or ceramic mold which maintains the solidified casting at the desired annealing temperature for the necessary length of time and then permits the same to cool slowly at, for example, 30° C./hour. Any other means which will permit this annealing and slow cooling operation to be carried out on the casting either in situ, i. e., in the mold, or apart from the mold may be used.

The invention is illustrated, but not limited, by the following specific example of the preparation of an alloy of preferred composition and its fabrication into sheets. Wherever possible alternative modes of operation have been discussed, but it will be recognized that various additional modifications can be made without deviating from the invention.

#### EXAMPLE

For purposes of illustration, a melt consisting essentially of nominally 16% Al, 3% Mo and the remainder Fe (specifically 16% Al, 3.3% Mo and 80.7% Fe) by weight was prepared. The metals used to form the melt contained minute amounts of impurities normally associated therewith, such as Si, C, S, P and Cu, which can be disregarded for the purposes of this invention.

The melting operation was carried out in a high purity magnesium oxide crucible and was accomplished by induction heating in a pressure-vacuum furnace of 30 pound capacity.

The furnace chamber was pumped down to a pressure of about 200 microns and the iron and molybdenum then melted. Pressure in the furnace increased as the components melted due to the release of the dissolved gases. The molten iron and molybdenum solution was given a decarburizing treatment with wet hydrogen, followed by treatment with dry hydrogen (dewpoint –90° F. or better) to effect deoxidation.

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The hydrogen was then purged from the chamber with pure dry helium, followed by evacuation to remove hydrogen dissolved in the melt during the decarburization and deoxidation treatments. Substantially all hydrogen dissolved in the melt is removed during this evacuation. The system was refilled with helium and the aluminum then added to the melt.

As soon as the aluminum had been added, the chamber was pumped down to a pressure of approximately five millimeters, the temperature adjusted optically and the melt poured through a graphite pouring cup (coated with magnesium oxide) into a steel slab mold coated with magnesium oxide mold wash. The mold cavity measured approximately 10" x 5" x 1".

While the foregoing shows casting the slab under 5 mms. of helium, any reduced pressure and inert gas, e. g., argon at 5 mms. may be satisfactorily utilized. However, it is essential, for at least two reasons, that the melt be cast under a reduced pressure of an inert gas. First, this removes hydrogen, which is undesirable, from the melt. Secondly coupled with the design of the mold, it makes possible a solidification rate which produces the necessary fine-equiaxed cast grain structure. This is established by the fact that when the melt of this example is cast in a steel slab mold under 5 mms. of helium, a fine-equiaxed cast grain structure is obtained, whereas casting under one atmosphere of helium in a steel slab mold and a ceramic mold, under otherwise comparable conditions, gives a large columnar cast grain structure and a large-equiaxed cast grain structure, respectively.

A typical analysis of the casting shows that it includes negligible amounts of carbon, hydrogen, oxygen and nitrogen. Significant amounts of these materials should be avoided, since they effect a weakening of the alloy.

It will be recognized that, in this stage of the process, the melt may be cast into any suitably shaped mold, i. e., the melt may be cast into a mold which gives the desired final shape or it may be poured into an ingot or slab-type mold for the purpose of forming an ingot or slab for subsequent hot and/or cold rolling, swaging, extruding or drawing into strips, sheets, rods, wire and other shapes.

The casting produced in the manner described above was permitted to cool to the point (about 1450° C.) where it was sufficiently solid, although still red hot, to be removed from the mold. Subsequent analysis showed that the casting possessed a single phase and had a highly desirable fine-equiaxed cast grain structure due, as noted above, to regulating the rate of solidification by the use of a mild steel mold and the reduced inert gas pressure. Any other equivalent technique for similarly regulating solidification to give a fine-equiaxed cast grain structure could be used, such as the addition of misch metal or other rare earth grain refiners or ultrasonic shaking.

The red hot casting was removed from the mold and placed in a furnace at a temperature of 1050° C. and allowed to soak at this temperature for a period of two hours. This annealing step eliminated substantially all of the internal stresses set up in the casting as it solidified.

Thereafter, the furnace was shut down and the annealed casting permitted to slowly cool therewith at the rate of about 30° C. per hour. When the casting had reached a temperature of about 200° C., at which point rapid cooling would not impart any stresses thereto, it was removed from the furnace and machined without difficulty to remove surface imperfections. Machining of a comparable casting which was not subjected to the annealing and slow cooling operations caused very severe cracking.

The machined casting of the invention was thereafter hot rolled. This was accomplished using a laboratory two-high mill in substantially conventional manner and

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starting with the casting heated to a temperature of 1050° C. The following rolling method was used:

A first pass of 0.050", and a second pass of 0.025" were followed by a series of passes of 0.010" until the mill began to labor (temperature of casting at this point was about 800° C. (1472° F.)). The casting was then reheated for five to ten minutes until the original temperature of 1050° C. (1922° F.) was again reached. The second series of passes was begun with a 0.025" pass, followed by a series of 0.010" passes until the temperature of the slab again decreased to about 800 C. (1472° F.). This was continued to a thickness of 0.250". At this point, the temperature was dropped to 950° C. (1742° F.) and two passes of 0.010" were given between reheatings until a thickness of about 0.125" was reached.

After the hot-rolling operation, the resulting sheet was sandblasted, lightly surface ground, or cleaned by equivalent cleaning technique in order to remove any slivers or other loosely adhering material. The sheet was then cold-rolled at 575° C. although any temperature below the recrystallization temperature may be used. Rolling at 575° C. (1067° F.) was initially carried out on a two-high mill allowing the alloy to reheat for approximately five minutes (or long enough to heat the material thoroughly). At a thickness of approximately 0.040" the rolling was transferred to a four-high laboratory mill and finished thereon to a thickness of .020".

The resulting product demonstrates an unexpectedly high tensile strength at room temperature in the vicinity of 82,000 p. s. i. Heating this product to about 300° C. (572° F.) seemed to increase impact strength sharply. Shearing and forming characteristics also showed a marked improvement when the sheet was heated to between 200° C. (392° F.) and 350° C. (662° F.). For example, the sheet, when recrystallized, could be given a 90° bend or more at 250° C. (482° F.), whereas the cold-worked sheet of the same thickness required about 300° C. When further reduced to thicknesses below 0.007", little or no heating was required for sharp bends.

While the foregoing example is specifically concerned with a preferred alloy composition consisting essentially of 16% Al, 3.3% Mo and 80.7% Fe, satisfactory products, which are characterized by being single phase alloys, may be obtained using from 10 to 18% Al, usually at least 12% Al, and from 1 to 5% Mo with the rest Fe. Additionally, at least a part of the Mo, and in some cases possibly all of it, can be replaced by another strengthening additive, such as Ti, Ta, V, Nb, Cr, Ni, B and W. For instance, a small amount of V or Ti, for example, .3%, may be used in combination with the Mo in which case the total amount of strengthening additive should not exceed 5%. The most unique and preferred products, however, are single phase alloys consisting essentially of 15-17% Al, 2-4% Mo and the rest Fe. Of these, the product described above containing nominally 16% Al, 3% Mo and the remainder Fe has been found exceptionally desirable, either where the alloy is cast in final form or where the casting is subsequently worked into a desired shape as in the case of the sheets described above.

The following characteristics, in addition to those hitherto recited, are demonstrated by the preferred products of the invention and aptly emphasize the unique nature thereof:

(1) Excellent oxidation resistance to temperatures as high as 1260° C. (2300° F.).

(2) Outstanding resistance to corrosion by oxidizing solutions, such as concentrated nitric acid, and other normally corrosive media, including citric acid, fruit juices, glacial acetic acid, and concentrated ammonium hydroxide, at room temperature, as well as sulfur bearing vapors at high temperature. Polished samples exposed to normal atmospheric conditions and handled daily for approximately a year still retain their original polished appearance. Hence, the alloys offer potential utility for

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kitchenware and like applications where stainless properties are desired.

(3) High electrical resistivity of 160 micro-ohm-cm. This characteristic, coupled with the alloys' oxidation resistance, makes the same suitable for resistance heating elements. Qualitative tests performed on heating elements made of these alloys show a comparatively long life for elements of this type.

(4) The alloys can be machined satisfactorily at room temperature (20-25° C.) and can also be arc or spot welded without difficulty.

(5) The alloys have a density of about 6.58 grams/cm.<sup>3</sup> which is about 20% less than stainless steel. The products thus possess a high strength/weight ratio which is obviously advantageous, especially in the aircraft industry.

(6) Excellent magnetic properties. For example, maximum permeability as high as 130,000, initial permeability of 6500 and a coercive force of 0.017 oersted have been obtained on suitably heat treated laminations where in the heat treatment involves heating at 1050° C. for one hour, furnace cooling to 600° C. (i. e. a temperature above the order-disorder range), holding at 600° C. for ten minutes and then quenching with water. These magnetic properties and the hardness and inherent wear resistance of the alloys make same suitable for use as recorder heads and the like. These products may also be used in many other forms and diverse manner, e. g., as magnetic shields, valves, pipes and chemical vessels subjected to corrosive media, infrared emitters, high temperature engine and compressor parts, furnace parts and the like.

The ferritic alloys of this invention possess properties which are superior to most ferritic stainless steels and reasonably close to the values obtained for austenitic stainless steels. This is shown by the following data wherein the Thermenol product referred to is an iron-base alloy consisting essentially of nominally 16% aluminum and 3% molybdenum with the remainder substantially all iron. This product was prepared in the manner hitherto described followed by an additional heat treatment as set forth below:

TABLE 2

Comparative stress-rupture data on several high temperature alloys

| Alloy (Balance Fe)                                 | Test Temperature, °F. | Stress (p. s. i.) | Stress-Rupture Life (Sheet Values), hours | Remarks                                  |
|----------------------------------------------------|-----------------------|-------------------|-------------------------------------------|------------------------------------------|
| Type 403: Stainless 12% Cr.                        | 1,100                 | 25,000            | 20                                        | Jet engine compressor Blades (ferritic). |
| Type 316: Stainless 17% Cr, 12% Ni, 2.5% Mo.       | 1,200                 | 28,000            | 100                                       | Jet engine components (austenitic). Do.  |
| Type 321: Stainless 18% Cr, 8% Ni (Ti stabilized). | 1,200                 | 22,000            | 100                                       |                                          |
| Thermenol.....                                     | 1,100                 | 25,000            | 670                                       |                                          |
|                                                    | 1,200                 | 25,000            | 47.6                                      |                                          |

According to the invention, it has also been found that the characteristics of the cold-rolled alloy can be remarkably enhanced by subjecting same to an additional heat treatment followed by air-cooling. This is the heat treatment referred to above in connection with the Thermenol product set forth in Table 2. In that particular instance, the Thermenol sheet was heated in a furnace to about 1050° C. for two hours and thereafter cooled in still air. This additional treatment is optional in the case where the alloy is cast in final form and no other substantial shaping operation is necessary. However, it is highly desirable as an after treatment for rolled sheet materials, especially cold-rolled sheet, and other articles obtained by subjecting the cast product to further shaping operations.

Optimum temperatures and times for this additional heat-treating step will necessarily vary dependent upon other operating factors, particularly the size of the rolled sheet or other article. Heating the article to temperatures as high as 1150–1200° C. at least until uniformly heated throughout at such temperatures, and possibly for an hour or two thereafter, is generally suitable. Higher temperatures should be avoided because of the tendency of excessive grain growth at such temperatures. A preferred temperature range for this heating step is 1050–1100° C. with 1000° C. about the lowest temperature which can be used.

After this additional heating operation, the hot and/or cold-rolled sheet, or other product is preferably still-air cooled to room temperature (20–25° C.). However, in lieu of still air cooling, other means providing a substantially equivalent rate of cooling may be utilized.

The importance of this aftertreatment, and especially the criticality of the temperature employed in the heating step is stressed by the fact that it effects an increase in stress-to-rupture life of about five times for cold-rolled sheets over the life of similar sheet material after heating at 800° C. (1472° F.) for two hours. It would appear at first glance that the solution of a second phase is responsible for this large increase in strength, but tests show no evidence of a second phase. Possibly there is a substructure effect, but in any event there is no definite explanation for the unexpectedly great improvement in stress-to-rupture strength and other properties brought about by the aftertreatment.

The effect of alloy composition and the above-discussed aftertreatment on 0.020" thick cold-rolled sheet is further illustrated by the following high temperature stress-rupture data:

TABLE 3

| Sample No. | Alloy Composition                       | Form of Alloy Material          | After-Treatment                               | Stress-Rupture Times in Hrs. using 25,000 p. s. i. load and tested as 1200°F. |
|------------|-----------------------------------------|---------------------------------|-----------------------------------------------|-------------------------------------------------------------------------------|
| 1          | 15.6% Al, Remainder Fe.                 | 0.020" thick cold rolled sheet. | None                                          | 0.5                                                                           |
| 2          | 15% Al, 3.3% Mo, Remainder Fe.          | do                              | 1,050° C. for 2 hrs. followed by an air cool. | 47.6                                                                          |
| 3          | 17.2% Al, 3.3% Mo, Remainder Fe.        | do                              | do                                            | 69                                                                            |
| 4          | 17.2% Al, 3.3% Mo, Remainder Fe.        | do                              | 900° C. for 2 hrs. followed by an air cool.   | 24                                                                            |
| 5          | 17.2% Al, 3.3% Mo, Remainder Fe.        | do                              | None                                          | about 5                                                                       |
| 6          | 13.8% Al, 4% Mo, Remainder Fe.          | do                              | 1,050° C. for 2 hrs. followed by an air cool. | 54.4                                                                          |
| 7          | 16% Al, 3.3% Mo, 0.05% C, Remainder Fe. | do                              | do                                            | 19.3                                                                          |
| 8          | 14% Al, 2% Mo, 0.3% V.                  | do                              | do                                            | 17.2                                                                          |
| 9          | 15% Al, 5% Mo, Remainder Fe.            | do                              | do                                            | 61                                                                            |
| 10         | 16% Al, 1.68% Mo, 0.3% Ti, Bal. Fe.     | do                              | do                                            | 73.2                                                                          |
| 11         | 16% Al, 1% Ti, Bal. Fe.                 | do                              | do                                            | 9.9                                                                           |

From Table 3 the following conclusions can be drawn:

1. The basic binary alloy containing about 16% Al, the remainder iron, in the cold-rolled sheet form, with no aftertreatment has a very low high temperature strength.

2. The addition of elements such as Mo, V and Ti definitely improves the high temperature strength of the binary mixture; compare especially samples 1 and 5.

3. Coupled with the aforesaid additives, maximum high temperature strengths are attained by an after-treatment of about 1050° C. for about 2 hours followed by a still-air cool. The effectiveness of this heat treatment can be seen by comparing samples 3 and 4 in the table.

4. Higher percentages of Mo increase the high temperature strength. However, no more than 5% Mo should be used since greater amounts cause difficulties in cold rolling.

5. The addition of Mo, V and Ti and the like is for the purpose of increasing the high temperature strength of the basic Al-Fe alloys by strengthening the existing ferritic solid solution. As previously noted, these element additions in small amounts appear to have substantially no effect upon the grain size or oxidation resistance of the resultant alloys.

Various other modifications may be made in the process specifically exemplified above. For example, other conventional hot and/or cold rolling techniques may be utilized although it will be recognized that heavier than normal passes may be employed in view of the fine-equiaxed cast grain structure of the present alloys. Thus, another method which may be satisfactorily utilized for hot rolling the casting is the following:

The casting is given a 10% reduction between reheatings (reheating time five to ten minutes) until a thickness of 0.250" is obtained. At this thickness the temperature is reduced to 950° C. (1742° F.) and rolled to a thickness of about 0.125". The lower finishing temperature of 950° C. produces a finer grained material than that obtained by rolling at 1050° C. all the way.

The use of larger capacity mills also requires some deviation in the hot rolling procedure. For instance, it is possible with a Sendzimir hot planetary mill to reduce castings of, e. g., 1" thickness, to about 0.1" in a single pass.

While the process illustrated above shows both hot and cold rolling to fabricate sheets from the alloys of the present invention, it should be appreciated that desirable products can also be obtained by only hot rolling or cold rolling. The distinction between these two steps is that, in cold rolling, the alloy is worked at a temperature below the recrystallization temperature, whereas hot rolling denotes working the alloy at a temperature above the recrystallization temperature. Stated in another way, the metal is recrystallized during hot rolling, while in the cold rolling process, the crystal structure remains unrecrystallized and the crystals are elongated. Cold rolling at, for example, 575° C. gives the material a regular cold worked appearance and elongates the grains into long fibered structure. The greater the percentage of cold work, the higher the mechanical properties of the alloy (tensile strength, yield strength, hardness, impact strength, etc.). Cold rolling may be performed to reduce the material to a relatively thin gage, such, for example, as 0.002" or even thinner.

Moreover, the cold rolled sheet alloys may be recrystallized at about 750° C. for one hour to give a recrystallized fine grain material which apparently can only be obtained in this way.

Cold rolled strip of a reasonable thickness may also be formed and sheared if warmed. The exact temperature of warming is dependent upon the forming configuration and material thickness of the alloy.

After the alloy has been rolled into strip form of a suitable thickness, it has a naturally formed coating of aluminum and iron oxides upon the surface. If it is desired to remove this coating for any purpose (spot welding, soldering, brazing, etc.), this may be done by the following treatment:

Immerse the strip in hot NaOH (e. g., 20% solution by weight) for a few minutes. Wash in water at room temperature. Immerse strip at room temperature in HNO<sub>3</sub> (e. g., 30% solution by volume) for a few min-

utes. Wash in water and thereafter dry to obtain the strip in oxide-free form.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is, therefore, to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

This application is a continuation-in-part of our co-pending application Serial No. 448,398, filed August 6, 1954.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. A method of making a ferritic alloy having a substantially single phase, fine-equiaxed cast grain structure and consisting essentially of 12 to 18% aluminum, 2 to 4% of a strengthening additive selected from the group consisting of molybdenum and titanium, and iron, said method comprising the steps of melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure.

2. The method of claim 1 wherein the strengthening additive is molybdenum.

3. The method of claim 1 wherein the melt consists essentially of 15 to 17% Al, 2 to 4% Mo and the remainder Fe.

4. The method of claim 1 wherein the rate of solidification of the ingot is controlled by casting under a reduced pressure of an inert gas.

5. The method of claim 4 wherein the inert gas is helium.

6. The method of claim 1 wherein the solidified ingot is annealed by heating at a temperature of about 1050° C. at least until it is uniformly heated throughout to this temperature.

7. A method of making a ferritic alloy having a substantially single phase, fine-equiaxed cast grain structure and consisting essentially of 12 to 18% aluminum, 2 to 4% of a strengthening additive selected from the group consisting of molybdenum and titanium, and iron, said method comprising the steps of melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure, annealing the solidified ingot and eliminating stresses therein, and continuing with the slow cooling of said ingot while avoiding the occurrence of further stresses therein, said ingot being maintained in the substantially single phase form throughout said process.

8. The method of claim 7 wherein the annealed ingot is slow cooled at the rate of 30° C. per hour.

9. A method of making a high temperature magnetic ferritic article having a fine-equiaxed cast grain structure and consisting essentially of 12 to 18% Al, 2 to 4% Mo, and the remainder Fe, which comprises melting together the constituents, casting the melt into an ingot under a reduced pressure of an inert gas, slow cooling the ingot through the solidification range and producing a substantially single phase composition having a substantial amount of a fine-equiaxed cast grain structure, annealing said ingot to eliminate stresses therein, slowly cooling the ingot while avoiding the occurrence of further stresses therein, maintaining said substantially single phase fine-equiaxed cast grain structure throughout said annealing and cooling steps, and thereafter shaping said ingot.

10. The method of claim 9 wherein said shaping operation comprises rolling the ingot into sheet form.

11. The method of claim 9 wherein the ingot, after being shaped, is aftertreated by heating at a temperature of about 1000° C. to 1200° C. at least until it is uniformly heated throughout to this temperature.

12. The method of claim 10 wherein the ingot, after

being rolled, is aftertreated by heating to a temperature of about 1050° C. to 1100° C. at least until it is uniformly heated throughout to this temperature and thereafter still-air cooling.

13. The method of claim 9 wherein said shaping operation comprises working said ingot by both hot and cold rolling.

14. A method of making a high temperature magnetic ferritic sheet having a fine-equiaxed cast grain structure and consisting essentially of 15 to 17% Al, 2 to 4% Mo, and Fe, which comprises melting together the constituents, casting said melt into an ingot under a reduced pressure of helium, slow cooling the ingot through the solidification range and producing a substantially single phase composition having a substantial amount of a fine-equiaxed cast grain structure, furnace annealing said ingot to eliminate any stresses therein, furnace cooling said ingot to a temperature at which more rapid cooling imparts substantially no internal stresses and maintaining said ingot in its substantially single phase form throughout said annealing and cooling steps, thereafter hot and cold rolling said ingot to sheet form of desired thickness, heating the resulting sheet uniformly throughout to a temperature of about 1050 to 1100° C. and then still-air cooling said sheet.

15. The method of claim 14 wherein said hot and cold rolling operations are performed at temperatures of about 1050° C. and at about 575° C., respectively.

16. The method of claim 14 wherein the cold rolled sheet is recrystallized by heat treatment above its recrystallization temperature.

17. A method of making a high temperature magnetic ferritic sheet having a fine-equiaxed cast grain structure and consisting essentially of 15 to 17% Al, 2 to 4% Mo, and Fe, which comprises melting together the constituents, casting said melt into an ingot under 5 mms. pressure of helium, slow cooling the ingot through the solidification range and producing a substantially single phase composition having a substantial amount of a fine-equiaxed cast grain structure, furnace annealing said ingot at a temperature of about 1050° C. until it is uniformly heated throughout to this temperature, furnace cooling said ingot at the rate of 30° C. per hour to a temperature at which rapid cooling imparts substantially no internal stresses, maintaining said ingot in its substantially single phase form throughout said annealing and cooling steps, thereafter hot and cold rolling said ingot to sheet form of desired thickness, heating the resulting cold rolled sheet at a temperature of about 1050° C. until uniformly heated throughout to this temperature and then still-air cooling.

18. The method of claim 17 wherein the melt is decarburized and deoxidized before casting.

19. A high temperature and magnetic single phase ferritic alloy consisting essentially of 15 to 17% Al, 2 to 4% Mo, and the remainder Fe, said alloy being substantially free of elemental carbon, oxygen, nitrogen and hydrogen and being produced by melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure.

20. A high temperature and magnetic single phase ferritic alloy consisting essentially of nominally 16% Al, 3.3% Mo, and the remainder Fe said alloy being produced by melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure.

21. The alloy of claim 19 in the form of a cold rolled sheet.

22. The alloy of claim 19 in the form of a casting.

23. A substantially single phase, stress-free, high temperature and magnetic ferritic alloy having a fine-equiaxed cast grain structure and consisting essentially of 12

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to 18% aluminum, 2 to 4% of a strengthening additive selected from the group consisting of molybdenum and titanium, and iron, said alloy being produced by melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure.

24. An alloy as defined in claim 23 including a minor amount of a rare earth metal addition.

25. An alloy as defined in claim 24 wherein said addition is misch metal.

26. The method of claim 9 wherein said shaping operation comprises working said ingot into sheet form by rolling and the resulting sheet is aftertreated by heating at 1050° C., for an hour, furnace cooled to 600° C., and then quenched.

27. The method of claim 9 wherein said shaping operation comprises extrusion.

28. A substantially single phase, stress-free, high temperature and magnetic ferritic alloy having a fine-equiaxed cast grain structure and consisting essentially of 12 to 18% aluminum, 2 to 4% of a strengthening additive se-

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lected from the group consisting of molybdenum and titanium, and iron, said alloy being produced by melting together the constituents, casting the melt into an ingot, slow cooling the ingot through the solidification range and producing a composition having a substantial amount of a fine-equiaxed cast grain structure, annealing the solidified ingot and eliminating stresses therein, and continuing with the slow cooling of said ingot while avoiding the occurrence of further stresses therein, said ingot being maintained in the substantially single phase form throughout said process.

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