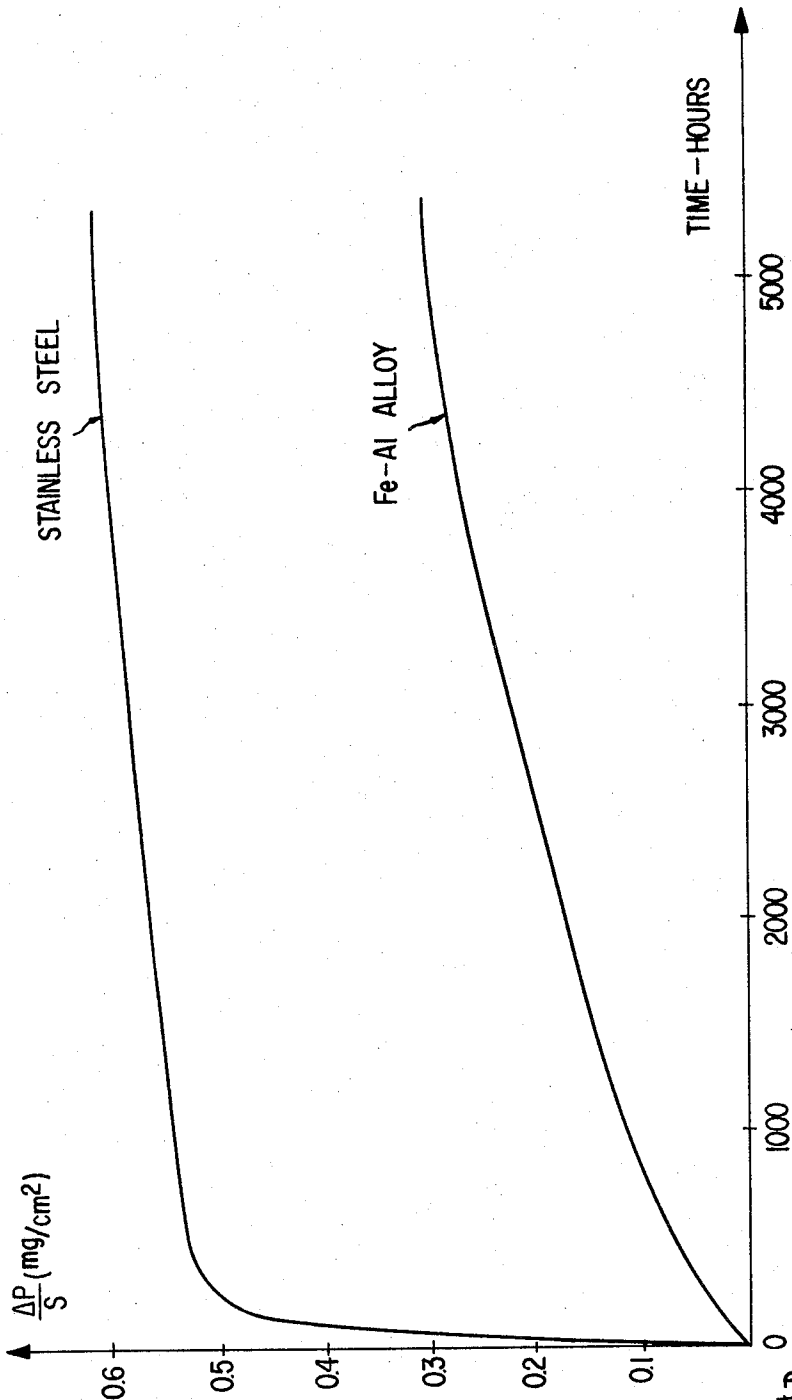


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IRON-ALUMINUM ALLOYS CONTAINING LESS THAN 84% BY WEIGHT IRON AND
AN ADDITIVE AND PROCESS FOR PREPARING THE SAME
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IRON-ALUMINUM ALLOYS CONTAINING LESS THAN 84% BY WEIGHT IRON AND AN ADDITIVE AND PROCESS FOR PREPARING THE SAME

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4 Claims. (Cl. 75—124)

ABSTRACT OF THE DISCLOSURE

Iron-aluminum alloys of greater than 16% by weight aluminum and less than 84% by weight iron and containing from 0.4% to 4% by weight, preferably 1% to 4% by weight, of an additive selected from the group consisting of zirconium, niobium, titanium, yttrium, the rare earths and boron and a process for preparing said alloys. Advantageously, the amount of iron ranges from 69% to 82% by weight, and the amount of aluminum from 18% to 31% by weight.

The present application is a continuation-in-part of application Ser. No. 261,152, filed Feb. 26, 1963, now U.S. Patent 3,303,561. The present application accordingly incorporates by reference the disclosure in said U.S. Patent 3,303,561 and the same is to be read as being a part of the present disclosure.

The present invention relates to a process for the preparation of an iron-aluminum alloy and, by way of new industrial products, the alloys obtained as a result of the application of said process.

Accordingly, it is an object of the present invention to provide a process for the manufacture of an alloy containing principally iron and aluminum of which the proportion of aluminum may be increased considerably without the attendant difficulties encountered heretofore.

Another object of the present invention resides in the provision of a process for producing an iron-aluminum alloy in which the aluminum content may be increased to a range above that normally feasible heretofore, without producing a product of which the brittleness is so great as to preclude any subsequent machining operations.

Another object of the present invention resides in the provision of a novel iron-aluminum alloy containing, by weight, approximately 18 to 31% of aluminum of which the brittleness is relatively low and which permits of subsequent hot or cold working operations.

A further object of the present invention resides in the provision of Fe-Al alloys in which the brittleness is controlled to a degree not realizable heretofore.

A further object of the present invention resides in the provision of a process for the manufacture of iron-aluminum alloys in which the thermal stresses are reduced and incipient boundary separations are controlled to fall within acceptable values.

A still further object of the present invention resides in a novel alloy principally containing iron and aluminum and having a relatively high proportion of aluminum which has magnetic properties and may be produced in the form of thin sheeting or foil.

Another object of the present invention resides in the provision of a process for the manufacture of an iron-aluminum alloy which permits the obtaining of very

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small thicknesses, accurate dimensions, and cold working treatments as well as subsequent heat treatments.

A further object of the present invention resides in the provision of a process for producing a low density iron-base magnetic alloy and in the resulting product which not only exhibits such low density properties, but also an oxidation resistance that is considerably higher than that of other iron-aluminum alloys as well as stainless steel.

Still another object of the present invention resides in the provision of a process for producing an iron-aluminum alloy having neutron absorption properties that are distinctly lower than those of stainless steel and having a yield strength that is considerably higher than that of stainless steel.

Still a further object of the present invention resides in the provision of a process for producing an iron-aluminum alloy and the alloy resulting from such process which may be used in nuclear reactors and has such properties and characteristics as to obviate the need for enriched fuels.

The present invention will be more clearly understood from a perusal of the following description of a number of examples of practical application of the process in accordance with the present invention for the preparation of an iron-aluminum alloy, the said examples being given only for illustrative purposes and without implying any limitation on the present invention. The single figure of the accompanying drawing shows a diagram illustrating the corrosion of an alloy in accordance with U.S. Patent 3,303,561, the patent application, in a carbon dioxide gas atmosphere as compared to that of a stainless steel under similar conditions.

Examples I through VI illustrate the basic process of U.S. Patent 3,303,561 and are included herein for a complete disclosure so as to provide a full understanding of the present invention.

EXAMPLE I

The alloy to be produced has the following composition:

Electrolytic iron	-----kgs--	3
Aluminum of 99.99% purity	-----kg--	1
Zirconium	-----grams--	4

(a) *Melting and casting.*—The 3 kilograms of electrolytic iron are melted and brought to a temperature of 1,600° C. in a vacuum of the order of 10⁻⁴ millimeters Hg; aluminum of 99.99% purity is then added thereto, followed by zirconium; the temperature is reduced to 1,450° C. and the molten mixture is poured off in vacuo, again of about 10⁻⁴ mm. Hg, into an ingot-mold which has been heated to 620° C.

Finally, the cooling rate is limited to approximately 50° C. per hour. It should be noted in passing that preheating is of course necessary in this example only on account of the fact that the casting mass employed in this example is small.

(b) *Roughing-down.*—The ingot which is obtained from Step (a) above after cooling is fitted with a metallic jacket, for example, of ordinary steel (XC 12 or XC 35 in particular). The covering of the ingot may be carried into effect by means of any one of the methods of conventional cladding, for example, by welding a sheet which has previously been wrapped around the ingot, by cold-state hydrostatic cladding, etc. The thickness of the jacket is obviously designed so that the subsequent mechanical treatments permit a thickness to remain which is such that there is no danger of tearing. This thickness was of the order of 2 mm. in the example described.

The composite work-piece formed by the ingot which is covered with its jacket is subjected to a series of rolling passes at 1050° C., each pass necessarily resulting in a

reduction in thickness which is sufficient to work-harden the metal right through.

The presence of the jacket makes it possible to facilitate the surface flow of the alloy and permits the presence of deformations which the ingot would not withstand if it were treated in the uncovered state.

In the example referred-to, each pass resulted in a reduction in thickness of 2 mm., while between two successive passes, there was carried out a reheating for a period of two minutes, thereby bringing the temperature back to 1050° C. The thickness of the composite work-piece can thus be reduced without difficulty to approximately 2 mm. It is apparent that the reheating treatment is only necessary on account of the fact that the temperature of the work-piece falls substantially as a result of the small dimensions of the latter.

The composite work-piece can then be freed of its steel jacket (the thickness of which has obviously been substantially reduced to the same extent as those of the work-piece) by different methods. The jacket, which in the example described only remains in the form of a film of the order of a few tenths of a millimeter, can be, for example:

Detached by machine-cutting of the jacket along one of the sides of the casing,

Destroyed by chemically dissolving the jacket in a mixture of 50% nitric acid and 50% water (the iron-aluminum alloy having good resistance to attack by dilute nitric acid),

Destroyed by selective oxidation of the jacket by air heating or heating in an oxidizing atmosphere.

(c) *Cold working*.—The alloy which is thus obtained may be subjected to subsequent mechanical operations which result in limited deformations, for example, deformation by rolling at room temperature with annealing treatments between successive rolling passes.

EXAMPLE II

(a) *Melting and casting*.—A cast is prepared under conditions which are similar to those of Example I starting with 2.9 kilograms of electrolytic iron, 1.1 kilograms of aluminum and 4 grams of zirconium. The temperature is then raised to a few tenths of degrees above the solidification temperature (or liquidus temperature) of the alloy and the latter is poured off in vacuo into a preheated ingot-mold. The cooling process is then carried out as in Example I. The alloy which is thus cast has the following composition by weight:

	Percent
Iron	72.2
Aluminum	27.7
Zirconium	0.1

In addition thereto, analysis shows traces of carbon, nitrogen, phosphorus and sulphur in the following proportions:

	Percent
Carbon	0.01
Nitrogen	0.01
Phosphorus	0.002
Sulphur	0.002

(b) *Roughing-down*.—The ingot thus produced is capable of undergoing lathe turning work when use is made of tools having great hardness such as tungsten carbide tools. The quality of machining is improved by maintaining the alloy at 400° C. during the machining operation.

The said machining operation may not be necessary in the case of certain surface conditions and when the roughing-down operation consists in a rolling process which can be performed after cladding according to a procedure which is similar to that described in the previous example. However, such a machining operation is necessary for the purpose of shaping the ingot when the treatment involves extrusion of the jacketed ingot.

When the extrusion process is intended to result in a full rod or slug, the lathe turning operation is performed with a view to obtaining a cylinder having a rounded front end. The work-piece which is thus machined is covered by means of any conventional process with a steel jacket having a shape which is adapted to that of the said work-piece and a thickness of a few millimeters. It may be useful to replace mild steel by other metals or alloys such as iron-aluminum alloys containing a low percentage of aluminum, which have the advantage of better oxidation resistance and, in certain cases, nickel or cupronickel.

The composite part which is thus obtained is then press-extruded at 950° C. At this temperature, it is possible to reach an extrusion ratio of the order of 1:30, or in other words, it is possible to prepare rods of 11 mm. diameter from machined ingots of 60 mm. diameter.

A similar process makes it possible to obtain tubes having a thickness which is less than one millimeter. In this case, the lathe turning operation is performed with a view to producing a hollow cylinder which is then clad both internally and externally.

After extrusion, the separation of the alloy and its steel jacket can be carried out in accordance with any one of the processes which have already been referred to in Example I, for example, by chemical dissolution in a solution composed of 50% water and 50% nitric acid which rapidly dissolves the jacket, by oxidation of the jacket, by air heating or in an oxidizing atmosphere. In the latter case, the jacket disappears whereas the alloy is not attacked by virtue of its high resistance to oxidation.

(c) *Cold working*.—The extruded product obtained may, in certain cases, be employed as it stands, inasmuch as it has a good surface to finish. However, it may, if necessary, be subjected to a further cold working treatment and may, for example, be threaded on a thread-cutting lathe. In fact, the grain size after extrusion is reduced to 20 or 30 microns and accordingly permits machining thereof.

The part which has been either machined or extruded may be subjected to a heat treatment for a period of one hour at 800° C.; after this heat treatment, the extruded product has the following characteristics:

Temperature, ° C.	Ultimate Tensile Strength, kgs./mm. ²	Yield Strength, kgs./mm. ²	Elongation at Fracture, Percent
20	154	40	3
340	91	37	15
500	36	25	50

¹ Brittle fracture.

EXAMPLE III

The same operations as in Example II (melting, casting, machining, jacketing, extrusion and elimination of the jacket) have also been applied to an alloy containing 25% aluminum by weight, the composition of which is as follows:

	Percent
Iron	74.9
Aluminum	25.0
Zirconium	0.1

and which shows traces of impurities in the following proportions:

	Percent
Carbon	0.01
Nitrogen	0.01
Phosphorus	0.002
Sulphur	0.002

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After heat treatment at 800° C., the product obtained has the characteristics which are given in the table below:

Temperature, ° C.	Ultimate Tensile Strength, kgs./mm. ²	Yield Strength, kgs./mm. ²	Elongation at Fracture, Percent
20.....	172	26	8
372.....	52	32	27
524.....	32	28	50

¹ Brittle fracture.

The product which is obtained as a result of extrusion may be subjected to further processing so as to permit a final cold rolling in light passes. This processing will consist (if necessary after burnishing of the extruded product), for example, of a further jacketing operation, followed by a rolling operation between 500° and 600° C. for the purpose of orienting the crystals. The product thus obtained, still in the jacketed state, can be cold rolled.

The single figure of the accompanying drawing represents the oxidation (expressed as increase in weight per unit area), against time (expressed in hours) of two materials in a carbon dioxide atmosphere at 700° C. under a pressure of 60 kg./cm.². The curve I corresponds to the iron-aluminum alloy in accordance with Example III herein. Curve II corresponds to an 18-12 niobium-stabilized stainless steel which is known for its good resistance to corrosion by carbon dioxide gas at high temperature. It may be readily seen from this figure that, at the end of a period of exposure of 5,500 hours, the corrosion of the iron-aluminum alloy is less than one half that of stainless steel.

EXAMPLE IV

(a) *Melting and casting.*—An alloy containing 79.6% iron, 17.2% aluminum and 2.8% beryllium is prepared from the following constituents:

Iron	kgs.....	3
Aluminum	kg.....	0.650
Beryllium	kg.....	0.105
Zirconium	grams.....	15

The melting and casting operations are carried out as in the previous examples; in the "as cast" state, this alloy has the following properties:

Grain size, approximately 0.15 mm.
Brinell hardness, $\Delta=320$.

(b) *Roughing-down.*—The ingot is rolled at a temperature of 1050° C. in passes which each result in a reduction in thickness of 1 mm. to a final thickness of 2 mm.; in this state, the alloy has a Brinell hardness $\Delta=330$.

Thereafter, a heat treatment at 1,100° C. makes it possible to reduce the Brinell hardness number to 260.

EXAMPLE V

(a) *Melting and casting.*—The same operations of melting and casting are applied to an alloy containing 25% aluminum having the following composition by weight:

	Percent
Iron	74.9
Aluminum	25
Zirconium	0.1

and impurities in the form of traces:

	Percent
Carbon	0.01
Nitrogen	0.01
Phosphorus	0.002
Sulphur	0.002

(b) *Roughing-down-hot-state deformation.*—Rolling passes are effected at 1,050° C. and a reheating treatment is performed between each rolling pass for a period of two minutes.

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A reduction value of less than 90% can be obtained with final thicknesses of the order of one millimeter.

(c) *Cold-state deformation.*—After hot rolling, the rolling treatment can be repeated at room temperature.

It is accordingly possible as a result of cold rolling to obtain reduction values of 50%.

After rolling, the Vickers hardness number of the product is 500 HV; heat treatments by annealing at 950° C. make it possible to reduce this hardness number to 280 HV.

EXAMPLE VI

The same operations as in Example II (melting, casting, ...) have been applied to an alloy having the following composition:

	Percent
Iron	68.9
Aluminum	31
Zirconium	0.1

and impurities of the same order as in Examples II and III.

The product obtained has the following characteristics:

Temperature, ° C.	Ultimate tensile strength, kgs./mm. ²	Yield strength, kgs./mm. ²	Elongation at rupture, percent
20.....	154	44	1.5
200.....	157.5	40	3.5
400.....	271	38.5	13
524.....	222	16.5	46

¹ Brittle fracture.

² Ductile fracture.

The examples which are given above, although obviously not limitative, show that the process in accordance with U.S. Patent 3,303,561 makes it possible to obtain iron-aluminum alloys in which the proportion of aluminum substantially exceeds the values of 16 to 18% by weight which were hitherto accepted as the limit starting from which alloys no longer had mechanical properties which permitted their subsequent working. The iron-aluminum alloy which may now be produced by this process makes it possible to approach the limit of solubility of aluminum in iron (34% approximately) while retaining good mechanical properties. If such mechanical properties are not essential requirements, a small precipitation of aluminum is permissive at the expense of a very substantial reduction of mechanical characteristics, thereby making it possible to reach a proportion of approximately 40%.

The examples described below illustrate embodiments of the present invention, in which the alloy includes additives selected from the group consisting of yttrium and rare earths in proportions such that the total percentage of said additives contained in the solidified alloy is within the range of 0.4 to 4%.

When the alloy is for use in the nuclear field, the addition element is chosen among those which have the smallest neutron-capture cross-section; yttrium will usually be adopted. In other cases, and for purposes of economy, mixtures of rare earths can be employed of the type known as "Mischmetal."

The invention also provides improved iron-aluminum alloys obtained as a result of the application of the process referred to above, the said alloys containing addition elements selected from the group consisting of yttrium and rare earth metals in a proportion which ranges from 0.4 to 4% by weight, preferably at least 1% to 4% by weight, and having grain dimensions smaller than 20 μ after mechanical working.

In the preferred case of alloys which only contain iron, aluminum and such additives, the aluminum content is advantageously comprised between 18 and 31%, this last-mentioned value corresponding to the appearance of a precipitate of the phase Fe-Al₂.

The incorporation of the additive or additives is carried out during the melting process, preferably by introducing it or them in the bath of molten iron. During the next phase (up to solidification of the ingot), losses are liable to occur; in order that the final content should comprise between 0.4 and 4% by weight, it may therefore be found necessary to introduce into the molten mass an addition which would correspond to a higher percentage content than that which is sought.

The casting and solidification processes being conducted under conditions which are comparable with those described in Examples I-VI, a substantial reduction in the mean grain size of the ingot is noted, with the advantages thus obtained. In the as-cast state under the same conditions of production, the grain size is, for example, reduced by a factor of the order of 10 with an addition of 1% of lanthanum or yttrium. The result thereby achieved is a reduction in brittleness and an improvement in machinability of as-cast ingots; lathe turning can be accomplished under the usual conditions of temperature and in addition yields products of a quality which is at least equal to that of machining at 400° C. which is described in Example II.

This reduction in grain size is retained at the time of roughing-down, in particular when carried out by the hot extrusion process after cladding as described above. It is possible in particular to obtain grains having a size of the order of 10 microns after extrusion at 950° C. and, in a general manner, the grain size remains smaller than 20 microns.

An attempt can be made to explain the achievement of a reduction rather than an increase in grain size, although it will be understood that the explanation which now follows is given only by way of indication and cannot be considered as lying at the heart of the present invention. The addition of rare earths in proportions higher than 0.4% is accompanied in the as-cast state by the precipitation at the grain boundaries of a second phase which is totally different from the main phase which forms the matrix (Fe-Al phase up to 31% by weight of aluminum). At the time of extrusion, these precipitates are aligned in the direction of extrusion in fibers having a density which increases with the percentage of rare earths contained in the alloy. The increase in grain size is accordingly prevented by this precipitation.

In the case in which lanthanum is employed as an additive, the precipitates observed are Fe-Al-La compounds. This precipitate is brittle, but the mechanical properties of the alloy are nevertheless improved. It is possible that the beneficial effect of lanthanum in this particular case not only lies in its grain-refining action but also in the fact that the matrix itself becomes more ductile as a result of the purification which is due to the precipitate or to the lanthanum.

The effect of reduction in grain size is maintained in the case of annealing operations, even at high temperature. Thus, a level-temperature stage of one hour at 1150° does not result in any perceptible increase in grain size since the grains are stabilized by the precipitates.

This stabilization constitutes an appreciable advantage which is of special value for welding purposes. It is in fact possible to weld iron-aluminum alloys having a high aluminum content (for example 40% in terms of atomic ratio) by conventional methods such as by arc welding in an argon atmosphere. However, the welded zone has a casting structure which is much more brittle than that of the base alloy. In order to prevent the alloy from melting, diffusion welding can be performed in the solid state but, in order to break up the oxide layer and ensure a good weld, the parts to be assembled have to be heated to at least 1100° C. for a period of a few minutes; this treatment produces a substantial increase in grain size within the alloy according to the above examples, whereas the alloy which contains 0.4 to 4% of rare earths retains

its fine grain structure and good properties which result therefrom.

Finally, the alloy which is prepared in accordance with the present invention retains improved machinability after extrusion as well as enhanced mechanical properties.

EXAMPLE VII

This example refers to a lanthanum alloy which exhibits after roughing-down an elongation of 11% under tension at normal speed and at room temperature.

The alloy to be produced has a composition which is comparable with that which is given as Example I, but lanthanum is added. The melt is prepared from:

Electrolytic iron	-----kg. (75%)	3
Aluminum of 99.99% purity	-----kg. (24%)	0.960
Lanthanum	-----gr. (1%)	40

(a) *Melting and casting.*—The conditions of melting and casting are similar to those of the process described hereinabove, i.e., the iron is melted and brought up to 1600° C. in vacuo, aluminum is added, lanthanum is added at the same time as aluminum, the temperature is reduced to 1450° C. and the casting operation is performed in an ingot-mold which has been pre-heated to 620° C. Finally, the cooling rate is limited to approximately 50° C. per hour.

The conditions of casting in vacuo as adopted in the case which is contemplated resulted in a loss of lanthanum and analysis of the ingot revealed only 0.7% by weight of lanthanum in addition to the usual traces of carbon, nitrogen, phosphorus and sulphur.

(b) *Roughing-down.*—The roughing-down process can consist of a series of operations which are similar to those described in Example II.

The ingot can subsequently be machined on a lathe by using tools of high hardness. The machined workpiece is clad with a steel jacket a few millimeters in thickness. The composite workpiece obtained is press-extruded and the steel jacket is removed, for example by chemical dissolving in a solution composed of 50% water and 50% nitric acid.

As has been indicated above in a general manner, it is no longer necessary to maintain the alloy at 400° C. during the machining operation.

(c) *Cold working.*—The extruded product obtained has a better state of surface than that of the product according to Example II. Moreover, it can be cold-worked and this operation (which is already possible with the alloy in accordance with said Example II) is further facilitated by the smaller grain size which is in the vicinity of 10 microns after extrusion.

The machined workpiece can undergo a heat treatment for a period of one hour at 1150° C. without any appreciable increase in grain size.

After treatment for a period of one hour at 800° C. followed by slow cooling (30° C. per hour), the extruded product has the following characteristics:

Temperature, ° C.	Ultimate tensile strength, kg./mm. ²	Yield strength, kg./mm. ²	Elongation at rupture (Normal deformation rate), percent
20.....	85-95	35-40	11
200.....	100	32	16
400.....	73	35	30
600.....	35	34	75

The above example shows that the invention makes it possible to improve those iron-aluminum alloys in which the proportion of aluminum exceeds 18% by weight to an extent which greatly facilitates machining, reduces the wear of tools and lowers the price and duration of the machining operation.

EXAMPLE VIII

The iron and aluminum contents of the alloy produced are the same as in Example I, but the final percentage of lanthanum is 3.75% by weight. The successive steps are the same. The extruded product has the following characteristics at 20° C.:

Ultimate tensile strength -----kg./mm.²--- 66
Yield strength -----kg./mm.²--- 58
Elongation at fracture -----percent--- 1.0

The ductility of the alloy is much decreased by the increase in lanthanum content; on the contrary the elastic limit is increased.

EXAMPLE IX

The iron and aluminum contents are again the same as in Example I, but lanthanum is replaced with yttrium, the final proportion of which is 0.4%. The successive process steps are the same as above. The product exhibits the following characteristics:

Temperature ° C.	Ultimate tensile strength, kg./mm. ²	Yield strength, kg./mm. ²	Elongation at fracture, percent
20-----	84	29	10
200-----	91	29	15
400-----	72	32	35
600-----	41	38	60

EXAMPLE X

The composition and process steps are the same as in Example IX, except a regards the yttrium content, which is increased to 3.15%. The product has the following characteristics at 20° C.:

Ultimate tensile strength -----kg./mm.²--- 80
Yield strength -----kg./mm.²--- 55
Elongation at fracture -----percent--- 2

It is wholly apparent that the scope of the present invention extends not only to the process which has just been described and to all alternative forms thereof which are within the scope of equivalency thereof, but also, by way of new industrial products, to the alloys which are obtained as a result of the application of the process in accordance with the present invention.

Thus, while we have described several specific examples in accordance with the present invention, it is obvious that the same is not limited thereto, but is susceptible of numerous changes and modifications within the scope of a person skilled in the art, and we therefore do not wish to be limited to the details described herein, but intend

to cover all such changes and modifications as are encompassed by the scope of the appended claims.

We claim:

1. An alloy principally composed of iron and aluminum and of a small amount of at least one additive selected from the group consisting of zirconium, niobium, titanium, yttrium, the rare earths, boron and mixtures thereof in which the aluminum content is greater than about 16% by weight of the alloy and the iron content is less than about 84% by weight of the alloy while the additive content is less than one-tenth the content, by weight, of the aluminum content but more than at least 1% by weight of the ingot, said alloy being characterized by a relatively low brittleness permitting machining operations.

2. A process for the preparation of an iron-aluminum alloy comprising: melting an amount of iron which corresponds to a proportion less than 84% by weight of the alloy, adding to the molten iron the other constituents of the alloy, casting the melt at a temperature slightly above the solidification point of the alloy, cooling the alloy to solidify it in the form of an ingot, and subjecting the ingot to hot-state mechanical working and deformation to destroy the casting structure, including the incorporation with the melt of at least one additive selected from the group consisting of yttrium and rare earths in proportions such that the total percentage of said additives contained in the solidified alloy is within the range of at least 1% to 4% by weight.

3. A process according to claim 2, wherein the percentage content of iron ranges from 69% to 82% by weight and the percentage content of aluminum ranges from 18% to 31% by weight.

4. A binary iron-aluminum alloy in which the aluminum content is greater than 18% by weight containing a proportion ranging from at least 1% to 4% by weight of additives selected from the group consisting of yttrium and the rare earths and having grain sizes smaller than 20 μ after mechanical working and deformation in the hot state.

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