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2,987,394

IRON-ALUMINUM BASE ALLOYS

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No Drawing. Filed Mar. 25, 1959, Ser. No. 801,724
5 Claims. (Cl. 75-124)

This invention relates to iron-aluminum base alloys having improved ductility and superior high-temperature oxidation and water-corrosion resistance.

Development of new iron-aluminum base alloys has been stimulated by the need for metals suitable for use in neutronic power reactors. Heretofore, stainless steels and zirconium or zirconium alloys have been the preferred structural metals because of their good oxidation and water-corrosion resistance. Iron-aluminum base alloys have lower thermal neutron absorption cross-sections than the stainless steels and are less expensive than zirconium. Consequently, from the aspects of neutron economy and cost, alloys of the present class would possess certain advantages as reactor structural materials. Heretofore, their use in reactors has not been feasible because of poor formability or tendency to become embrittled after high temperature fabrication operations, such as hot-roll bonding or high temperature brazing.

Materials like Alfenol (16 w/o Al, rem. Fe) and Thermenol (16 w/o Al, 3.3 w/o Mo, rem. Fe) are characterized by low level ductility, which requires that all working of the metal be performed warm (about 1000° F.) or hotter. Even after full anneal, the alloy remains brittle. A more recently developed alloy, having much improved fabrication properties, is described in the co-pending application of Ida and Mueller, entitled "Alloy Especially Suited to Cladding Nuclear Fuel Elements," Serial No. 687,606, filed October 2, 1957, now Patent No. 2,941,883, issued June 21, 1960. This material, however, is subject to a sharp drop-off in ductility after it has been subjected to temperatures above about 1600° F. to 1900° F., depending upon the composition. These temperatures could be experienced in hot roll bonding, brazing or service conditions, thus limiting the usage of such alloys.

A purpose then of the present invention is to provide a new iron-aluminum base alloy which exhibits generally enhanced fabricability. Specifically, it may be extensively cold worked subsequent to preliminary ingot breakdown and grain refinement, and given annealing treatments to as high as 2000° F. to 2200° F. while maintaining an acceptable ductility. Accordingly, greater creep resistance may be obtained without sacrificing ductility.

The oxidation resistance of the present alloy is much greater than that of the stainless steels above about 1500° F. Its thermal-neutron absorption cross-section is approximately 20 to 30% lower than usual cross-sections for stainless steels now being used in nuclear reactors. Both types of material have comparable fabrication properties.

The presence of zirconium in the present alloy also imparts increased resistance to water corrosion as compared to the known prior art iron-aluminum alloys. This feature makes it particularly attractive with reference to applications in pressurized-water power reactors.

In its preferred embodiment the present alloy consists essentially by weight of from about 4%-10% Al, 2%-8% Cr, 0.5%-3% Zr and 1%-5% Cb, the remainder being Fe. There may be added, combined with the aforesaid elements, from 0-4% by weight of Mo and from 0-2% by weight of Ti. Weight percentages as expressed herein are weight percentages of the whole.

Incidental impurities may also be present, such as are associated with refined metals in very small amounts. Be-

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cause the alloy has a high content of carbide forming elements, relatively large quantities of carbon can be tolerated without loss of ductility or high temperature oxidation resistance. As a result, the alloy of the present invention can be derived from commercial ferro-metal alloys as makeup materials. Contrary to usual practice then, high purity additions are not required since carbon may be present in an amount up to about 0.20% by weight. The use of ferro-metals, such as ferrochrome, ferrocolumbium, ferromolybdenum, and ferrotitanium permits great savings over the cost of high-purity elemental constituents.

The present alloy may be prepared according to standard metallurgy techniques. Vacuum melting which is usually necessitated in making iron-aluminum base alloys like Thermenol and Alfenol is not necessary. Another exception to standard procedure, is that ferrometals may be used for makeup, as explained above.

Melting is generally performed between 2800° F. and 2900° F. Microscopic examinations of the as-cast alloy indicate the presence of an intermetallic compound which comes out as a fine dendrite structure that readily breaks up and disperses when the ingot is worked. Ingot breakdown is accomplished by means of a hot working procedure to approximately 75% reduction. Further reduction to sheet may be effected by either warm or cold rolling with intermediate anneals at 1600° F. to 1900° F.

The following rolling schedule has been found to give satisfactory results: 50% reduction at 2000° F., a second 50% reduction at 1800° F., and the remainder at about 1000° F. to 1200° F., all rolling being done at a rate of approximately 5% to 15% reduction per pass. When final thickness has been attained, the sheet is annealed at a temperature between about 1600° F. and 1900° F. However, the present alloy remains ductile after annealing at temperatures up to about 2200° F.

It will be appreciated that unusually high annealing temperatures may be used in connection with the present invention. This is made possible by zirconium additions which operate to increase alloy ductility subsequent to elevated temperature treatments. The effect of zirconium on the room temperature elongation of test specimens is illustrated in the following table, wherein every second example contains zirconium according to this invention.

Table I.—Effect of zirconium on room temperature ductility after annealing at various temperatures

No.	Alloy	Percent Elongation			
		1,400° F. Anneal	1,600° F. Anneal	1,800° F. Anneal	2,000° F. Anneal
1	7.3 Al, 5.6 Cr, 0.6 Ti, 1.0 Cb, rem. Fe.	12.5	21.0	19.0	1.5
2	7.3 Al, 4.9 Cr, 0.6 Ti, 1.2 Cb, 1.0 Zr, rem. Fe.	16.5	17.0	12.0	9.0
3	7.2 Al, 5.0 Cr, 1.0 Cb, rem. Fe.	19.0	22.0	8.5	6.5
4	7.5 Al, 4.9 Cr, 1.0 Cb, 0.5 Zr, rem. Fe.	17.0	19.0	19.0	12.0
5	6.8 Al, 4.5 Cr, 1.3 Ti, 1.0 Cb, 1.1 Mo, rem. Fe.	13.5	12.0	6.0	00.5
6	7.6 Al, 5.0 Cr, 1.0 Cb, 1.0 Mo, 2.6 Zr, rem. Fe.	12.0	13.0	14.0	11.5

As is seen from the foregoing tabulation, zirconium markedly increases ductility after annealing at 1800° F. and 2000° F. This ductility increase occurs at the expense of only a slight decrease in ductility in the neighborhood of 1400° F. to 1600° F. annealing temperatures. Accordingly, the present alloy is decidedly less brittle after being subjected to high temperature service or fabrication conditions than similar materials which do not contain zirconium.

Comparative stress-rupture tests on representative

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samples of the present alloy and similar alloys from which the zirconium had been omitted demonstrated that zirconium additions effected an outstanding increase in stress-rupture life. Experimental data are presented in Table II.

Table II.—Effect of zirconium on stress-rupture life at 1650° F. and 2000 p.s.i.

Alloy	Life (hrs.)
7.5 Al, 4.8 Cr, 0.6 Ti ¹	2.6
6.8 Al, 7.3 Cr, 1.7 Ti, 1.1 Mo ¹	3.3
7.2 Al, 4.9 Cr, 1.0 Cb ¹	8.5
6.9 Al, 7.8 Cr, 1.28 Ti, 2.68 Mo, 0.8 Si ¹	6.8
7.6 Al, 5.0 Cr, 1 Cb, 1.0 Mo, 2.6 Zr ²	42.2
7.7 Al, 4.7 Cr, 1.2 Cb, 2.1 Mo, 1.3 Zr ²	35.5
7.2 Al, 7.9 Cr, 1.1 Cb, 1.0 Mo, 1.2 Zr, 1.9 Ti ²	27.7

¹ Alloy not containing zirconium.

² Alloy containing zirconium according to this invention.

Autoclave trials of the present alloy at 680° F. have shown it to be superior to Thermenol (16 w/o Al, 3.3 w/o Mo, balance Fe), which has been accepted by several nuclear laboratories as being equivalent to the 300 series stainless steels. Corrosion resistance increases with increasing zirconium content. Comparative data are presented in Table III.

Table III.—Corrosion data for 680° F.—2500° p.s.i. water autoclave test

Alloy	Weight gain (mg./cm. ² /1,000 hrs.)
Thermenol.....	0.51
Alfenol.....	0.41
7.5 Al, 4.9 Cr, 0.5 Zr, 1.0 Cb, rem. Fe.....	0.33
7.5 Al, 5.0 Cr, 1.5 Zr, 1.0 Cb, 2.1 Mo, rem. Fe.....	0.26
7.5 Al, 5.0 Cr, 3.0 Zr, 2.0 Cb, 2.0 Mo, rem. Fe.....	0.11

In view of the fact that considerable attention during recent years has been given to the application of Thermenol as cladding material for high temperature service, some additional data was obtained with respect to the relative air-oxidation resistance and tensile strength of Thermenol as compared to representative alloys of the present invention. According to the figures in Table IV, the present alloy is substantially superior to Thermenol as regards chemical stability in air at high temperatures, and evidences tensile strengths and ductility of a much higher level.

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Table IV.—Tensile and oxidation data for thermenol and examples of the present alloy

Alloy	Ultimate Tensile Strength (p.s.i.)	Percent Elongation	mg./cm. ² /100 hrs. at 1,800° F.
Thermenol (16 w/o Al, 3 w/o Mo, rem. Fe).....	76,000	1	0.67
7.5 w/o Al, 4.8 w/o Cr, 0.5 w/o Zr, 0.9 w/o Cb, rem. Fe.....	106,000	15	0.31
6.8 Al, 4.6 Cr, 3.3 Cb, 0.6 Zr, rem. Fe.....	107,000	18	0.26

It is to be understood that many variations in the composition of the materials specifically described in detail will be apparent to a person skilled in the art, and that the present invention is limited only by the scope of the appended claims.

What is claimed is:

1. An iron-aluminum base alloy having improved ductility comprising by weight about 4% to 10% aluminum, 2% to 8% chromium, 1% to 5% columbium, and 0.5% to 3% zirconium, remainder iron.

2. An iron-aluminum base alloy having improved ductility comprising by weight about 4% to 10% aluminum, 2% to 8% chromium, 1% to 5% columbium, up to 4% molybdenum, up to 2% titanium and 0.5% to 3% zirconium, remainder iron.

3. An iron-aluminum base alloy having improved ductility comprising by weight about 7% aluminum, 5% chromium, 0.75% zirconium, 4% columbium, remainder iron.

4. An iron-aluminum base alloy having improved ductility comprising by weight about 7% aluminum, 5% chromium, 0.5% zirconium, 1% columbium, remainder iron.

5. An iron-aluminum base alloy having improved ductility comprising by weight about 7% aluminum, 5% chromium, 2% zirconium, 1% columbium, 2% molybdenum, remainder iron.

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