United States Patent [19]

Stephens et al.

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[54] **PROCESS FOR MAKING A HIGH TOUGHNESS-HIGH STRENGTH IRON ALLOY**

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[52] **U.S. Cl.** .... **148/2**; 148/12 F; 148/12.4

[58] **Field of Search** .... 148/12.4, 12 F, 2, 3, 148/31; 75/124, 125

[56] **References Cited**

U.S. PATENT DOCUMENTS

2,683,677	7/1954	Walters, Jr. et al.	75/124
3,262,777	7/1966	Sadowski	148/142
3,284,191	11/1966	Hadrean et al.	75/124
3,475,164	10/1969	Hadrean	75/124

FOREIGN PATENT DOCUMENTS

1073784	6/1967	United Kingdom	75/125

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[57] **ABSTRACT**

A steel alloy is produced by a process which includes using cold rolling at room temperature and subsequent heat treatment at temperatures ranging from 500° to 650° C.

The resulting alloy exhibits excellent strength and toughness characteristics at cryogenic temperatures. This alloy consists essentially of about 10 to 16 percent by weight nickel, to about 1.0 percent by weight aluminum, and 0 to about 3 percent by weight of at least one of the following additional elements: copper, lanthanum, niobium, tantalum, titanium, vanadium, yttrium, zirconium and the rare earth metals, with the balance being essentially iron. The improved alloy possesses a fracture toughness ranging from 200 to 230 ksi$\sqrt{\text{in.}}$ and yield strengths up to 230 ksi.

9 Claims, No Drawings

PROCESS FOR MAKING A HIGH TOUGHNESS-HIGH STRENGTH IRON ALLOY

ORIGIN OF THE INVENTION

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

STATMENT OF COPENDENCY

This application is a division of application Ser. No. 803,822 which was filed June 6, 1977.

FIELD OF THE INVENTION

This invention relates to a method of making an improved steel alloy which exhibits both high toughness and high strength at cryogenic temperatures.

BACKGROUND OF THE INVENTION

Cryogenic pressure vessels and pipes for storing and transporting liquefied gases must have high strength and toughness to be able to withstand both thermal and mechanical shocks. Further, alloys with high strength and toughness are also desirable for use in extra-terrestrial applications since the sheet thickness required is reduced, thereby saving both weight and space. It should be noted that data pertaining to the strength and toughness of alloys at room temperature does not necessarily indicate the toughness behavior at lower temperatures and alloys which exhibit high strength characteristics do not necessarily possess correspondingly high toughness.

Attempts have been made in the prior art to produce a high strength, high toughness steel alloy capable of use at cryogenic temperatures. A number of examples of such attempts can be found in the patented art. For example, U.S. Pat. No. 3,838,407 (Parker et al) discloses a Fe—12Ni—0.5Ti alloy which is said to be suitable for cryogenic use and to exhibit a Charpy V-Notch toughness value up to about 170 ksi $\sqrt{\text{in.}}$ and a yield strength of about 150 ksi. Moreover, further attempts at producing high strength steel alloys for cryogenic use are disclosed in U.S. Pat. Nos. 3,132,938 (Decker et al) and 3,514,284 (Eiselstein). The former discloses a steel comprising 17 to 19% Ni, 8 to 9% Co, 2.8 to 3.5% Mo, 0.05 to 0.15% Al as well as other elements in small amounts. The latter discloses a nickel-iron alloy which comprises 36 to 42% Ni, Nb and Ta and up to 0.015% Al, with the remainder being Fe and small amounts of other elements. Other patents of interest include U.S. Pat. Nos. 3,348,981 (Goda et al) and 3,388,988 (Nagashima et al). The former discloses an alloy which is said to possess good strength characteristics at low temperatures and which comprises 0.04% to 0.17% Al together with Mn, Cr, and C. The alloy disclosed in the Nagashima et al patent is also said to to tough at low temperatures. This alloy comprises 4.5 to 7.5% Ni, together with Al, Mn, Cr, W, Mo and other elements. Also, U.S. Pat. No. 3,338,709 (Baker et al) discloses a high strength steel said to have good toughness which comprises 8 to 10% Ni, 3 to 5% Co, 0.5 to 2.5% Mo, 0.1% Al and other elements. A final patent of interest in U.S. Pat. No. 3,232,777 (Sadowski) which relates to maraging steels using high levels of chromium and molybdenum. The steels disclosed are said to be of high strength and ultra tough. In an example of interest, the steel alloy is composed of 9.5 to 13.5% Ni, 2.5 to 8Cr, 1.9 to 4.2% Mo, up to 0.75% Al, Ti, Ni and other elements in small amounts. It will be understood that the patents discussed above are exemplary only and that this listing is in no way represented to be exhaustive.

SUMMARY OF THE INVENTION

In accordance with the invention, an improved high strength, high toughness steel alloy is provided which is particularly adapted for use at cryogenic temperatures. The steel alloy of the invention consists essentially of about 10 to 16 percent by weight nickel, 0 to about 1.0 percent by weight aluminum, and 0 to about 3 percent by weight at least one of the following additional elements: copper, hafnium, lanthanum, niobium, tantalum, titanium, vanadium, yttrium, zirconium, and the rare earth metals, with the balance essentially iron. Two embodiments of the invention which possess particularly good characteristics have the compositions Fe—12Ni—0.24Al and Fe—12Ni—0.24Al—2.0 Cu. These alloys exhibit fracture toughnesses up to 230 ksi $\sqrt{\text{in.}}$ and yield strengths of about 230 ksi.

The alloys of the invention are preferably produced by a process which comprises heating the alloy composition to produce a cast ingot, hot rolling the ingot to produce a sheet, subjecting the sheet to a second rolling operation at a temperature ranging from the temperature of the first hot rolling to room temperature, and annealing the sheet at temperatures ranging from 500° C. to 900° C., followed by water quenching. Heightened toughness and strength characteristics are obtained when the second rolling operation is made at room temperatures, followed by annealing at temperatures in the range of 500° C. to 650° C.

Other features and advantages of the invention will be set forth in, or apparent from, the detailed description of a preferred embodiment found hereinbelow.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

As discussed hereinabove, the present invention concerns an Fe—Ni—Al alloy which possesses high strength and high toughness characteristics at cryogenic temperatures. Such very low temperatures are encountered by systems wherein the steel alloys are exposed to liquefied gases or an extra-terrestrial or space environment.

As also was discussed, the alloy composition consists essentially of about 10 to 16 percent by weight nickel, 0 to about 1.0 percent by weight aluminum and 0 to about 3 percent by weight of one of the following additional elements: copper, hafnium, lanthanum, niobium, tantalum, titanium, vanadium, yttrium, zirconium, and the rare earth metals, with the balance being essentially iron. Preferred compositions of the invention are Fe—12—Ni—0.24Al and Fe—12Ni—0.24Al—2.0Cu which possess exceptional strength and toughness characteristics at cryogenic temperatures.

The Fe—Ni—Al alloy of the invention is preferably prepared by the process to be described. The process begins with melting high purity starting materials to produce an ingot. To adequately homogenize the ingot, the alloy is given a minimum of four melts. The ingot is hot rolled at 1100° C. after annealing for one-half hour at that temperature. Final rolling can be accomplished over the temperature range from that of the initial hot rolling operation to room temperature. The alloy is then subjected to heating treatment in an argon atmosphere for two hours at temperatures ranging from 500° C. to 900° C., followed by water quenching.

Certain steps in the process described above are extremely important in producing an alloy having optimum properties. In accordance with a preferred embodiment of the process includes final rolling at room temperature (cold rolling) followed by heat treatment at temperatures ranging from 500° to 650°.

The microstructure of the alloy of the invention is dependent upon the annealing conditions and the amount of reactive metals of aluminum and the additional elements of copper, hafnium, lanthanum, niobium, tantalum, titaium, vanadium, yttrium, zirconium, and the rare earth metals, which are present. For example, toughness in the Fe—12Ni—0.24Al—2.0Cu composition referred to above is achieved by the additions of nickel and the reactive element aluminum, the aluminum reacting with the interstitial impurities normally contained in iron alloys, while strengthening is achieved by the copper rich precipitates developed in this alloy which range in size from about 100 to 400 Angstroms in diameter.

Microstructural differences can be controlled by the various heat treatments used in the process of this invention. For example, the hot rolling step of the process occurs in the austentite region producing a face-centered-cubic (FCC) arrangement. On cooling this FCC structure transorms to a body-centered-cubic (BCC) structure. The air cooling utilized should be sufficiently slow to produce a microstructure consisting mainly of ferrites with some carbides and austentite present. The various annealing temperatures, and the water quenching step which follows, will determine how much of the ferrite structure is transformed to martensite. A higher annealing temperature produces a greater amount of transformed structure.

Measurements have been made to determine the toughness of the steel alloy using the following precracked Charpy method. Specimens were oriented longitudinally in the sheet bar with a 45° notch across the thickness. After annealing, each specimen was fatigue cracked to an initial crack length to specimen width ratio of approximately 0.4. Testing was conducted in a three-point bending apparatus immersed in a liquid nitrogen bath or at room temperatures. The specimen was positioned between a 6.35-mm-diameter center roller and two similar rollers that provide a support span of 38.1 mm. A crosshead speed of 1.3 millimeters per minute was used. A load/deflection curve was generated on an X-Y plotter from the outputs of a load cell which supported the bend apparatus and a double cantilever clip-in displacement gage. The gage caused the deflection by means of the vertical movement of a ceramic rod riding on the bend bar. The fracture toughness

parameter, K was determined from the load/deflector curve using the relation:

$$K = \frac{SP\sqrt{A_1 1 A_2}\,f(a/w)}{BW\,3/2}$$

where A_1 is the area under the curve to the maximum load, A_2 is the area under the curve to P, a is the specimen crack depth, B is the specimen thickness, f(a/w) is the value of the power series for the initial crack length to specimen width radio (a/w), P represents any point on the linear portions of the load/deflection curve, S is the span for the three-point bending, and W is the specimen width.

The methods used herein for measuring toughness and strength of the steel alloy of the invention are standard procedures and should be well known to those skilled in the art.

Specific properties exhibited by the steel alloys of this invention are shown in Table 1 below. The table provides a comparison with a Fe—12Ni—0.5Ti composition disclosed in U.S. Pat. No. 3,836,407 (Parker et al) discussed hereinabove. It will be understood that although the process used for the production of the Fe—12-Ni—0.5 Ti alloy of the Parker et al patent is different from that of the present invention, a comparison can be made between the elemental composition and the toughness and strength characteristics of the different alloys can be determined.

TABLE 1.

FRACTURE TOUGHNESS AND YIELD STRENGTHS OF VARIOUS Fe-12Ni ALLOYS

Testing Temperature	−196° C.						25° C.					
Annealing Temperature	550° C.		685° C.		820° C.		550° C.		685° C.		820° C.	
	Y.S.[1]	K[2]	Y.S.	K	Y.S.	K.	Y.S.	K.	Y.S.	K.	Y.S.	K.
Fe-12Ni-0.5Al	129	287	128	86	139	57	85	297	86	148	89	134
Fe-12Ni-0.24Al	130	258	160	216	151	199	88	291	129	171	101	164
Fe-12Ni-0.12Al	120	227	134	157	126	117	86	325	106	130	99	111
Fe-12Ni-0.24Al-2.2Cu	230*	200*	—	—	—	—	—	—	—	—	—	—
Fe-12Ni-0.42Ti	150	55	129	162	117	172	115	171	96	154	97	161

*Annealing temperatures of 450° C.

[1]Yield strength in ksi

[2]Fracture toughness in ksi√in

Table 1 demonstrates the enhanced toughness and strengths of the alloys of this invention, particularly as provided by the preferred embodiments of Fe—12Ni—0.24Al and Fe—12Ni—0.24Al—2.2Cu. It should be noted that the Fe—12Ni—0.24Al alloy has also exhibited good weldability. In this regard, a gas-tungsten arc weld was employed to weld this alloy using the same composition as the filler metal. Following a post weld anneal at 550° C., the fracture toughness and strength properties of the weld metal and the heat-effected-zone were equivalent to the base alloy which was heat treated at 550° C., but contained no welds. As can be seen from Table 1, the referred compositions provide fracture yield strengths of up to 230 ksi and toughness in the range of 200 to 230 ksi √in. In contrast, the prior art composition exhibits toughness and strength values approaching 172 ksi √in. and 150 ksi, respectively.

As is also evident from Table 1, the temperatures at which the alloy is annealed is very important in producing a high strength, high toughness product. A preferred range for producing optimum results lies between about 500° C. and 650° C., with alloys annealed in this range exhibiting high toughness values corresponding to that given for the 550° C. annealed condition. Testing has also shown that high strength, high toughness characteristics of steel alloys can be temperature dependent, in that the high values exhibited at room temperature may not be retained under cryogenic conditions. This is particularly evident in the Fe—12Ni—0.5Ti alloy annealed at 550° C. where the fracture toughness at 25° C. is 1.71 ksi $\sqrt{in.}$ but drops to 55 ksi $\sqrt{in.}$ at −196° C.

Although the invention has been described relative to exemplary embodiments thereof, it will be understood that other variations and modifications can be effected in this embodiments without departing rom the scope and spirit of the invention.

What is claimed is:

1. A process for producing a steel alloy possessing high strength and high toughness at cryogenic temperatures, said process comprising

heating iron and additional component materials consisting essentially of about 10 to 16 percent by weight nickel, up to about 1.0 percent by weight aluminum, and up to about 3 percent by weight of an element selected from the group consisting of: copper, hafnium, lanthanum, niobium, tantalum, titanium, vanadium, yttrium, zirconium, and the rare earth metals, to produce a cast ingot,

hot rolling said ingot to produce a sheet,

subjecting said sheet to a second rolling operation at a temperature in a range from the temperature of said first hot rolling operation to room temperature,

annealing said sheet at temperatures in a range between about 500° C. to 900° C., and

water quenching said sheet.

2. The process according to claim 1 wherein the second rolling operation is carried out at room temperature.

3. The process according to claim 2 wherein the annealing step is carried out at temperatures ranging from about 500° C. to 650° C.

4. The process according to claim 3 wherein the nickel is present in an amount of about 11 to 14 percent.

5. The process according to claim 4 wherein the nickel is present in an amount of about 12 to 12.5 percent.

6. The process according to claim 3 wherein the aluminum is present in an amount of about 0.2 to 0.5 percent.

7. The process according to claim 6 wherein the aluminum is present in an amount of about 0.24 percent.

8. The process according to claim 5 wherein the aluminum is present in an amount of about 0.24 percent.

9. The process according to claim 8 wherein the additional element comprises copper present in an amount of about 1.8 to about 2.2 percent.

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