United States Patent [19]

Chow

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[54] **HIGH POST-IRRADIATION DUCTILITY PROCESS**

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[51] **Int. Cl.** **C21d 1/00**, C21d 7/02

[58] **Field of Search** 148/12.3, 12

[56] **References Cited**

UNITED STATES PATENTS

3,347,715	10/1967	Pfeil	148/12
3,573,109	3/1971	Levy	148/12.3
3,623,920	11/1971	Kondo et al.	148/12.3
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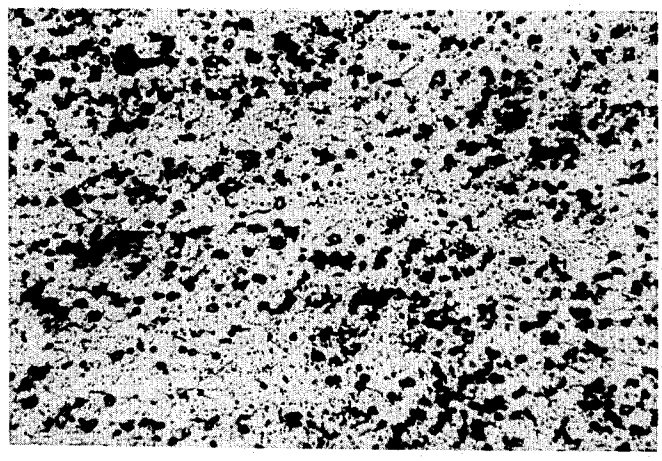
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[57] **ABSTRACT**

A method of treating certain stainless steels to improve the ductility characteristics after irradiation at high temperatures involving working at room temperatures or lower to a 90 percent reduction of area followed by low temperature recrystallization annealing.

7 Claims, 2 Drawing Figures



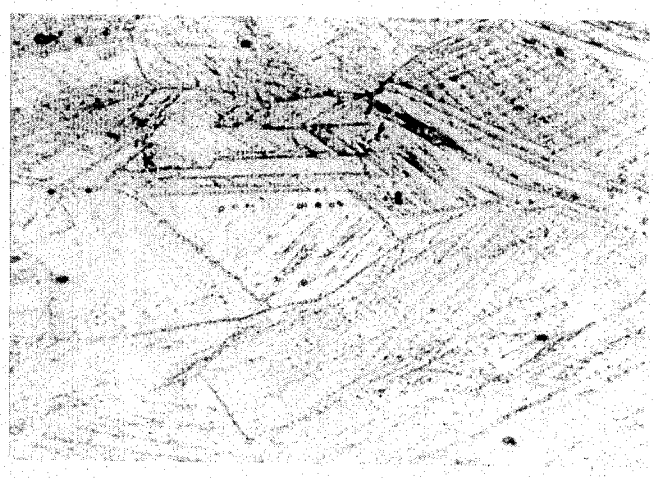


Fig. 1

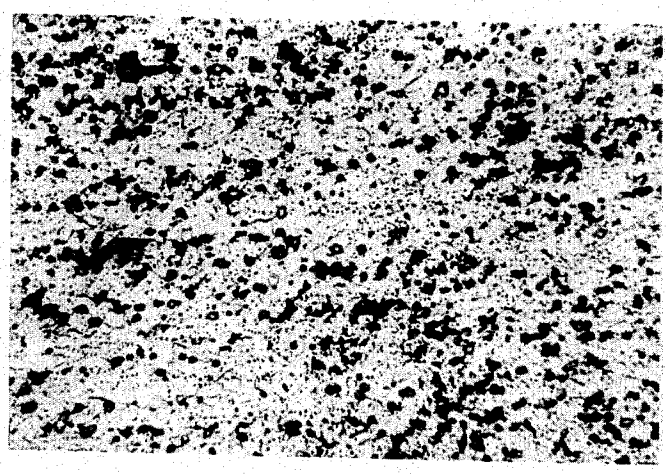


Fig. 2

HIGH POST-IRRADIATION DUCTILITY PROCESS

BACKGROUND OF THE INVENTION

The development of breeder nuclear reactors which will operate at high temperatures for long periods of time between shutdowns requires the use of materials which will not become embrittled and crack under the adverse conditions of temperature and intense radiation. For example, it is known that the effect of irradiation on stainless steel at temperatures of about 1000° F. is normally to reduce substantially the ducility of the metal. The problem of cracking in service is one of the more severe difficulties faced by breeder reactor designers.

In U.S. Pat. No. 3,620,252 there is described a process for treating cobalt alloys to improve their ducility under the described conditions. However, as cobalt during reactor service produces radioactive cobalt **60** which has a very long half life as a gamma source and some of it tends to circulate with the coolant, the resulting maintenance problems make it preferable to avoid the cobalt alloys.

SUMMARY OF PRESENT INVENTION

In accordance with this invention, it has been found that chromium nickel stainless steel alloys, which are otherwise suitable for reactor use and breeder reactors in particular, can be improved in their ability to remain ductile after being subject to irradiation at high temperatures for long periods of time. By chromium-nickel stainless steel alloys is meant for the purposes of this invention an austenitic alloy of iron having as its major alloying ingredients chromium and nickel, with chromium as the major additive and nickel as the minor additive, and have no more than 2 percent manganese. More detailed information on such stainless steel alloys including AISI designated types are given in the Metals Handbook, 8th E., Vol. 1, pub. by American Society of Metals, pp. 408–409.

In accordance with this invention, a stainless steel semifinished sample or article is cold worked until the material is too hard to work, usually when the cross section area is reduced at least 50 percent, is then given an intermediate low temperature recrystallization, is cold worked again until the maximum dimension of substantially all grains in the sample do not exceed 3 microns, the cross section area being reduced at least 90 percent, and is then given a final annealing at recrystallization temperatures to produce long term stability.

It is thus a principal object of this invention to provide an improved process for treating stainless steel alloys to retain ductility during and after irradiation.

Other objects of this invention will hereinafter become obvious from the following description of preferred embodiments of this invention.

BRIEF DESCRIPTION OF THE DRAWING

FIG. **1** is a photo-micrograph (1000 X) of a section of normal type **316** stainless steel after being subject to irradiation.

FIG. **2** is a photo-micrograph (1000 X) of similar material which had been first treated in accordance with a preferred embodiment of this invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In accordance with a preferred embodiment of this

invention a cylindrical rod of a chromium-nickel stainless steel alloy at a temperature up to 300° F, but preferably at or below room temperature is cold worked, such as by either swaging or rolling, reducing the cross section by at least 50 percent or until it becomes too hard to work, is given an intermediate low temperature anneal by heating it to at least recrystallization temperature, in the range of 1200°–1500° F. until it is soft enough to resume working, after which working is resumed until the maximum dimension of substantially (that is, at least 90 percent) all grains in the alloy is not in excess of 3 microns, and then low temperature annealing is repeated for 10–16 hours or a sufficient length of time to produce long term stability of the grain structure. The final annealing is for a substantially longer time than the intermediate annealing.

More than one intermediate annealing step may be employed, or in some cases, it may be possible to produce the 90 percent reduction in area without an intermediate annealing. Multiple annealings as described insure that an extremely fine-grain structure results, in which the maximum dimension of substantially all grains does not exceed 3 microns.

The following examples illustrate this invention;

Example 1

A 1 inch cylindrical sample of Type **316** stainless steel was cold swaged at room temperature to a diameter reduced by 45 percent and recrystallized at 870° C. for 16 hours.

The worked sample was then irradiated to 1.3×10^{20} *nvt* (E>0.82 *MeV*) and specimens were prepared and tested for ductility at several temperatures with the results shown in Table A:

TABLE A

Test Tamp., °C	Tensile Strength, psi.	Total Elong.,%
25	148,000	10
650	42,800	25
750	29,500	7
800	17,800	6.5

FIG. **1** is a photomicrograph of the microstructure prior to irradiation.

Example 2

A sample identical to that employed in the preceding example was cold swaged at room temperature down to a 1/2 inch diameter then subjected to low temperature annealing at 1200°–1500° F. for 10 hours. After cooling back to room temperature, swaging was resumed until the diameter was about 0.3 inch. The sample was again annealed, at 1400° F. for 10–16 hours, after which it was subject to the same radiation exposure as the specimen in Example 1.

Specimens were then tested for ductility at various temperatures with the results as shown in Table B.

TABLE B

Test Temp. °C.	Yield Strength psi	Tensile Strength psi	Total Elong. %
25	100,000	121,000	23
650	26,000	35,100	32
750	12,800	15,200	35
800	10,600	12,000	30

FIG. **2** is a photomicrograph of the microstructure of the specimen prior to irradiation showing the small grain size.

A comparison of the results shown in Tables A and B shows a remarkable increase in ductility of the stainless steel when the present inventive process is applied.

Examples 3 and 4

Tests corresponding with those described in the preceding examples were run utilizing Type **304** stainless steel. Similar results were obtained.

What is claimed is:

1. A process of improving resistance to postirradiation embrittlement of chromium-nickel stainless steel alloys comprising the steps of:

a. working a sample of such alloy until sufficiently hard to prevent further working;

b. subjecting said sample to an intermediate low temperature anneal at or above recrystallization temperature for a sufficient length of time to decrease hardness to permit further working;

c. reworking said sample until maximum dimension of substantially all grains do not exceed 3 microns; and

d. annealing said sample at a temperature at or above recrystallization to stabilize the crystal structure.

2. The process according to claim **1** in which working takes place up to a temperature of 300°F.

3. The process according to claim **2** in which the intermediate annealing temperature is in the range of 1200°–1500° F.

4. The process according to claim **3** in which final annealing takes place at a temperature in the range of 1200°–1500°F. until recrystallization takes place.

5. The process according to claim **4** in which the working temperature is room temperature.

6. The process according to claim **4** in which more than one intermediate annealing is employed.

7. The process according to claim **3** in which the final annealing is for a substantially longer period than the intermediate anneal to increase long term stabilization characteristics of the grain structure.

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