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(54) Ni-base alloy member and method of producing the same.

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Description

The present invention relates to a member made of a Ni-base alloy, particularly a member of a Ni-base alloy in the form of a spring or bolt used in a nuclear reactor, as well as a method of producing the same.

Hitherto, springs used in nuclear reactors have been produced by a process which has the steps of forming the spring from a blank, subjecting the formed spring to a solution heat treatment, effecting a cold rolling at a working ratio of 30% and then subjecting the rolled spring to an aging treatment. In this process, sufficiently high mechanical strength at high temperature and a superior spring property, both of which are essential requisites for the springs used in nuclear reactors, are ensured by the steps of cold working and subsequent aging treatment. Unfortunately, however, no specific consideration has been made in this conventional process in regard to the resistance against stress corrosion cracking (referred to as "SCC", hereinunder) at crevices, which is a practical problem encountered when the springs are applied to actual apparatus.

The springs, particularly springs for nuclear reactors such as the expansion spring incorporated in the control rod drives of a boiling water reactor (BWR), are often used in places where crevices exist under application of high level of stress. A countermeasure against SCC, therefore, is essential for such springs.

Currently, a material called "inconel x750" (commercial name) having a superior strength and corrosion resistance is most widely used as the material of springs intended for use in nuclear reactors. In most cases, the spring blank formed from this material is subjected to a cold working at working ratio of 30 to 40% after having been subjected to a solution heat treatment, and then subjected to a direct aging treatment. The cold working after the solution heat treatment is conducted to give a final shape to the spring. On the other hand, the aging treatment following the cold working contributes to the improvement in the spring property and strength at high temperature essential for the springs used in nuclear reactors. However, although the cold working at a working ratio of 30 to 40% is conducted between the solution heat treatment and the aging treatment, no investigation has been made as to whether this cold working at working ratio of 30 to 40% is effective for the improvement in the resistance to crevice SCC which poses a serious problem when the springs are put into practical use.

Japanese Patent Application Laid-Open No. 69517/79 mentions the intergranular corrosion in a solid solution hardening alloy but does not mention at all the SCC resistance of a precipitation hardening alloy. Unlike the solid solution hardening alloy, the precipitation hardening alloy does not exhibit any specific relationship between the crevice SCC susceptibility and the intergranular corrosion susceptibility.

EP—A—56480 published after the priority date claimed in the present application describes a member made of a Ni-base alloy and for use exposed to pure water in a nuclear reactor. The alloy has high resistance to stress corrosion cracking and consists of by weight 15 to 25% Cr, 1 to 8% Mo, 0.4 to 2% Al, 0.7 to 3% Ti, 0.7 to 4.5% Nb and balance Ni.

EP—A—66361 also published after the present priority date discloses a Ni-base alloy resistant to hydrogen embrittlement and chloride stress cracking which contains 15 to 22% Cr, 10 to 28% Fe, 6 to 9% Mo, 2.5 to 5% Nb, 1 to 2% Ti and up to 1% Al.

Accordingly, an object of the invention is to provide an Ni-base alloy member having superior SCC resistance, as well as a method of producing the same, thereby to overcome or reduce the above-described problems of the prior art.

To this end, the present inventors have conducted extensive experiments in which the working ratio of the cold working executed between the solution heat treatment and the aging treatment was varied to investigate how the crevice SCC resistance is influenced by the cold working ratio. The approach has been from two aspects: namely, a constant strain test with crevice in pure water of high temperature and pressure and metallographic observation of structure. At the same time, similar investigation was applied also to the condition of aging treatment (direct aging and two-stepped aging). As a result, the following facts were newly found by the inventors through the experiments.

(1) A cold plastic working at working ratio of 10 to 30%, executed between the solution heat treatment and the direct aging treatment, results in a large crevice SCC susceptibility. However, the crevice SCC resistance is increased when the cold plastic working is conducted at a working ratio of 40%. It proved also that this alloy has precipitate of discontinuous and granular Cr carbide in the grain boundary. In contrast, the conventional process increases the SCC susceptibility due to continuous precipitate of Cr carbide in the grain boundary. Therefore, the cold working ratio should be not smaller than 40% in terms of reduction of cross-sectional area.

(2) A cold plastic working executed at a working ratio of 10 to 20% between the solution heat treatment and two-stepped aging treatment results in a remarkable crevice SCC susceptibility. The crevice SCC susceptibility, however, is drastically decreased when the working ratio of the cold plastic working is increased beyond 25%. In this case, the relationship between the cold plastic working ratio and the crevice SCC susceptibility can easily be judged through metallography. Namely, while an apparent primary recrystallization is observed in the case of the cold plastic working at high working ratio (25% or higher) in which the crevice SCC susceptibility is extremely small, no primary recrystallization is found in the case of cold plastic working conducted at small working ratio (10 to 20%) in which the crevice SCC susceptibility is large. From this fact, it is assumed that the mechanism of crevice SCC in high temperature water occurring in the material subjected to two-stepped aging treatment is different from that of crevice SCC occurring in

the material subjected to a direct aging treatment. More specifically, it is understood that the crevice SCC in high temperature water occurring in material treated by two-stepped aging is closely related to the behaviour of the apparent primary recrystallization, rather than to the depletion of Cr due to the precipitation of Cr carbide. The apparent primary recrystallization is actually a decomposition of the matrix into a Widmanstätten type or a dendritic type structure containing Eta phase (Ni_3Ti). Such a structure exhibits a high SCC resistance. In the structure obtained through the conventional process, however, no primary recrystallization grains are observed, and further the Eta phase is precipitated in columnar form along the grain boundary and it is surrounded by precipitate free zone due to depletion of the elements consumed for formation of Eta phase. The structure obtained through the conventional process, therefore, exhibits a low SCC resistance.

The present invention has been accomplished on the basis of the newly found facts as explained hereinabove.

The method of the invention is set out in claims 1, 2, 3 and 5.

By the method there can be produced members which exhibit stress corrosion cracking depth of not greater than 200 µm when subjected to pure water of 288°C under a pressure of 86 Kg/cm^2 and having a dissolved oxygen content of 8 ppm, for 500 hours at a strain of 1%.

The alloy of the member produced preferably has a structure containing discontinuous granular precipitate of Cr carbide along the grain boundary or an apparent primary recrystallization structure which precipitates in Eta phase Widmanstätten form or in dendritic form.

When the Ni-base alloy member of the invention is produced by a process including the direct aging treatment of alloy at a temperature in the range between 650 and 750°C, the cold plastic working ratio is preferably in the range between 60 and 70% in terms of reduction of cross-sectional area.

When the Ni-base alloy member of the invention is produced by a process including the two-stepped aging treatment consisting of an aging at 800 to 950°C and another aging at 600 to 750°C, the cold plastic working ratio is preferably in the range between 40 and 70% in terms of reduction of cross-sectional area. This process affords an advantage in that the working ratio can be made smaller than that in the process including direct aging.

Hereinunder, an explanation will be made as to the reasons for the numerical restrictions imposed on the contents of the components.

For attaining a sufficiently high SCC resistance, the Cr content should be 14% at the smallest, but any Cr content exceeding 25% degrades the hot workability and, in addition, a noxious phase known as TCP phase is formed to deteriorate the cold workability, mechanical properties and corrosion resistance. From these points of view, the Cr content is preferably in the range between 14 and 25%, more preferably 14 and 20% and most preferably 14 and 17%.

Fe is an element which is effective in stabilizing the structure of the matrix and improving the corrosion resistance. A too large Fe content, however, permits the production of noxious phase such as Laves phase and, therefore, the Fe content is preferably not greater than 30%, more preferably not greater than 10% and most preferably 5 to 9%.

Al, Ti and Nb contribute to the precipitation hardening due to formation of intermetallic compounds through reaction with Ni. In order to obtain an age hardenability, however, at least a combination of not smaller than 0.2% of Al and not smaller than 0.5% of Ti is essential. It is possible to obtain alloys of desired high strength by increasing the Al and Ti contents while adding Nb thereto, but the property is degraded if the Al, Ti and Nb contents are increased excessively. Therefore, Al, Ti and Nb contents are preferably 0.2 to 2% 0.5 to 3% and 0.7 to 4.5%, respectively. More preferably, the Al, Ti and Nb contents are 0.4 to 1.0%, 2 to 3% and 0.7 to 1.5%, respectively.

Nb has greater effect on the precipitation hardening than Al and Ti and, therefore, the addition of Nb is necessary for obtaining high hardenability required especially for the material of springs and bolts used in nuclear reactors. However, a too large Nb content will undesirably deteriorate the mechanical properties and reduce the workability due to formation of coarse carbide and intermetallic compounds. Therefore, the Nb content is preferably in the range between 0.5 and 1.5%.

In order to increase the strength at room temperature it is necessary to add C, but the C content should not exceed 0.15% because C content exceeding 0.15% inconveniently lowers the cold plastic workability. The content is preferably in the range between 0.02 and 0.08%.

Not greater than 1% of Si and not greater than 1.5% of Mn are added as deoxidizers in the course of production of the alloy. Addition of more than 1% of Si and more than 1.5% of Mn undesirably lowers the cold plastic workability of the alloy. Preferably, the Si content and Mn content are selected to range between 0.02 and 0.5% and 0.1 and 1%, respectively.

In the Ni-base alloy member of the invention, the balance of the composition consists substantially of Ni. The Ni content is preferably not smaller than 65%, and more preferably not smaller than 70%.

The direct aging treatment is conducted at a temperature in the range between 650 and 750°C. The two-stepped aging treatment is conducted preferably by heating the material to and maintaining the same at 800 to 900°C for 1 to 30 hours, cooling the material and then heating to and maintaining at 600 to 750°C for 1 to 30 hours. It is also preferred that the solution heat treatment is conducted at a temperature ranging between 950 and 1150°C.

Embodiments of the invention are described below by way of example with reference to the drawings, in which

Figs. 1a to 1f are illustrations of various springs used in a nuclear reactor, as embodiments of the Ni-base alloy member in accordance with the invention;

Figs. 2(a) to (d) are microscopic photos of the structures of Ni-base alloy member of the invention obtained through a direct aging treatment (magnification 400);

Figs. 3(a) to (d) are microscopic photos of structures of Ni-base alloy member of the invention obtained through two-stepped aging treatment (magnification 400); and

Fig. 4 shows microscopic photos at a large magnification of structures which are obtained through direct aging treatment and two-stepped aging treatment, respectively.

The members of the Ni-base alloys of the invention are described here particularly in the form of springs and bolts which are subjected to pure water of high pressure and temperature in nuclear reactors.

Figs. 1a to 1f illustrate shapes of various springs and bolts used in nuclear reactors. A description will be made hereinafter as to the natures of these springs and bolts, as well as the method of producing these springs and bolts.

Figs. 1a and 1b illustrate an expansion spring 12 which is used for securing graphite seal segments 11 to the inner surface of an index tube 10. The expansion spring 12 has the form of a web-like ring cut at a portion thereof as at 13. The breadth or axial height is 10 mm while the diameter is 60 mm. This expansion ring 12 is made by a process having the steps of preparation of molten alloy material, solidification, rolling, solution heat treatment, cold rolling at a working ratio of 25% or higher, forming and two-stepped aging treatment.

Figs. 1c and 1d illustrate a garter spring 22 for securing graphite seal segments 21 to a piston tube 20. The garter spring 22 is a coiled spring having a coil length of 166 mm and formed from a wire of 0.36 mm dia. This garter spring is produced by a process which has the steps of preparation of molten alloy material, solidification, solution heat treatment, drawing, coiling at a working ratio of 25% or higher and two-stepped aging treatment.

Fig. 1e shows a spring 32 which is interposed between a tie plate 30 and a channel box 31, while Fig. 1f shows a spring 41 associated with a cap screw 40. These springs 32 and 41 are produced by the same process as the expansion spring shown in Figs. 1a and 1b. In Fig. 1f, a reference numeral 42 designates a guard. The cap screw 40 is produced by a process having the steps of forging or rolling, solution heat treatment, and threading by thread rolling or machining. The aforementioned two-stepped aging treatment is conducted following the threading.

Example

Inconel x750 (commercial name) consisting essentially of, by weight, 72.92% of Ni, 15.48% of Cr, 6.91% of Fe, 0.57% of Al, 2.60% of Ti, 0.95% of Nb+Ta, 0.04% of C, 0.20% of Si and 0.23% of Mn was used as the blank.

Table 1 shows the result of a crevice constant strain test conducted in pure water of high temperature and pressure under the following condition.

test temperature: 288°C
pressure: 86 Kg/cm^2
dissolved oxygen content: 8 ppm
crevice forming material: graphite fiber wool
strain: about 1.0%
test time: 500 hours

Table 1

alloy	aging treatment condition	cold working ratio	crevice SCC susceptibility			
Inconel x750	direct aging treatment	0%	◑	◑	○	○
		10%	●	●	●	●
		20%	●	●	●	●
		30%	●	●	●	●
		40%	○	○	○	○
		50%	○	○	○	○
		60%	○	○	○	○
	two-stepped aging treatment	0%	●	●	●	●
		10%	●	●	●	●
		20%	●	●	●	●
		30%	○	○	○	○
		60%	○	○	○	○

● : high SCC susceptibility (crack depth greater than 1000 µm)

◑ : medium SCC susceptibility (crack depth 200 µm to 1000 µm)

○ : low SCC susceptibility (crack depth 0 to 200 µm)

From Table 1, it will be understood that, when the inconel x750 is subjected to a cold plastic working at a reduction of cross-sectional area of 10 to 60% after a solution heat treatment (1066°C 1-hour heating followed by water cooling) and before a direct aging treatment (704°C 4 to 20-hours heating followed by air cooling), the crevice SCC susceptibility is varied according to the value of the reduction of cross-sectional area. Namely, the crevice SCC susceptibility is impractically large when the cold plastic working is conducted respectively at reduction of cross-sectional area of 10, 20 and 30%. This means that the cold plastic working at working ratio not greater than 30% is detrimental to the crevice SCC resistance. The crevice SCC susceptibility is remarkably decreased as the cold plastic working ratio is increased to 50% and then to 60%. Such large cold plastic working ratio, however, is obtainable only through a heavy cold plastic working.

In the case where the same cold plastic working is applied between a solution heat treatment (1066°C 1-

hour heating followed by water cooling) and a two-stepped aging treatment (843°C 24-hours heating followed by air cooling, and 704°C 4 to 20-hours heating followed by air cooling), the crevice SCC resistance is remarkably improved when the cold plastic working is conducted respectively at a working ratio of 30 and 60%, although the crevice SCC susceptibility is still high when the working ratio is 10% and 20%.

In order to examine how the crevice SCC susceptibility of inconel x750 is affected by the metallography, test pieces of this material were immersed in a mixed acid (92 ml HCl + 3 ml HNO_3 + 5 ml H_2SO_4) and were observed by a microscope to obtain microscopic photos (magnification 400) as shown in Figs. 2(a) to (d) and Figs. 3(a) to (d). More specifically, Figs. 2(a) to (d) show the metallographies as obtained through a direct aging treatment when the cold working ratio is 10%, 20%, 30% and 60%, respectively, while Figs. 3(a) to (d) shows those obtained through two-stepped aging treatment when the cold plastic working is conducted at working ratio of 10%, 20%, 30% and 60%, respectively. When the aging treatment is conducted through a direct aging, the grains become deformed as the cold working ratio is increased. However, no specific relationship is observed between the crevice SCC susceptibility and the metallography. On the other hand, when the aging is conducted through a two-stepped aging treatment, the metallographies exhibiting large crevice SCC susceptibility show only intergranular corrosion, whereas the metallographies exhibiting small crevice SCC susceptibility show a comparatively large number of apparent primary recrystallization grains, as well as intergranular corrosion. This suggests that there is a certain relationship between the apparent primary recrystallization behaviour and the crevice SCC susceptibility in hot water, when the aging is conducted through two-stepped aging treatment, and that the crevice SCC resistance of the alloy of the invention can be improved effectively by finally attaining a metallography of apparent primary recrystallization structure through a suitable combination of the cold working ratio and heat treatment. The apparent primary recrystallization is ruled by the cold working ratio and the condition of the intermediate heat treatment. More specifically, the apparent primary recrystallization is promoted to remarkably improve the crevice SCC resistance, as the cold working ratio is increased.

Fig. 4 shows microscopic photos of metallographies of inconel x750 alloy subjected to direct aging treatment and two-stepped aging treatment, after cold plastic working at working ratio of 0%, 30%, and 60%, respectively. In the alloy subjected to the direct aging treatment, continuous precipitate of Cr carbide is observed along the grain boundary when the cold plastic working ratio is 0% and 30%. It is, therefore, understood that the low crevice SCC resistance in the alloy cold-worked at such low working ratio is attributable to the continuous precipitate of Cr carbide in the grain boundary. In contrast, in the alloy subjected to a plastic working at working ratio of 60% in accordance with the invention, no continuous precipitate of Cr carbide is found along the grain boundary, but the Cr carbide is precipitated in a granular form along the grain boundary and within the grain.

On the other hand, in the alloy subjected to two-stepped aging treatment under the cold plastic working ratio of 0%, columnar precipitate of Eta phase (Ni_3Ti intermetallic compound) is formed along the grain boundary and is surrounded by Precipitate Free Zone. It is, therefore, understood that the low crevice SCC resistance in the alloy having cold working ratio of 0% is attributable to the columnar precipitate of the Eta phase and the presence of the precipitate free zone around the Eta phase. Unlike the case of the cold working ratio of 0%, the Eta phase is precipitated in Widmanstätten or dendritic form in the alloy subjected to cold plastic working at ratio of 30% and 60% according to the invention. The phase itself appearing as apparent primary recrystallization structure mentioned before owes to this dendritic Eta phase.

From the facts described heretofore, it is understood that the cold working at a working ratio not smaller than 40% is effective in the production of springs and bolts having superior crevice SCC resistance suitable for use in nuclear reactors, when the aging is conducted through a direct aging treatment, whereas, when the aging is conducted through two-stepped aging treatment, the cold working at a working ratio not smaller than 25% is effective. In this case, it is understood also that the crevice SCC resistance can easily be evaluated through the observation of the metallography, i.e. the state of precipitate of Cr carbide and the existence of the apparent primary recrystallization structure.

Claims

1. A method of producing a member made of a Ni-base alloy of high stress corrosion cracking resistance comprising the steps of: providing an alloy consisting of, by weight, not more than 0.15% of C, not more than 1% of Si, not more than 1.5% of Mn, 14 to 25% of Cr, not more than 30% of Fe, 0.2 to 2% of Al, 0.5 to 3% of Ti, 0.7 to 4.5% of Nb and the balance Ni and unavoidable impurities, the amount of Ni being at least 65%; subjecting said alloy to a solution heat treatment; effecting a cold plastic working on said alloy with a reduction of cross-sectional area of between 50 and 70%; and subjecting said alloy to an aging treatment at a temperature in the range between 650 and 750°C.

2. A method of producing a member made of a Ni-base alloy of high stress corrosion cracking resistance comprising the steps of: providing an alloy consisting of, by weight, 0.02 to 0.08% of C, 0.02 to 0.5% of Si, 0.1 to 1% of Mn, 14 to 20% of Cr, not more than 10% of Fe, 0.4 to 1% of Al, 2 to 3% of Ti, 0.7 to 1.5% of Nb and the balance Ni and unavoidable impurities, the amount of Ni being at least 65%; subjecting said alloy to a solution heat treatment; effecting a cold plastic working on said alloy with a reduction of cross-sectional area of between 50 and 70%; and subjecting said alloy to an aging treatment at a

temperature of between 650 and 750°C.

3. A method of producing a member made of a Ni-base alloy of high stress corrosion cracking resistance comprising the steps of: providing an alloy consisting of, by weight, not more than 0.15% of C, not more than 1% of Si, not more than 1.5% of Mn, 14 to 25% of Cr, not more than 30% of Fe, 0.2 to 2% of Al, 0.5 to 3% of Ti, 0.7 to 4.5% of Nb and the balance Ni and unavoidable impurities, the amount of Ni being at least 65%; subjecting said alloy to a solution heat treatment; effecting a cold plastic working on said alloy with a reduction of cross-sectional area of between 30 and 70%; subjecting said alloy to an aging treatment at a temperature of between 800 and 950°C; and subjecting said alloy to another aging treatment at a temperature of between 600 and 750°C.

4. A method according to claim 3, wherein said cold plastic working is effected at a reduction of cross-sectional area of between 40 and 70%.

5. A method of producing a member made of a Ni-base alloy of high stress corrosion cracking resistance comprising the steps of: providing an alloy consisting of, by weight, 0.02 to 0.8% of C, 0.02 to 0.5% of Si, 0.1 to 1% of Mn, 14 to 20% of Cr, not more than 10% of Fe, 0.4 to 1% of Al, 2 to 3% of Ti, 0.7 to 1.5% of Nb and the balance Ni and unavoidable impurities, the amount of Ni being at least 65%; subjecting said alloy to a solution heat treatment; effecting a cold plastic working on said alloy with a reduction of cross-sectional area of not less than 25%; subjecting said alloy to an aging treatment at a temperature of between 800 and 950°C; and subjecting said alloy to another aging treatment at a temperature of between 600 and 750°C.

6. A method according to claim 5, wherein said cold plastic working is effected with a reduction of cross-sectional area of between 40 and 70%.

7. A member made of a Ni-base alloy produced by a method according to any one of the preceding claims which has a structure containing discontinuous granular precipitate of Cr carbide along the grain boundary or an apparent primary recrystallization structure.

8. Use of a member made of Ni-base alloy produced by a method according to any one of the preceding claims as a component of a nuclear reactor exposed to pure water of high temperature and pressure.

9. Use of a member made of a Ni-base alloy according to claim 8, as a spring or a bolt exposed to pure water of high temperature and pressure in a nuclear reactor.

Patentansprüche

1. Verfahren zum Herstellen eines Bauteiles aus einer Legierung auf Ni-Basis mit hoher Spannungskorrosionsrißfestigkeit, bei dem eine Legierung gebildet wird, die aus nicht mehr als 0,15 Gew.% C, nicht mehr als 1 Gew.% Si, nicht mehr als 1,5 Gew.% Mn, 14 bis 25 Gew.% Cr, nicht mehr als 30 Gew.% Fe, 0,2 bis 2 Gew.% Al, 0,5 bis 3 Gew.% Ti, 0,7 bis 4,5 Gew.% Nb and dem Rest Ni sowie unvermeidlichen Verunreinigungen besteht, wobei die Menge an Ni wenigstens 65 Gew.% beträgt, diese Legierung einer Lösungsglühbehandlung unterworfen wird, an dieser Legierung eine plastische Kaltbearbeitung mit einer Abnahme der Querschnittsfläche zwischen 50 und 70% bewirkt wird und diese Legierung eine Alterungsbehandlung auf einer Temperatur zwischen 650 und 750°C unterworfen wird.

2. Verfahren zum Herstellen eines Bauteiles aus einer Legierung auf Ni-Basis mit hoher Spannungskorrosionsrißfestigkeit, bei dem eine Legierung gebildet wird, die aus 0,02 bis 0,08 Gew.% C, 0,02 bis 0,5 Gew.% Si, 0,1 bis 1 Gew.% Mn, 14 bis 20 Gew.% Cr, nicht mehr als 10 Gew.% Fe, 0,4 bis 1 Gew.% Al, 2 bis 3 Gew.% Ti, 0,7 bis 1,5 Gew.% Nb and dem Rest Ni sowie unvermeidlichen Verunreinigungen besteht, wobei die Menge an Ni wenigstens 65 Gew.% beträgt, diese Legierung einer Lösungsglühbehandlung unterworfen wird, an dieser Legierung eine plastische Kaltbearbeitung mit einer Abnahme der Querschnittsfläche zwischen 50 und 70% bewirkt wird und diese Legierung eine Alterungsbehandlung auf einer Temperatur zwischen 650 und 750°C unterworfen wird.

3. Verfahren zum Herstellen eines Bauteiles aus einer Legierung auf Ni-Basis mit hoher Spannungskorrosionsrißfestigkeit, bei dem eine Legierung gebildet wird, die aus nicht mehr als 0,15 Gew.% C, nicht mehr als 1 Gew.% Si, nicht mehr als 1,5 Gew.% Mn, 14 bis 25 Gew.% Cr, nicht mehr als 30 Gew.% Fe, 0,2 bis 2 Gew.% Al, 0,5 bis 3 Gew.% Ti, 0,7 bis 4,5 Gew.% Nb und dem Rest Ni sowie unvermeidlichen Verunreinigungen besteht, wobei die Menge an Ni wenigstens 65 Gew.% beträgt, diese Legierung einer Lösungsglühbehandlung unterworfen wird, an dieser Legierung eine plastische Kaltbearbeitung mit einer Abnahme der Querschnittsfläche zwischen 30 und 70% bewirkt wird, diese Legierung einer Alterungsbehandlung auf einer Temperatur zwischen 800 und 950°C unterworfen wird und diese Legierung einer weiteren Alterungsbehandlung auf einer Temperatur zwischen 600 und 750°C unterworfen wird.

4. Verfahren nach Anspruch 3, bei dem die plastische Kaltbearbeitung mit einer Abnahme der Querschnittsfläche zwischen 40 und 70% bewirkt wird.

5. Verfahren zum Herstellen eines Bauteiles aus einer Legierung auf Ni-Basis mit hoher Spannungskorrosionsrißfestigkeit, bei dem eine Legierung gebildet wird, die aus 0,02 bis 0,8 Gew.% C, 0,02 bis 0,5 Gew.% Si, 0,1 bis 1 Gew.% Mn, 14 bis 20 Gew.% Cr, nicht mehr als 10 Gew.% Fe, 0,4 bis 1 Gew.% Al, 2 bis 3 Gew.% Ti, 0,7 bis 1,5 Gew.% Nb and dem Rest Ni sowie unvermeidlichen Verunreinigungen besteht, wobei die Menge an Ni wenigstens 65 Gew.% beträgt, diese Legierung einer Lösungsglühbehandlung unterworfen wird, an dieser Legierung eine plastische Kaltbearbeitung mit einer Abnahme der Quer-

schnittsfläche von nicht weniger als 25% bewirkt wird, diese Legierung einer Alterungsbehandlung auf einer Temperatur zwischen 800 und 950°C unterworfen wird und diese Legierung einer weiteren Alterungsbehandlung auf einer Temperatur zwischen 600 und 750°C unterworfen wird.

6. Verfahren nach Anspruch 5, bei dem die plastische Kaltbearbeitung mit einer Abnahme der Querschnittsfläche zwischen 40 und 70% bewirkt wird.

7. Bauteil aus einer Legierung auf Ni-Basis, hergestellt mit einem Verfahren nach einem der vorhergehenden Ansprüche, das ein Gefüge, das eine diskontinuierliche körnige Ausscheidung von Cr-Carbid entlang der Korngrenze enthält, oder ein scheinbares, primäres Rekristallisationsgefüge hat.

8. Verwendung eines Bauteiles aus einer Legierung auf Ni-Basis, hergestellt mit einem Verfahren nach einem der vorhergehenden Ansprüche, als Bauteil eines Atomkernreaktors, das reinem Wasser auf hoher Temperatur und hohem Druck ausgesetzt ist.

9. Verwendung eines Bauteiles aus einer Legierung auf Ni-Basis nach Anspruch 8, als Feder oder Bolzen, die oder der reinem Wasser auf hoher Temperatur und hohem Druck in einem Atomkernreaktor ausgesetzt ist.

Revendications

1. Procédé de fabrication d'un élément constitué d'un alliage à base de nickel ayant une résistance élevée à la fissuration par corrosion sous tension comprenant les étapes consistant: à préparer un alliage constitué, en poids, de pas plus de 0,15% de C, pas plus de 1% de Si, pas plus de 1,5% de Mn, entre 14 et 25% de Cr, pas plus de 30% de Fe, entre 0.2 et 2% de Al, entre 0,5 et 3% de Ti, entre 0,7 et 4,5% de Nb et le reste en Ni et en impuretés inévitables, la teneur en Ni étant au moins de 65%; à soumettre ledit alliage à un traitement thermique de mise en solution; à effectuer un travail de déformation plastique à froid sur ledit alliage avec une réduction de surface de la section transversale entre 50 et 70%; et à soumettre ledit alliage à un traitement de vieillissement à une température située entre 650 et 750°C.

2. Procédé de fabrication d'un élément constitué d'un alliage à base de nickel ayant une résistance élevée à la fissuration par corrosion sous tension comprenant les étapes consistant: à préparer un alliage constitué, en poids, de 0,02 à 0,08% de C, de 0,02 à 0,5% de Si, de 0,1 à 1% de Mn, de 14 à 20% de Cr, pas plus de 10% de Fe, de 0,4 à 1% d'Al, de 2 à 3% de Ti, de 0.7 à 1,5% de Nb et le reste en Ni et en impuretés inévitables, la teneur en Ni étant au moins de 65%; soumettre ledit alliage à un traitement thermique de mise en solution, à effectuer un travail de déformation plastique à froid sur ledit alliage avec une réduction de surface de la section transversale entre 50 et 70%; et à soumettre ledit alliage à un traitement de vieillissement à une température comprise entre 650 et 750°C.

3. Procédé de fabrication d'un élément constitué d'un alliage à base de nickel ayant une résistance élevée à la fissuration par corrosion sous tension comprenant les étapes consistant: à préparer un alliage constitué, en poids, de pas plus de 0,15% de C, pas plus de 1% de Si, pas plus de 1,5% de Mn, de 14 à 25% de Cr, pas plus de 30% de Fe, de 0,2 à 2% d'Al, de 0,5 à 3% de Ti, de 0.7 à 4,5% de Nb et le reste en Ni et en impuretés inévitables, la teneur en Ni n'étant pas inférieure à 65%; à soumettre ledit alliage à un traitement thermique de mise en solution; à effectuer un travail de déformation plastique à froid sur ledit alliage avec une réduction de surface de la section transversale entre 30 et 70%; à soumettre ledit alliage à un traitement de vieillissement à une température comprise entre 800 et 950°C; et à soumettre ledit alliage à un autre traitement de vieillissement à une température comprise entre 600 et 750°C.

4. Procédé selon la revendication 3, dans lequel ledit travail de déformation plastique à froid est effectué avec une réduction de la surface de la section transversale comprise entre 40 et 70%

5. Procédé de fabrication d'un élément constitué d'un alliage à base de nickel ayant une résistance élevée à la fissuration par corrosion sous tension comprenant les étapes consistant à: préparer un alliage constitué, en poids, de 0,02 à 0,8% de C, 0,02 à 0,5% de Si, de 0,1 à 1% de Mn, de 14 à 20% de Cr, pas plus de 10% de Fe, de 0,4 à 1% d'Al, de 2 à 3% de Ti, de 0.7 à 1,5% de Nb et le reste en Ni et en impuretés inévitables, la teneur en Ni étant d'au moins 65%; à soumettre ledit alliage à un traitement thermique de mise en solution; à effectuer un travail de déformation plastique à froid sur ledit alliage avec une réduction de la surface de la section transversale qui n'est pas inférieure à 25%; à soumettre ledit alliage à un traitement de vieillissement à une température comprise entre 800 et 950°C; et à soumettre ledit alliage à un autre traitement de vieillissement à une température comprise entre 600 et 750°C.

6. Procédé selon la revendication 5, dans lequel ledit travail de déformation plastique à froid est effectué avec une réduction de la surface de la section transversale comprise entre 40 et 70%.

7. Elément fabriqué en un alliage à base de nickel obtenu par un procédé selon l'une quelconque des revendications précédentes qui a une structure contenant des précipités granulaires discontinus de carbure de Cr le long des limites des grains ou une structure apparente de recristallisation primaire.

8. Utilisation d'un élément fabriqué en alliage à base de nickel obtenu par un procédé selon l'une quelconque des revendications précédentes, comme composant d'un réacteur nucléaire exposé à de l'eau pure à haute température et à haute pression.

9. Utilisation d'un élément fabriqué en alliage à base de nickel selon la revendication 8, sous forme d'un ressort ou d'un boulon exposé à de l'eau pure à haute température et à haute pression dans un réacteur nucléaire.

FIG. 1a

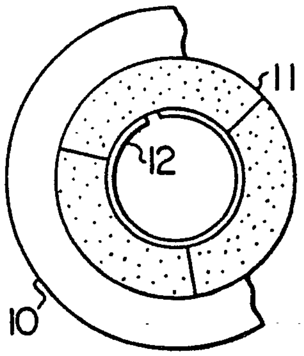


FIG. 1b

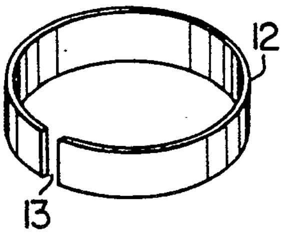


FIG. 1c

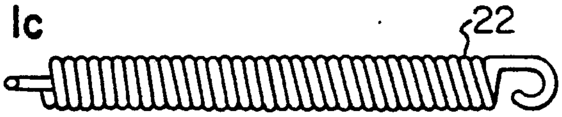


FIG. 1d

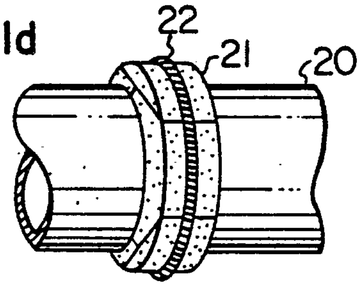


FIG. 1e

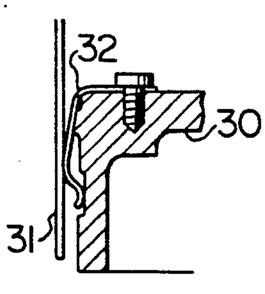


FIG. 1f

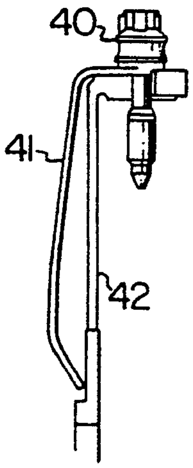
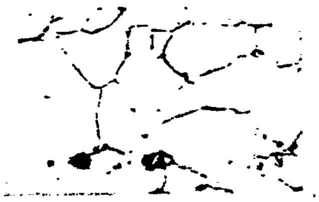
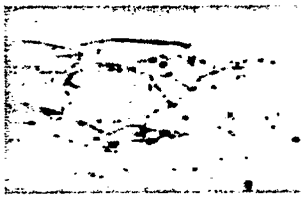
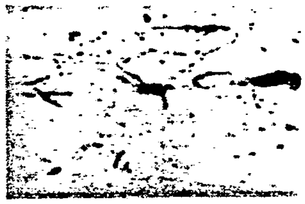
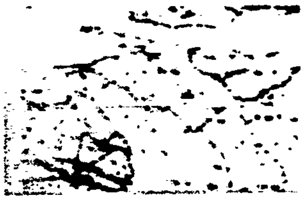
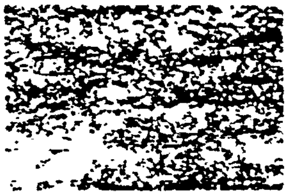
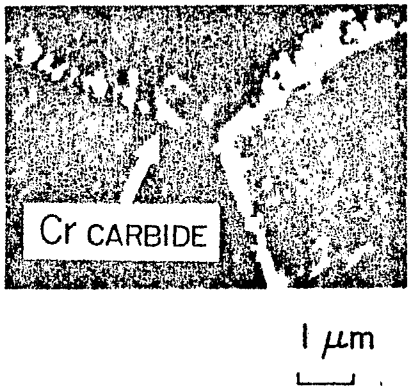
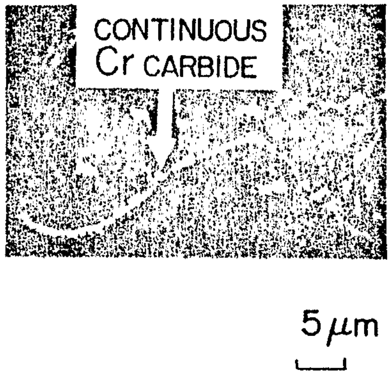
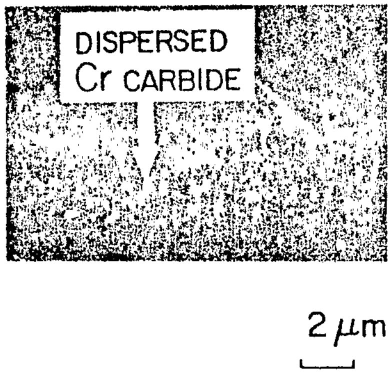
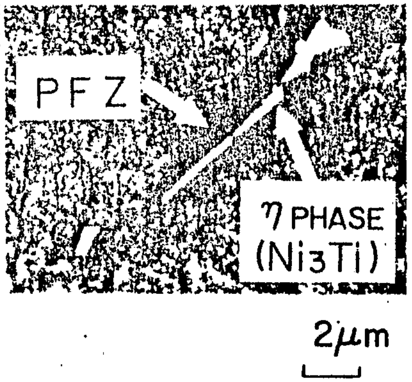
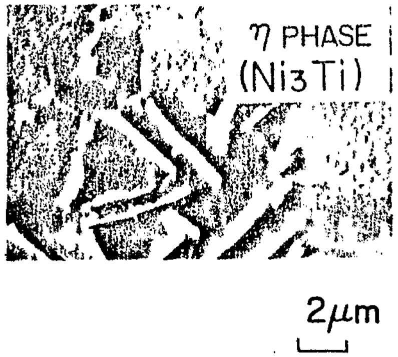
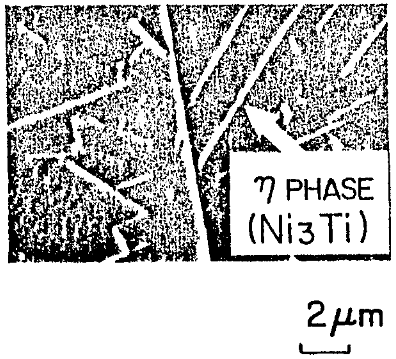


	FIG. 2	FIG. 3
COLD PLASTIC WORKING RATIO (%)	704°c, 20h	843°c, 24h + 704°c, 20h
0 (a)		
20 (b)		
30 (c)		
60 (d)		

25µm 25µm

1066°c, 1h → COLD PLASTIC WORKING → AGING TREATMENT

FIG. 4

HEAT TREATMENT \ COLD PLASTIC WORKING RATIO (%)	0	30	60
DIRECT AGING TREATMENT 1066°c, 1h ↓ COLD PLASTIC WORKING ↓ 704°c, 20h			
TWO-STEPPED AGING TREATMENT 1066°c, 1h ↓ COLD PLASTIC WORKING ↓ 843°c, 24h ↓ 704°c, 20h			

IDENTIFICATION OF PRECIPITATE : AES ANALYSIS