

EFFECTS OF COLD WORKING ON MICROSTRUCTURAL AND MECHANICAL

PROPERTIES AND SCC BEHAVIOR OF ALLOY 718

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Abstract

Mechanical properties, hardening kinetics and SCC behavior in doped steam has been studied for several microstructures of alloy 718. This study showed that :

- no relation exists between strength level and SCC resistance of this material
- a microstructure with δ phase does not necessarily involve worse behavior
- cold working has a strong influence on hardening kinetics and strength
- very high strength can be achieved on slightly cold worked alloy 718 without affecting SCC resistance.

INTRODUCTION

In PWR primary coolant, Alloy 718 is used in the manufacture of highly stressed components such as bolts and springs. Until now, the field experience never mentions in-service failures caused by stress corrosion cracking (SCC) phenomena. (1-5). However, numerous laboratory studies show that initiation of intergranular Stress corrosion cracks can occur in PWR primary coolant (6-13). These studies concluded also that the initiation of these cracks is always very difficult. On the other hand, the alloy 718 exhibits a low resistance to crack growth by SCC mode (13-16), with probably a synergetic effect of hydrogen embrittlement (13).

It is also acknowledged that the microstructure influences severely the initiation and the growth of SC cracks (3, 6, 9). Cold work does not increase the susceptibility to SCC of the alloy (17,18). On the contrary, in BWR water, susceptibility to SCC seems to be decreased by cold work in the range 10 to 30% (17). For instance, δ and Laves phases weaken the resistance of alloy 718 to SCC in PWR primary environment (13,15,19). Presently, several products with very different microstructures and mechanical properties are used for the components in the nuclear industry (2).

Nevertheless, several applications require such high strength properties (YS > 1400 Mpa) that fabrication processes to be modified to such an extent that in-service behaviour may be affected.

In order to reach so high values of YS, it is necessary to cold work the products before aging thermal treatment. The effects of cold work on strength and ductility properties of alloy 718 are quite complex. Heacox (20) shows that strength and ductility level do not depend only on cold reduction but are also related to other parameters such as :

- grain size (influence of the thermal treatment before last elongation phase)
- aging steps (holding time, temperature)
- sizes of the product.

Finally, the process of fabrication will influence drastically the mechanical behavior and probably the SCC resistance in PWR environment of alloy 718. In this study, we present some results concerning the practice of cold work in the alloy 718 process and its efficiency to promote high mechanical properties. We deal also with the SCC resistance of this alloy in PWR primary water.

MATERIALS

Three industrial products in alloy 718 are used in this study. They are described in the table 1.

heat	Type of product	elaboration	description
1	wire $\varnothing = 8.13$ mm	VIM + VAR	cold work : 10%
2	sheet 4 x 21 mm	VIM + VAR	cold work : 20%
3	sheet 4 x 21 mm	VIM + VAR	cold work : 16%

Table n° 1 - Description of the products

Chemical composition of the three industrial heats are presented in the table n°2.

Heat	C (w%)	Mn (w%)	Si (w%)	S (w%)	P (w%)	Ni (w%)	Cr (w%)	Mo (w%)
1	0.024	0.03	0.07	0.0017	0.003	bal	17.89	2.97
2	0.034	0.03	0.02	0.002	0.008	bal	17.86	2.98
3	0.032	0.02	0.06	0.001	0.002	bal	17.96	3.02

Heat	Cu (w%)	Co (w%)	Ti (w%)	Al (w%)	B (ppm)	Nb (w%)	Fe (w%)
1	0.01	0.01	0.97	0.54	46	5.25	18
2	<0.1	-	0.94	0.51	< 50	5.16	18.5
3	0.01	0.01	0.95	0.54	50	5.26	19.2

Table n° 2 - Chemical composition of the heats.

The sheets have been used in the cold work studies. After annealing (970°C- 1h), they have been cold worked by pulling at different strain ratios comprised in the range from 0% to 25%. Different heat treatments have been performed on the sheets :

- one step aging treatment with the temperature in the range 660 to 780°C and duration in the range 1 to 16 hours,
- the two-step standard aging treatment : 720°C-8 h / furnace cooling (50°C/h) / 620°C-8 h / air cooling.

The wire has been used in the corrosion experiments. Solution annealing has been performed on samples after final cold work :

- 970°C / 1 h / Water cooling
- 1040°C / 1 h / Water cooling

These samples have been then aged with the standard treatment.

EXPERIMENTAL TECHNIQUES

The **tensile test** are conventional with the following conditions :

- test temperature : 25°C
- displacement speed : 1mm /min
- extensometer with a length capability of 10 mm
- gauge length : 20 mm

Vickers hardness test is conducted with a weight of 30kg on sample surfaces polished mechanically with paper grade 1200.

For **metallography**, the samples have been extracted in SL and ST direction, polished mechanically with paper grade 1200 and edged under controlled potential in chloro-acetic acid solution (3V, 10 s).

For **corrosion test**, a bent-beam four-point loaded specimen has been used. The specimen is a flat strip machined in the rod and mechanically polished until diamond paste (1µm) . The dimensions of the strip are 48 x 6 x 2 mm. The holder is composed of two pieces and a screw of stainless steel ; the four contact cylinders located between the specimen and the holder are hard alloy rods. The specimen is not insulated from the holder but it is not necessary since the test is performed in dry steam at high temperature. The deflection of the specimen is applied and measured as described in the ASTM G39 Standard. In conventional test, the specimen is not strained plastically. In the present study the strain applied is higher than the elastic one.

The deflection is calculated as follows :

$$y = (365 / t) \cdot \epsilon_{el} + (300 / t) \cdot \epsilon_{pl}$$

This equation which relates the deflection y to the plastic and elastic strain of the outer fibers of the specimen convex surface and to the thickness t of the sample has been developed in the laboratory (21). The results have been verified recently by using finite element calculation (22).

The exposure test is performed in a 12 liter autoclave with a stainless steel lining. A precise procedure is applied for the test in pure steam and in doped steam. In the two cases, the first step is washing-up of the autoclave with demineralized water at room temperature until the resistivity of the output water is higher than 1 MΩ . cm. Afterwards, the solution is introduced and deaerated by argon bubbling. A measurement of the dissolved oxygen concentration is done as the temperature is fixed at 120°C. If this concentration is less than 10 ppb, the temperature is increased to 350°C and a gas mixture of argon and hydrogen is injected in the autoclave in order to fix the hydrogen pressure of the steam. The hydrogen partial pressure is measured using a specific cell composed of a palladium-silver tubing. The temperature is then increased up to 400°C. The total pressure of the autoclave stabilizes at 200 bar, the hydrogen partial pressure at 0,8 bar.

For the exposure in pure steam, the initial solution is bi-distilled water.

For the exposure in doped steam, the initial solution is a bi-distilled water with addition of 30 ppm of Cl⁻, SO₄²⁻ and F⁻ ion as Na salts. These two environments have been extensively used at 400°C in laboratory for comparative studies of several Ni-base alloys in accelerated tests representative of PWR primary water (23, 24). Pure steam is less aggressive than doped steam : the limit stress for crack initiation is higher and crack growth rate lower in pure steam than in doped steam. The accelerating effect is due to temperature increase and also to aggressive species addition.

RESULTS

Metallographical examinations

Wire

In cold worked and aged conditions, the structure is homogeneous with a fine grain ($G_{afnor} = 11$) and a δ phase (Ni_3Nb) inter- and intra-granular precipitation. These precipitates come from a holding at $970^\circ C$ during the material processing conditions.

sheets

The **sheet A** (cold reduction : 16 %), issued from heat 3, has a homogeneous structure with a quite large grain ($G_{afnor} : 5.5$) without δ phase precipitates

The **sheet B** (cold reduction : 20%), issued from the heat 2, has a homogeneous structure with a very fine grain ($G_{afnor} : 10$) with an inter- and intra-granular precipitation of δ phase.

Tensile test

The results of the tensile test are compiled in the table n° 3.

product	conditions	YS (MPa)	UTS (MPa)	Strain (%)	Reduction of area (%)
wire	cold worked + aged	1479	1616	13.9	38.0
		1483	1613	12.6	38.0
	970°C + aged	1103	1487	23.1	40
		1100	1480	20.9	37.1
	1040°C + aged	1114	1400	25.6	52
		1114	1416	29.6	53
sheet	970°C-1 h / air cooling	375	844	53	60
		355	830	56	61
		350	830	55	60
sheet A	cold worked 16%	880	1060	26	42
		900	1057	26	53
sheet B	cold worked 20%	963	1086	22	-

Table n°3 - Results of tensile tests

Hardness test

We have performed hardness tests on sample with different cold reductions between 0% and 25% and aged with the standard aging treatment.

We have also performed hardness tests on solution annealed samples. In this case the aging treatment is composed of one step with a temperature fixed in the range $660^\circ C / 780^\circ C$ and for a duration up to 16 hours.

Corrosion tests

In order to evaluate the resistance of alloy 718, we have also tested alloy X750 in two different thermal conditions which have been characterized in PWR primary water. The less resistant structure A of alloy X750 is obtained by a two step heat treatment ($885^\circ C$ 24 h. / $700^\circ C$ 20 h.). The most resistant structure B is obtained by a single step heat treatment (25) and has been specially developed for alignment pins in PWR ($700^\circ C$ 20 h.).

Exposure to pure steam

Four samples of each case for alloy X750 and eight samples for alloy 718 have been exposed to pure steam at $400^\circ C$ during 2000 hours. The strain of the outer fiber of the samples was fixed at 0.5%. No crack initiates in this environment.

Exposure to doped steam

The same samples were exposed to a total of 550 hours of exposure breaking down into 5 periods (50 h., 100 h., 100 h., 100 h., 200 h.) in doped steam at $400^\circ C$. An examination of the samples after each period was done by optical means (magnification 60) and the determined cracked samples were not exposed to any other periods of exposure.

Three behaviors can be distinguished as presented on the figure 1, which plots the cracked sample percentage related to the cumulative exposure time. The first alloys to crack are **alloy X750 A** and **alloy 718 annealed at 970°C**. The first cracks are observed after the first period. A second group is composed of **alloy X750 B** and **alloy 718 annealed at 1040°C**. The first cracks appeared for this group after 250 hours. The last group is composed of **cold worked and aged alloy 718** which does not crack after 550 hours. The cracks are all intergranular and very deep. Very often, the sample was entirely cracked even for the first period (50 hours) That suggests a very high CGR ($\geq 40 \mu\text{m/h}$) for all the materials and the main effect of alloy conditions on the initiation time.

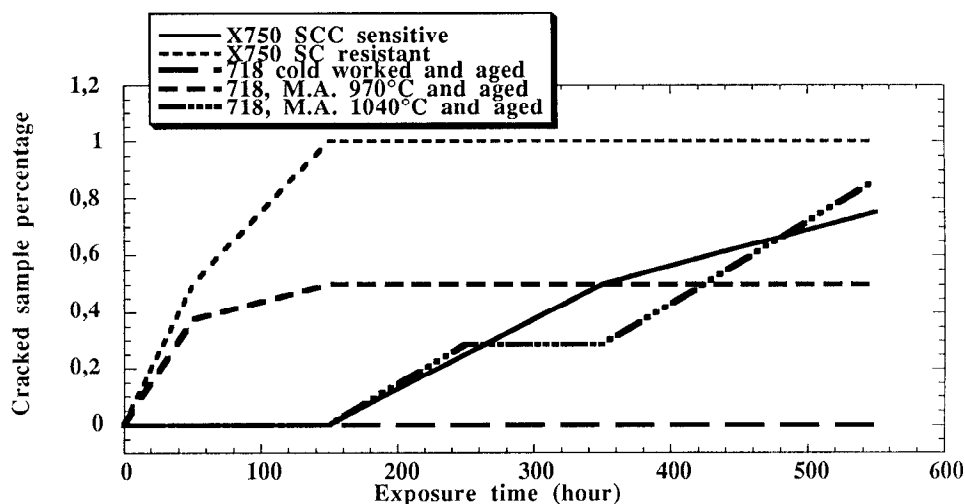


Figure n° 1 - Results of Corrosion resistance in doped steam at 400°C

DISCUSSION

Effect of cold work on the mechanical properties and on the hardening kinetics

Figure n°2 presents the evolution of the increase of hardness with cold work (as reduction of the cross section area) for the two sheets A and B before aging. These two sheets of same size present very different microstructures. As presented in the results section, the sheet A has a fine structure with δ phase and sheet B has a less fine structure without any δ phase precipitates.

We note that for these two products, the hardening obtained by cold work is identical in the range of this study. Indeed, if for sheet A, the hardness is due to the presence of δ phase precipitation, for the sheet B, the hardness is related to an effect of solid solution of Nb.

This equivalence of hardness allows us to extrapolate the comparison of the two materials in aged condition.

On figures 3 to 5, we note that the increase of hardness ΔH_v due to the aging thermal treatment depends on the holding time at a fixed temperature and on the cold reduction before aging.

However, the time does not seem to influence the shape of the curve $\Delta H_v = f(\epsilon)$ but only its position. The most interesting result is that the maximum increase of hardness is always obtained for a low cold reduction.

For cold reductions higher than 12%, and in the time and cold reduction domain of the study, the material could never reach the maximum increase of hardness. It may reach it for shorter holding times, but taking into account the high precipitation kinetics, the results would be very dependant on the heating rate.

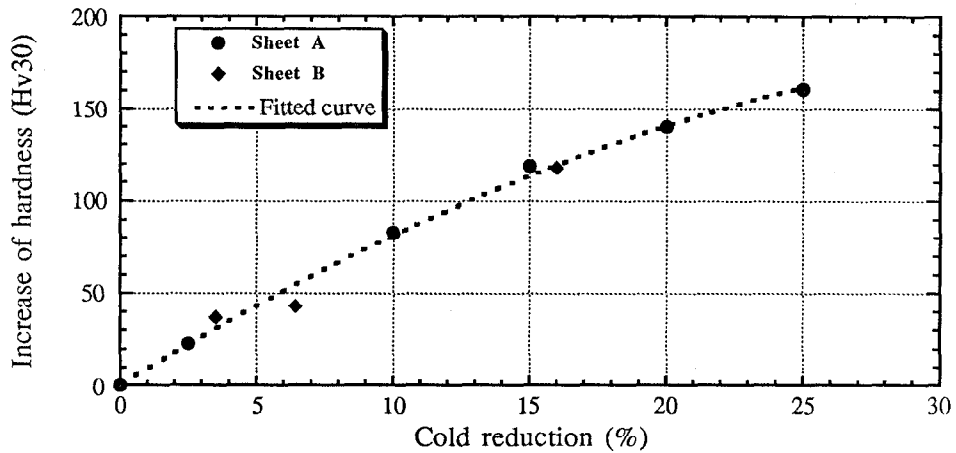


Figure n° 2 : Increase of hardness with cold reduction of annealed materials

Thus an adjustment of the aging thermal treatment is required in order to obtain the maximum increase of hardness. Figure 6 enables the determination, for a given temperature and time (in the domain of the study), of the cold reduction corresponding to the maximum increase of hardness due to the aging treatment. This maximum increase of hardness is in between 210 and 230 Hv₃₀, in the range 660°C / 780°C. This shift of 20 Hv₃₀, 10% of the average value, is due to the uncertainty of measurement.

The curves of figures 3 and 6 allow to adjust the thermal treatment of a cold worked specimen or the cold reduction of a product for a given thermal treatment, in order to take advantage of the maximum increase of hardness brought by the thermal treatment.

This approach is most efficient in the cases where the initial products have to undergo a small cold reduction (< 12%). For higher reductions, it would be appropriate to carry out the similar study at lower temperatures.

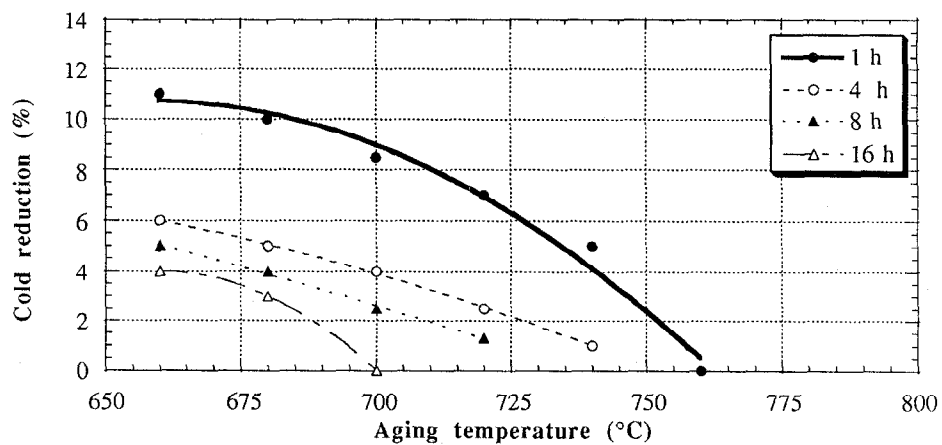


Figure n° 6 : Evolution of cold reduction corresponding to maximum increase of hardness (220 Hv₃₀) with temperature and time of aging treatment

For the highest cold reductions, and for the standard or the one-step aging thermal treatment, we note that the increase of hardness brought by the aging treatment drops. This loss of efficiency of the aging treatment can be accounted for by the combined effects of different phenomena :

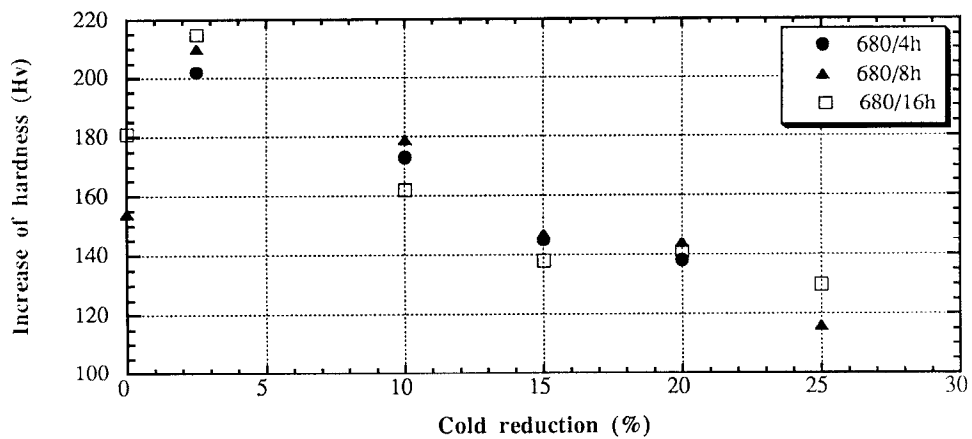


Figure n° 3 : Evolution of the increase of hardness with the cold reduction and the aging treatment

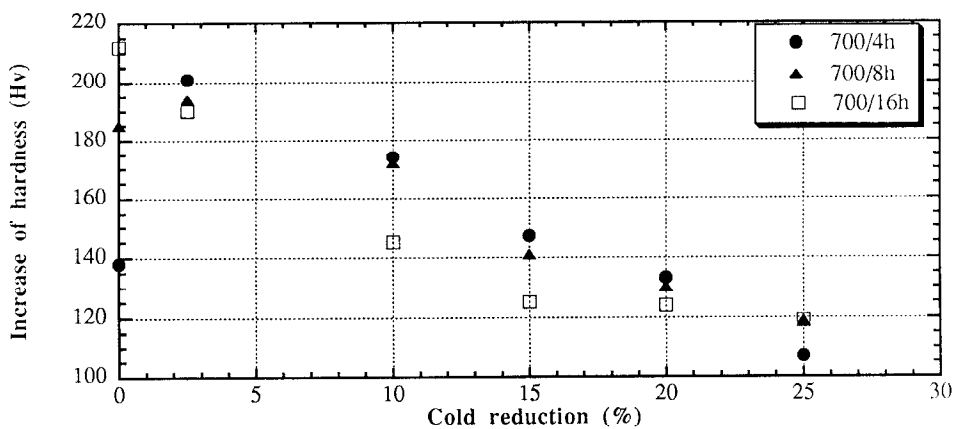


Figure n° 4 : Evolution of the increase of hardness with the cold reduction and the aging treatment

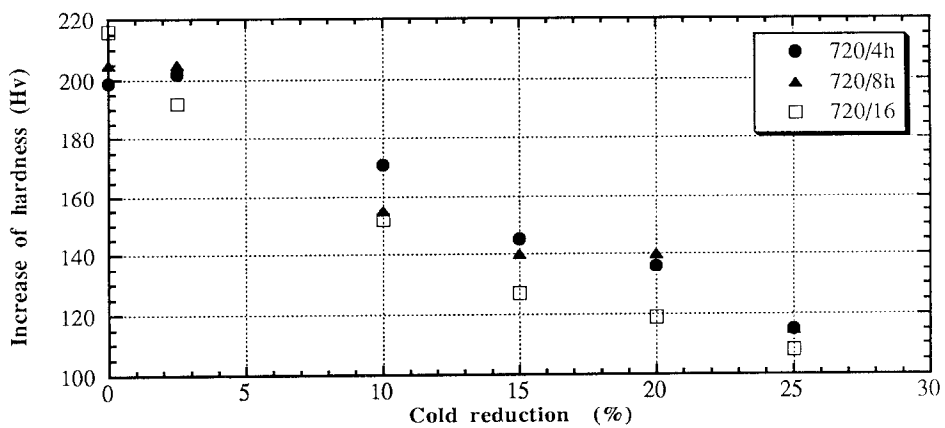


Figure n° 5 : Evolution of the increase of hardness with the cold reduction and the time of aging

- The material recovery. The latter starts at a temperature which is lower when cold reduction is higher. In fact, it is not necessary to impose a large cold reduction on the material to decrease significantly the temperature for the beginning of recovery. As a matter of fact, as long as the precipitation of γ' and specially γ'' is not initiated, the material recovery is easier. However, precipitation like recovery is promoted by the cold work but at a lesser extent, since the dislocation climb can occur at low temperature whereas the diffusion of Ti and Nb is not easy. In these conditions, we can assume that the recovery starts during the heating up of the aging thermal treatment and before the precipitation of γ' and γ'' .

- An accelerated nucleation and growth of precipitates. For a given temperature, the precipitation of γ' and γ'' will be activated by dislocations more efficiently when the cold reduction is higher (heterogeneous precipitation). The nucleation of precipitates occurs preferentially on crystallographical defects. As a consequence, the growth of the γ'' precipitates has more time to occur and their number is decreased (for a constant precipitate volume ratio). All the precipitates, redissolved during this growth, release the dislocations upon which they were pinned. These dislocations can then be removed by recovery. Two phenomena are opposed : firstly, the increase of nucleation which may produce numerous and small precipitates, and secondly, the possibility for the precipitates to grow during a longer time.

A measurement of the γ'' precipitate sizes has been carried out, for three different conditions of cold work : 0%, 15% and 25% before the standard aging thermal treatment. It is very difficult to perform in the case of high cold reduction because the precipitates have no longer a perfect disc shape. The growth of the precipitates occurs along the dislocation and their shape will be influenced by the nature of the dislocation.

e (%)	0	15	25
l (Å)	254	267	317
h (Å)	82	103	132
h/l	0.323	0.386	0.416

Table n°4 - Sizes of the precipitates

The table n°4 shows the evolution of the length and of the thickness of γ'' precipitates with the cold reduction. A large increase of the h/l ratio is noted, which indicates that, during their growth, their thickness increases more than their length. This modification of the shape of the precipitates is an proof of a loss of their coherency with the austenitic lattice. The shape of the γ'' precipitates depends on the coherency stresses produced by the misfit of the mesh parameter along the c axis, in between γ'' precipitate and the austenitic matrix ($\epsilon_{33} \approx 3.5\%$ for annealed and aged alloy 718). In the case of an overaging, there occurs for the γ'' precipitates a loss of coherency which is more sensitive along the c axis. Thus the precipitates are more spheric. Another source of the loss of efficiency of the aging treatment can be proposed. The nucleation of the precipitates on the dislocations can induce stacking faults in the dense planes of the atomic piling sequence of the γ'' precipitates (27, 28) :

.... ABCABCABCAB...
 ABCAB|ABCABC...

The sequence AB|AB corresponds crystallographically to the δ phase. So the stacking faults can be considered as a nucleus for the formation and the growth of δ phase which may expend at the expense of the γ'' precipitates during a long hold at high temperature and may cause a local overaging. Evidence of a local overaging has not been established for the studied materials.

This explanation of the evolution of the increase of hardness with the cold work can be extrapolated to the evolution of the tensile characteristics. Figure n°7 plots the increase of yield strength versus the cold reduction due to the aging with the standard thermal treatment, for the two sheets (Older results on wire from FRAMATOME (29) and results of Heacox (20)

are added). We note that the evolutions obtained for the increase of hardness and of yield strength are very similar. For very high cold reductions (> 40%), figure n°7 suggests that the yield strength may be lower after aging than before. Figure n°8 plots the increase of yield strength during the aging thermal treatment for different cold work conditions versus the yield strength in the equivalent unaged conditions. We note that the higher is the yield strength of the material before the aging treatment, the lower is the increase of the yield strength during the aging treatment. This increase tends to zero for 1680 MPa.

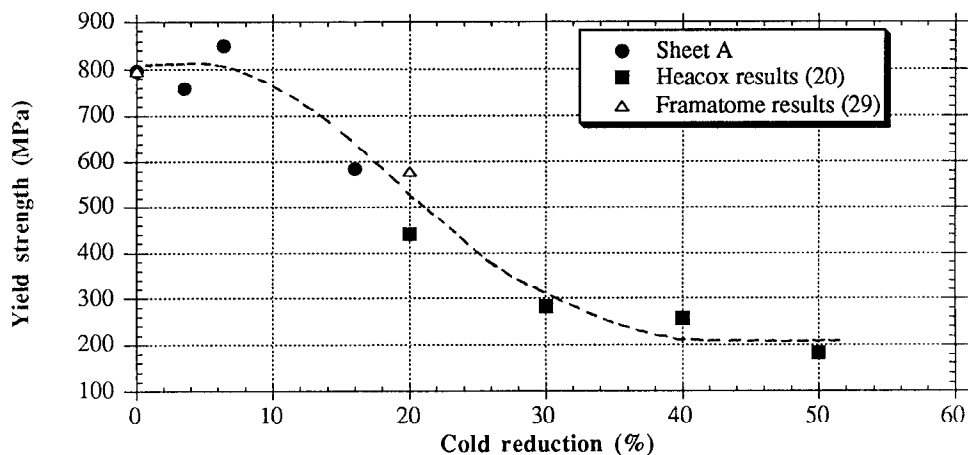


Fig n° 7 - Evolution of the increase of yield strength during the aging heat treatment with the cold reduction

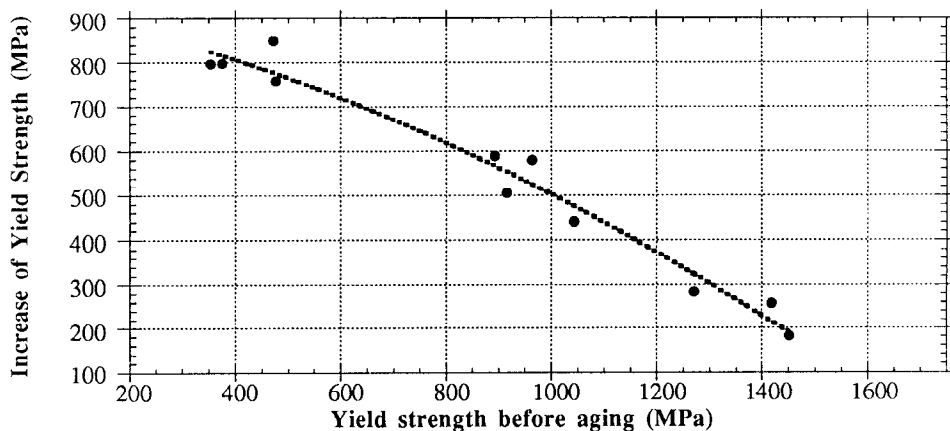


Fig n°8 - Increase of Yield strength during aging thermal treatment versus the initial yield strength

Influence of the microstructure and of the mechanical properties on the behavior in PWR primary water

Results obtained for alloy X750 are in agreement with those of the literature concerning the resistance to SCC of Ni base alloys, in PWR primary water and the influence of heat treatment (25). Thus this exposure test in doped steam at 400°C appeared as an accelerated test representative of the PWR primary coolant. Moreover, this conclusion is confirmed by one of the results of this study concerning the influence of the microstructure and more precisely of the δ phase on the resistance to SCC in PWR primary coolant. The two materials tested present very different microstructures. They have been obtained from a cold worked material (16%) by two different annealing heat treatments at 970 and 1040°C. The first one has a fine grain and a significant inter- and intra-granular precipitation of δ phase. The other has a bigger grain

without any δ phase. The most susceptible to SCC in the doped steam is the first one with the δ phase. This result is in total agreement with the usual behavior of the structures containing δ phase (19).

The influence of the cold work is more surprising. Usually, cold work is known as influencing badly the resistance to SCC. For instance, a cold reduction of 5 to 10% on alloy 600 shortens drastically the initiation time of cracks and increases by a factor 10 the crack growth rate (26). In the case of the alloy 718, a cold reduction of 10% performed before the aging heat treatment has brought to the material an improved resistance to crack initiation since no cracks are observed on sample after a cumulative exposure time of 550 hours in the doped steam at 400°C. In this study, samples are under imposed strain and for a cold worked material, this implies very high imposed stresses. At the end of the test, a measurement of the residual bending deflection has shown that the samples were always highly stressed. The absence of cracks cannot be attributed to a stress relaxation during the holding at 400°C. The presence of δ phase does not weaken the material. This could indicate that the δ phase is not directly responsible for the lesser SCC resistance of the material but that an indirect influence on the matrix is often obtained which can be suppressed by a proper control of the metallurgical and microstructural parameters. This result has to be confirmed and explained but it seems that the reason for this improvement of the resistance may be attributed to the combined effect of cold work, of the hardening precipitation and of the partial recovery of the matrix.

CONCLUSION

The main conclusions of this study are as follows :

- Very high mechanical properties can be obtained with alloy 718 by combining precipitation hardening and cold work. But the efficiency of the aging heat treatment is not obtained for any cold work. A compromise is necessary between the temperature of the heat treatment and the cold reduction. In order to obtain the most suitable adjustment, a set of curves are proposed which give the relation between the temperature, the time of the heat treatment and the cold reduction required for the maximum increase of hardness during precipitation.

- The decrease of the yield stress and of the hardness after the standard aging heat treatment, for cold reduction higher than 12% is probably due to the partial recovery of the matrix and to a loss of coherency of the γ' precipitates (more spherical precipitates)

- The exposure to doped steam at 400°C of stressed samples in alloy 718 has been found to be representative of the corrosion behavior of this alloy in the PWR primary water.

- A very good resistance in doped steam at 400°C of the cold worked alloy 718 has been obtained in spite of the presence of δ phase. High mechanical properties do not reduce systematically the resistance to the environment but the close control of the metallurgical parameters during the whole fabrication process of a component in alloy 718 can lead to very high mechanical properties and a very good resistance to the PWR primary coolant.

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