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Influence of δ phase precipitation on the stress corrosion cracking resistance of alloy 718 in PWR primary water

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ABSTRACT

The negative influence of δ phase on the intergranular stress corrosion cracking (IGSCC) resistance of alloy 718 is commonly taken for granted. In addition, δ phase formed at low temperature (about 1023 K) do not present the same characteristics than the one formed at higher temperatures (from 1173 to 1273 K). The aim of the present study is then to understand how δ phase precipitation could enhance crack initiation in alloy 718, whatever the form of δ phase is. For that purpose, several heat treatments leading to δ phase precipitation were realized on two alloy 718 heats, one sensitive to IGSCC and the second not. Specific slow strain rate tensile tests carried out on thin tensile specimens in simulated PWR primary medium at 633 K conclusively prove that δ phase has no effect on the intrinsic sensitivity to intergranular crack initiation of tested heats.

1. Introduction

Stress corrosion cracking (SCC) is a damaging mode in number of structural alloys used in pressurized water reactors (PWR) of nuclear power plants, particularly of nickel based alloys [1–5]. Even if alloy 718 is not the most commonly used structural alloy in term of mass when compared to alloy 690 constituting the vapour generator tubes, it is yet constitutive of highly stressed structures of fuel assemblies, such as springs, hold-down system screws, etc. The use of alloy 718 for such applications is both justified by its high mechanical properties and its excellent resistance to crack initiation in the very severe service conditions of the PWR. That excellent behaviour of alloy 718 is due to the formation during the aging heat treatment of coherent metastable Ni_3Nb γ'' precipitates associated with a smaller volume fraction of NiAl γ' precipitates. The sensitivity of alloy 718 to interdendritic segregations of elements with low diffusivity (e.g. niobium) formed during ingot solidification is also often reported [6–9]. During the alloy manufacturing steps, the stable Ni_3Nb δ phase could then precipitate for a wide range of temperatures and with various morphologies, quantities and distributions, depending from the local chemical composition variations. As the solubility temperature of δ phase precipitates directly depends from the local alloy niobium content, one should consider the highest temperature imposed by interdendritic segregations of niobium to fully dissolve δ phase. In the present case, that upper solvus temperature is equal to 1283 K.

Two kinds of δ phase have to be distinguished: δ phase formed in the temperature range (1173–1273 K) during the annealing heat treatment (labelled δ_{HT}) and δ phase precipitated at lower temperatures (around 1023 K) during the ageing heat treatment (denoted δ_{LT}). Whereas δ_{HT} is characterized by spheroidized coarse δ phase with platelet formation, δ_{LT} precipitates in the form of a film or continuous strings on the grain boundaries. In both cases, δ phase precipitation leads to the formation of areas denuded of γ'' hardening precipitates, called precipitate free zones (PFZ).

If considering alloy 718 SCC resistance when exposed to PWR primary environment, it is commonly taken for granted that the precipitation of δ phase enhances the intergranular SCC susceptibility of the alloy. The compilation of available PWR primary environment exposure data clearly demonstrates that the SCC propagation rate is directly dependent on the quantity of δ phase precipitated during ageing [10–12]. Indeed, the study of Sheth et al. [10] conclusively shows (considering identical ageing heat treatment) that an annealing heat treatment at 1269 K for 1 h, which leads to the precipitation of δ_{HT}, induces the same propagation rate as an annealing heat treatment at 1366 K for one hour, which gives a microstructure free of δ phase. Thus, the annealing heat treatment has little or no effect on the SCC resistance of alloy 718. Conversely, that work [10] clearly highlights that samples treated with the standard aerospace ageing cycle (993 K/8 h + 893 K/8 h) do not crack, whereas cracks are consistently observed for ageing cycles performed at a temperature greater than or equal to 1033 K (i.e. in the range of temperatures for which δ_{LT} can precipitate). Those results corroborate those obtained in PWR primary environment under irradiation by Garzarolli et al.

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[11,12]. They demonstrate that alloy 718 exhibited excellent SCC resistance when aged according to the conventional aeronautical route whereas it cracked when over-aged at 1033 K for ten hours. On the other hand, an annealing heat treatment at 1227 K (which is right in the δ_{HT} precipitation range) does not lead to cracking under neutron flux even after creep strains greater than 1%. That study is then consistent with the data obtained in autoclave test and reinforces the view that the presence of δ_{HT} phase does not represent an adverse factor for the SCC resistance of alloy, contrary to the precipitation of δ_{LT}.

However, nearly no data on SCC initiation in alloy 718 in PWR primary water is available. The first reason for this is that this alloy is known to be very resistant to SCC initiation in this environment. The second is that, up to now, there is no known SCC test capable of quantitatively accounting for this generic resistance to initiation. Recent work [13] has enabled to define, validate and model a slow strain rate (SSRT) test dedicated to the study of SCC initiation. It has necessitated the use of specific V-shaped tensile specimens. As a result, the aim of the present work is to perform a fine characterization of the influence of δ phase precipitation (δ_{HT} and δ_{LT}) on alloy 718 crack initiation behaviour in simulated PWR primary environment. Alloy 718 IGSCC resistance is then discussed in term of mechanical properties evolution induced by δ phase precipitation.

2. Materials and experimental procedures

The two heats of alloy 718 used in this study were obtained through a double melting process: vacuum induction melting plus electro slag remelting. The chemical compositions of the two tested coils (labelled heat A and heat B) are given in Table 1. In both cases, the cast ingot was hot and cold rolled down to a thickness of 0.3 mm before a solution annealing heat treatment at 1353 K during 30 s ended by air quenching. The microstructure of both studied heats is characterized by equiaxed fully recrystallized small grains (ASTM grain size number = 8-9). Primary carbides Ti(C,N) and NbC are observed too. It is worth noticing that the heat A is free of delta phase contrary to heat B (Tables 2a and 2b). Tensile specimens (Fig. 1(a)) were machined by stamping in the rolling direction of the sheet. Specimens were then heat treated in order to both have a fine and homogeneous precipitation of γ' and γ'' phases and precipitate expected δ phase (e.g. δ_{HT} or δ_{LT}) in more or less great quantity, as shown in Tables 2a and 2b. The volume fraction of δ phase was determined for each studied metallurgical state according to a former defined procedure using the software Aphelion developed by ADCIS (Caen, France) [14]. The reference state for heats A and B corresponds to specimens machined in the as-received sheet and then aged following the conventional aeronautical route: hold 993 K/8 h, cooling 328 K/h down to 893 K, hold 893 K/8 h and final furnace cooling at room temperature.

The different tested heat treatment conditions are presented in Tables 2a and 2b. δ_{HT} is formed owing to an annealing heat treatment in the temperature range (1213–1273 K) realized under high purity argon flow (removal of oxygen by using a titanium/zirconium turnings bed before the argon feed injector of the furnace). The further observed differences of SCC sensitivity are then only linked with the precipitation state of the specimens and with no other damaging phenomenon. Indeed, hydrogen has nearly no effect on alloy 718 SCC behaviour for the testing conditions used in the present work [15,16]. Specimens were then heat treated under vacuum following the standard aeronautical route. δ_{LT} effect on materials SCC sensitivity is studied owing to different ageing heat treatments.

For each heat treatment conditions, mechanical properties were determined at room temperature under laboratory air on a MTS™ electromechanical machine equipped with a 10 kN load cell. All tensile tests were performed at the constant strain rate of 10^{-3} s^{-1}. The values of ultimate tensile strength and elongation to rupture presented in Tables 2a and 2b are the average values obtained after ten tests with a good reproducibility.

IGSCC susceptibility of the different tested materials was evaluated owing to SSRT tests conducted in a static autoclave at 633 K. PWR primary environment was simulated by deionised and deaereted water in which was added 1200 ppm of B (added as boric acid) and 2 ppm of Li (added as lithium hydroxide) whereas the

Table 1
Chemical composition of studied heats of alloy 718 (wt%)

Heat	Ni	Fe	Cr	Mo	Al	Ti	Nb + Ta	Mn	Si	C
A	53.72	Bal.	18.22	3.07	0.44	1.02	5.12	0.060	0.10	0.0340
B	53.69	Bal.	18.30	3.04	0.48	1.05	5.17	0.046	0.06	0.0005

Table 2a
Mechanical properties and IGSCC susceptibility of studied heats of alloy 718 treated to precipitate δ_{HT}

Heat treatment cycle	Coil	ASTM grain size number	Room temperature mechanical properties		ISCC (%)	f_v (%)
			UTS (MPa)	E (%)		
As received + SAA, FC	A	8-9	1584	20.7	49.8	0
As received + SAA, FC	B	8-9	1367	19.2	0	1.7
1233 K/48 h + SAA, FC	A	8-9	1535	20.2	49.5	5.4
1233 K/48 h + SAA, FC	B	8-9	1377	18.8	0	5.5
1253 K/48 h + SAA, FC	A	8-9	1504	20.2	50.2	3.7
1253 K/48 h + SAA, FC	B	8-9	1347	19.1	0	3.5
1263 K/48 h + SAA, FC	A	8-9	1527	20.4	48.8	2.1
1263 K/48 h + SAA, FC	B	8-9	1348	18.7	0	1.9
1213 K/96 h + SAA, FC	A	8-9	1491	20.1	49.1	7.1
1213 K/96 h + SAA, FC	B	8-9	1336	18.9	0	6.9
1233 K/96 h + SAA, FC	A	8-9	1449	21.9	50.7	5.4
1233 K/96 h + SAA, FC	B	8-9	1321	19.3	0	5.3
1253 K/96 h + SAA, FC	A	8-9	1464	19.9	48.5	4
1253 K/96 h + SAA, FC	B	8-9	1302	19.5	0	3.7
1263 K/96 h + SAA, FC	A	8-9	1431	18.9	49.1	1.9
1263 K/96 h + SAA, FC	B	8-9	1298	19.4	0	1.8

SAA = standard aeronautical aging heat treatment, FC = furnace cooled.
UTS = ultimate tensile strength, E = elongation to rupture as determined after tensile tests in air at room temperature at a strain rate of 10^{-3} s^{-1}.
The values of I_{SCC} for each applied heat treatment cycle are the average value obtained after five SSRT tests. In all cases, the dispersion is not greater than 1%.

Table 2b
Mechanical properties and IGSCC susceptibility of studied heats of alloy 718 treated to precipitate δ_{LT}

Heat treatment cycle	Coil	ASTM grain size number	Room temperature mechanical properties		I_{SCC} (%)	f_v (%)
			UTS (MPa)	E (%)		
As received + SAA, FC	A	8-9	1584	20.7	49.8	0
As received + SAA, FC	B	8-9	1367	19.3	0	1.7
1013 K/8 h + 893 K/8 h, FC	A	8-9	1443	16.5	52.5	2.1
1013 K/8 h + 893 K/8 h, FC	B	8-9	1297	16.9	0	12.5
1033 K/8 h + 893 K/8 h, FC	A	8-9	1435	14.9	53.6	2.3
1033 K/8 h + 893 K/8 h, FC	B	8-9	1296	16.7	0	13.8
1053 K/8 h + 893 K/8 h, FC	A	8-9	1288	16.4	55.3	2.8
1053 K/8 h + 893 K/8 h, FC	B	8-9	1145	15.1	0	14.6
1053 K/16 h + 893 K/8 h, FC	A	8-9	1351	15.6	50.3	3.4
1053 K/16 h + 893 K/8 h, FC	B	8-9	1335	18.2	0	15.9
1053 K/32 h + 893 K/8 h, FC	A	8-9	1328	20.3	26.3	4.9
1053 K/32 h + 893 K/8 h, FC	B	8-9	1235	19.6	0	17.1

SAA = standard aeronautical aging heat treatment, FC = furnace cooled.
UTS = ultimate tensile strength, E = elongation to rupture as determined after tensile tests in air at room temperature at a strain rate of 10^{-3} s^{-1}.
The values of I_{SCC} for each applied heat treatment cycle are the average value obtained after five SSRT tests. In all cases, the dispersion is not greater than 1%.

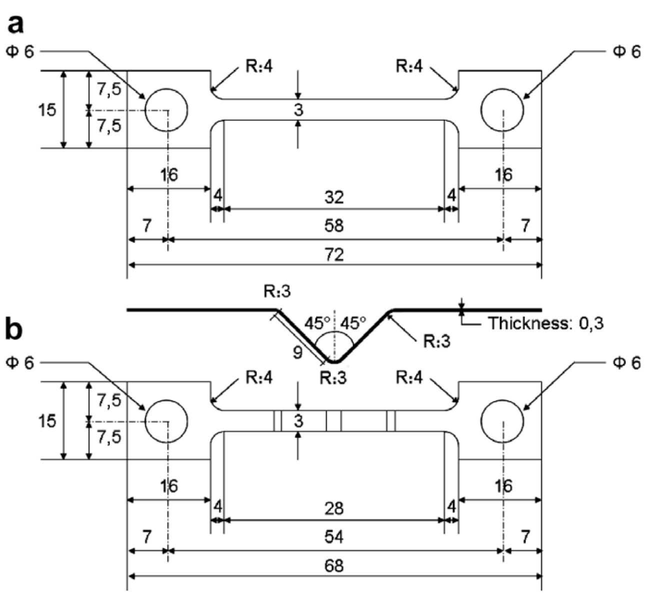

Fig. 1. Geometry of thin tensile specimens used (a) for mechanical properties characterization at room temperature and (b) for SSRT tests. Specimen dimensions are given in mm. In both cases, specimen thickness is equal to 0.3 mm.

partial pressure of hydrogen was set to 0.3 bar with a Pd–Ag membrane. It corresponds to a dissolved hydrogen content of about 35 cc/kg of water and the estimated pH at temperature is approximately equal to 7.5. As the present work aims at studying the effect(s) of δ phase on SCC initiation, specific thin tensile specimens were used (Fig. 1(b)). Those V-shaped specimens were obtained from tensile specimens presented on Fig. 1(a) by cold forming before the ageing heat treatment with a proper dye. Such geometry has two main advantages for crack initiation concerns. On the one hand, it enables to both control the strain rate and the extent of the volume at the apex of the V submitted to that imposed strain rate [13]. As a result, required loading conditions for SCC initiation are only fulfilled locally, at the apex of the V, hence the control of the localization of potential crack initiation site. In this study, all SSRT tests were performed with an imposed local strain rate of 2×10^{-8} s^{-1}. On the other hand, the arms of the V enable to be sure that, if a stress corrosion crack is formed at the apex of the V, that crack would then propagate until specimen rupture, because of the lever arms effect resulting from the V shape. To compare more easily the results of those SSRT tests, a SCC susceptibility factor (I_{SCC} – see Fig. 2) has been defined as the ratio between the cumulated length affected by intergranular brittle rupture and half of the total perimeter of the considered specimen. Because of the specific geometry of the tensile specimens used for SSRT tests, only the intrados of the apex of the V is submitted to tension (mechanical loading conditions compatible with crack initiation). As a result, only half of the total perimeter has to be taken into account in the definition of the SCC susceptibility factor. Observations of the fracture surfaces were performed on each part of the specimen after rupture by SEM on a Leo 435 VP apparatus operating either in secondary electron (SE) or in back-scattering electrons (BSE) mode.

3. Experimental results

First of all, whatever the δ phase precipitation heat treatment applied to heat A is, tested tensile specimens exhibit a mainly intergranular brittle fracture mode (Fig. 3(a)). In addition, the value of the SCC susceptibility factor remains quite constant, about half of the observed fracture surface, excepted for the longest times of heat treatment at 1053 K (Tables 2a and 2b). Indeed, for 32 h, I_{SCC} is equal to 26.3%. On the contrary, the observed fracture surfaces of heat B tensile specimens are always fully transgranular ductile (Fig. 3(b)).

To assess whether δ phase precipitation is responsible for the IGSCC susceptibility of heat A samples, the evolution of the SCC susceptibility factor has to be compared with those of mechanical properties and δ phase volume fraction (Fig. 4, Tables 2a and 2b). If considering δ_{HT} phase, a reduction of mechanical properties for both tested heats is observed when the temperature of heat treatment precipitation increases. This loss of strength is at least more than twice higher for heat A (−153 MPa for the UTS in the most severe heat treatment conditions) than for heat B (−69 MPa for the UTS in the same conditions). Besides, after comparison between SEM micrographs, the quantity of precipitated δ phase appears to decrease with increasing heat treatment temperature for both heats. Such an assessment is confirmed by the analysis of δ_{HT} volume fraction (Table 2a). For a given temperature, no major difference are observed between heat A and heat B for the two tested times (i.e. 48 and 96 h).

On the other hand, δ_{LT} phase precipitation heat treatments do not induce the same modifications of the behaviour of the tested heats (Table 2b and Fig. 4). Indeed, in both cases, the determined loss of mechanical properties is nearly the double of the one induced by δ_{HT} phase precipitation. In addition, for a same applied heat treatment, the quantity of precipitated δ_{LT} phase is far greater for heat B than for heat A (Fig. 4(c) and (d)). Indeed, the volume

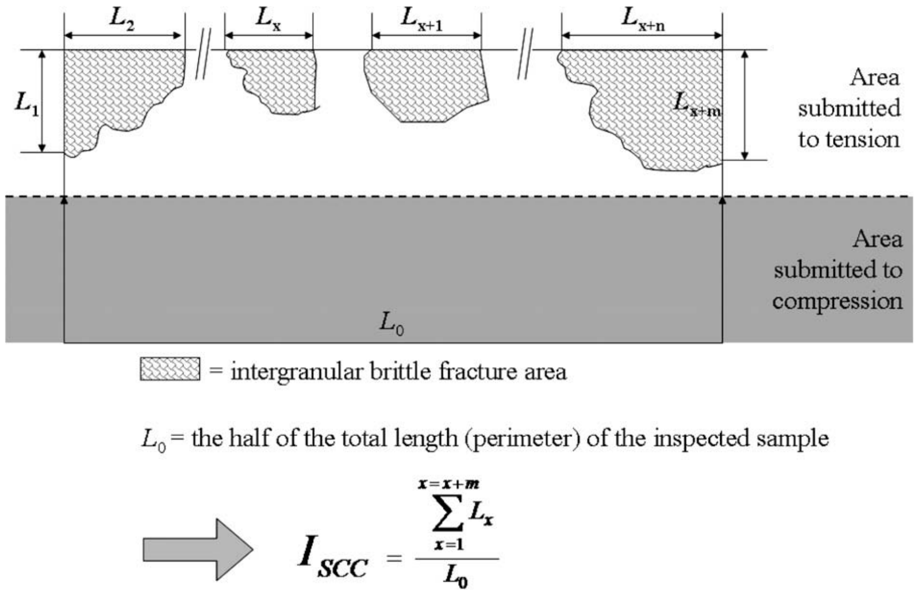

Fig. 2. Schematic sketch explaining the definition of the SCC susceptibility factor I_{SCC}.

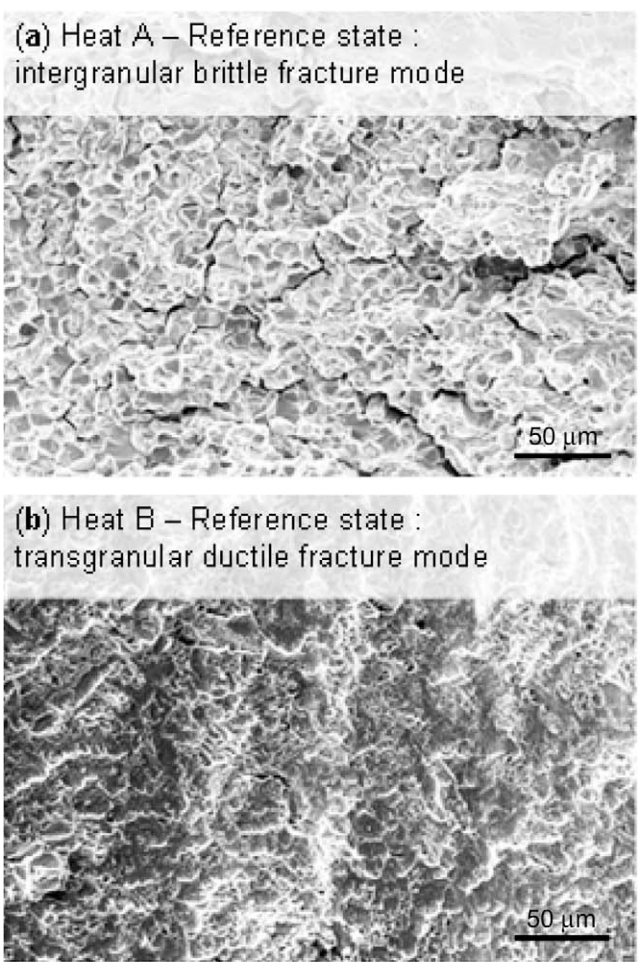

Fig. 3. SEM observation of fracture surface of V-shaped specimens tested at 633 K in PWR primary water with an imposed strain rate of $2 \times 10^{-8}\ s^{-1}$. (a) Heat A reference state, (b) heat B reference state.

fraction of precipitated δ_{LT} phase is no greater than 5% for heat A tensile specimens whereas it is always over 10% for heat B ones.

4. Discussion

For all the metallurgical states tested, a first degree analysis of experimental results collected for the heat A (Tables 2a and 2b) leads to conclude that, in worst cases (δ_{HT} phase precipitation heat treatments), its susceptibility to intergranular stress corrosion cracking is slightly influenced by the formation of the Ni_3Nb precipitates. Moreover, the lower is the temperature of heat treatment, the higher is the value of the precipitated δ phase volume fraction, hence confirming what was yet known for cast 718 alloy [14]. On the contrary, δ_{LT} phase precipitation heat treatments seem to improve the alloy behaviour for long lasting overageing treatments: for 32 h, the value for the I_{SCC} factor is equal to half of that for 16 h (Table 2b). Another striking result of the present work is that the heat B, which is unaffected by stress corrosion cracking in its reference state, never revealed an intergranular ductile fracture mode, even for very high volume fraction of δ phase (up to 17.1% after 32 h at 1053 K – see Table 2b). One should also know that, for all tested specimens, the intergranular damage which results from the corrosion process in PWR service conditions at 633 K has been characterized and is about the same for both heats, regardless of the applied δ phase precipitation heat treatment [17]. All these facts might lead to conclude that δ_{HT} phase as well as δ_{LT} phase have very little effect on the sensitivity of alloy 718 to crack initiation when exposed to PWR primary environment and, above all, that this impact is in no case detrimental to the alloy intrinsic SCC resistance.

However, the difference of I_{SCC} factor value observed between the two studied heats tested in the reference state has not been explained yet. Indeed, it is rather surprising that the heat free of delta phase is the most sensitive to IGSCC. An explanation to the environment-induced intergranular cracking of heat A could be found in the work of Wei et al. [18–21]. They proposed for nickel based superalloys (e.g. Inconel 718, Rene-95, X-750) a mechanism involving Nb in the intergranular embrittlement of those niobium containing alloys. The oxidation of both niobium-rich primary carbides NbC and intermetallic γ'' precipitates along the grain boundaries is believed to form a brittle film of niobium oxides (e.g. Nb_2O_5) which is described as a potential source for intergranular failure. Experimental evidence of such a mechanism does exist but it could not explain in the present case why heats A and B still

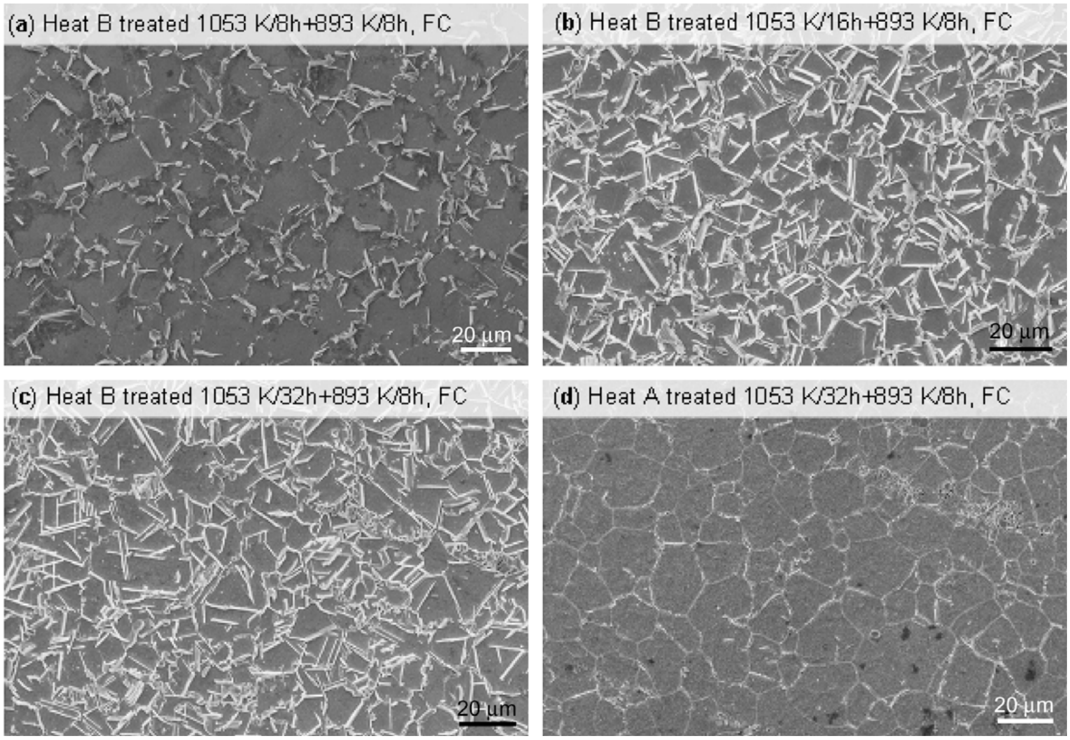

Fig. 4. SEM micrographs showing the microstructure of heat B after δ_{LT} phase precipitation heat treatment at 1053 K for (a) 8 h, (b) 16 h and (c) 32 h. Comparison of heat A (d) and heat B (c) precipitation state for the same ageing conditions (1053 K/32 h + 893 K/8 h, FC).

exhibit the same difference of susceptibility to IGSCC for the same volume fraction of precipitated δ phase or why the value of the ISCC factor for heat A is lower for long lasting overageing heat treatment.

Up to now, neither the chemical composition nor microstructural variations, including those induced by corrosion/oxidation phenomena in PWR primary water [17], have enabled to explain the experimental facts established in the present work. Consequently, one is compelled to take into account another parameter to properly analyze and explain the results of our study. Indeed, what is aimed here is to define and understand the effect(s) of δ phase precipitation on defect initiation induced by stress corrosion cracking in PWR primary medium. As a result, one must not forget that δ phase precipitation induces a lowering of the mechanical properties of alloy 718, due to the reduction of volume fraction of γ'' hardening precipitates. This matter of fact is clearly evidenced when considering the data presented in Tables 2a and 2b. All former discussed points have then to be re-analyzed with regards to the evolution of mechanical properties and strain localization. Thus, the formation of the non hardening δ precipitates has two main consequences in terms of mechanics. On the one hand, it reduces the quantity of Nb available in the alloy to form hardening γ'' precipitates. On the other hand, in the vicinity of δ precipitates, hardening precipitates depleted areas are formed.

If considering an alloy 718 sample free of delta phase, the hardening γ'' precipitates could be divided in two populations: those hardening the grain (labelled 'volume γ'' precipitates') and those involved in the hardening of the matter in the vicinity of grain boundaries (called 'interfacial γ'' precipitates'). That last family, as it contributes to the hardening of interfaces, may both enhance intergranular crack initiation and propagation. While a δ precipitation heat treatment is being done, the fraction of 'interfacial γ'' precipitates' reduces at the same rate that the volume fraction of δ phase increases. These two phenomena tend not only to modify the strain localization at grain boundaries but could also help to relieve internal stresses. Since the formation of PFZ in the vicinity of grain boundaries induces local variation of the alloy chemical composition, a local softening of interfaces occurs, hence a possible inhibiting effect on crack initiation due to the lowering of the mechanical loading imposed to grain boundaries.

Nevertheless, that mechanical analysis of the consequences of δ phase precipitation is not sufficient to explain the whole data. Thus, for nearly the same δ phase volume fraction, heat A specimen treated 1233 K/96 h exhibits a I_{SCC} factor value of 50.7% whereas this factor is equal to 26.3% for a heat A specimen treated 1053 K/32 h + 893 K/8 h. That last fact may suggest that the key mechanical parameter which controls the crack initiation behaviour of alloy 718 is the homogeneous or heterogeneous character of the deformation modes of grains. Such an assumption is consistent with what was already established about the influence of the microstructural state of precipitation on crack propagation and could explain that δ phase precipitation has nearly no influence on the intergranular crack initiation resistance of niobium-containing superalloys such as alloy 718.

5. Conclusions

Data presented in this paper confirm that δ_{HT} and δ_{LT} phase precipitation deeply affects the mechanical properties of alloy 718, hence possibly modifying the crack growth rate of a propagating defect. However, it is also clearly demonstrated that, in no case, δ phase impacts on the alloy crack initiation behaviour when exposed to PWR primary water, even for the highest precipitated volume fractions which could exist in such superalloys. In addition, for both tested heats, the formation of similar intergranular defects induced by corrosion processes is established but their critical crack initiation sites behaviour is not compulsory. All in all, the initiation of defects induced by environmental-enhanced intergranular stress corrosion phenomenon seems to be directly controlled by the deformation modes of grains in alloy 718.

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