STRESS CORROSION CRACKING OF ALLOY 718 IN

PRESSURIZED-WATER-REACTOR PRIMARY WATER

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ABSTRACT

Stress corrosion cracking tests were conducted in 360°C (680°F) pressurized-water-reactor (PWR) primary water using aerospace-grade Super 718 with heat treatment variations which altered the local grain boundary chemistry. The variation in heat treatments resulted in a range of grain boundary chemistries: from grain boundaries heavily decorated with δ phase to precipitate-free grain boundaries. Slow strain rate (SSR) tests of fatigue precracked compact specimens revealed that intergranular stress corrosion cracking (IGSCC) resistance is substantially improved by the elimination of δ phase from the grain boundaries. The highest IGSCC resistance is associated with a microstructure free of grain boundary δ phase and a matrix homogeneously strengthened by coarse γ'' and γ' precipitates.

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INTRODUCTION

Alloy 718 is the most widely used iron-nickel-base superalloy. The alloy advertises excellent strength, low cycle fatigue and creep behavior in addition to outstanding corrosion resistance in many environments. The alloy is weldable and highly fabricable. As a result of these properties alloy 718 has enjoyed extensive use in the aerospace industry and has become increasingly important to the nuclear power industry. Alloy 718 is employed in medium temperature 290 to 315°C (550 to 600°F) structural applications in the nuclear industry where high strength and excellent stress corrosion cracking resistance are important design criteria. Alloy 718 is strengthened by precipitation of γ^\prime (Ni₃ [Ti, Al]) and $\gamma^{\rm m}$ (Ni₃Nb).

The performance of precipitation-strengthened, corrosion-resistant alloys in light-water reactors is generally good, with only a few failures resulting in plant outages. These alloys are often used for bolts, springs, guide tube pins, and other structural members where high strength, relaxation resistance, and corrosion resistance are required. Failures, which are infrequent considering the number of these components in service, are attributed to fatigue, corrosion fatigue, and intergranular stress corrosion cracking. The metallurgical condition produced by thermomechanical processing greatly affects IGSCC susceptibility. In some materials (i.e. alloy X-750), changes from the original heat treatments, which were designed for aerospace applications (where high temperature mechanical properties are of prime concern) reduced the occurrence of IGSCC failures.

The Electric Power Research Institute (EPRI) is sponsoring a program to determine the optimal processing conditions and heat treatment for Super 718. Conventional 718, which is currently the form of alloy 718 most used by the nuclear industry, is subject to variable processing conditions from one forge shop to another. The melt history and fraction of scrap alloy 718 in the melt presents another uncontrolled variable. The resultant microstructure of Conventional 718 can include the presence of embrittling laves phase, carbide stringers and grain size variation from ASTM 2 to ASTM 7.

Demands arising from the jet engine industry facilitated the development of hot die forging and placed stringent requirements on Super 718 melt practices. Super 718 is hot-die forged to a grain size of approximately ASTM 8 to 10. Unlike conventional 718, laves phase, carbide stringers and duplex grain structure are minimized or eliminated. Super 718, which is fine grained and homogeneous, exhibits superior tensile and fatigue properties to Conventional 718.

The work reported here includes the physical and mechanical response of Super 718 to variations in heat treatment. It is part of a larger EPRI program subjecting the same material conditions to a spectrum of corrosion tests. The work described here includes optical, scanning and transmission electron microscopy performed at Columbia University to characterize differences in microstructure and nanostructure and their effects on IGSCC susceptibility as measured in a modified slow strain-rate (SSR) test conducted at the Babcock & Wilcox Research Center. As part of the EPRI program, these materials are also being subjected to crack initiation and crack growth rate tests in high temperature water by the Westinghouse Research and Development Center.

Experimental Procedures

Materials: Four different material conditions of Super 718 are being investigated. Results from two of these conditions will be

presented in this paper. Table I describes the heat treatments developed to obtain the desired range of grain boundary conditions. Heat Treatment #1 is the current standard jet engine heat treatment and will be referred to as the Conventional Heat Treatment (CHT). Heat Treatment #3 is a heat treatment which was developed by Idaho National Engineering Laboratory for 718 to be used in the liquid metal fast breeder reactor program. It employs a higher solution annealing temperature to dissolve δ phase, and the same precipitation strengthening treatment as the CHT. Heat Treatment #3 will be referred to as the Modified Heat Treatment (MHT).[1]

The Super 718 test material utilized in this study was obtained from a dimensionally incorrect gas turbine disc provided by the Wyman Gordon Company. The original heat of material was Teledyne Allvac Heat No. E790, which was vacuum induction melted and vacuum arc remelted (VIM/VAR). The chemistry of the Super 718 gas turbine disc is shown in Table II.

Microscopy: Photomicrographs of the modified and conventional heat treatments are shown in Figures 1(a) and (b) respectively. The CHT condition is precipitation heat treated below the δ solvus, whereas the MHT condition is precipitation heat treated above the δ solvus. Delta phase which precipitates at and pins grain boundaries is used in the forging industry to control grain size during hot working. Scanning electron microscopy (SEM) was used to document the presence or absence of delta in the CHT and MHT conditions. Course lenticular δ precipitates (orthorhombic Ni₃Nb) were observed along grain boundaries in the CHT condition as shown in Figure 2a. The absence of δ precipitates in the MHT condition is documented in Figure 2b. The MHT condition, which is precipitation heat treated above the δ solvus, results in significant grain growth (from ASTM 8 to ASTM 3). Dark field TEM micrographs reveal the coarse γ' and γ'' morphology of the MHT condition as compared with the CHT condition (Figures 3a and 3b).

To reveal the general microstructure, samples were immersion etched in 92 ml hydrochloric acid with 3 ml nitric acid, and then rinsed in 90 ml nitric acid with 45 drops of hydroflouric acid (Figures 1a and 1b). SEM samples were electropolished in a 5 pct HC1/5 pct HC10 $_4$ /ethanol solution and etched with Kallings waterless reagent (Figures 2a and 2b). The transmission electron microscopy (TEM) specimens were jet polished at -20C (-4.0°F) in a 10 pct HC10 $_4$ /30 pct n-Butanol/ethanol solution (Figures 3a and 3b).

<u>Tension Tests</u>: Round bar tension tests (2 in. gage length, 0.25 in. gage diameter) were performed in air at room temperature and at 360° C (680°F) as per ASTM E-8.

Stress Corrosion Cracking Tests: SSR tests were performed in 360°C (680°F) in flowing PWR primary water to determine the relative susceptibility of the material conditions to IGSCC. Specifications for the test environment were:

Dissolved Oxygen Solved Oxygen Solved Oxygen Solved Oxygen Solved Ppm 5700 ppm \pm 500 ppm Lithium 2 ppm (LiOH) (6.9 ppm) pH 6.5 (\pm 0.5) Conductivity <20 μ S/cm Dissolved Hydrogen Chloride <0.1 ppm <0.1 ppm

The SSR test technique employed is a modified version of the conventional slow strain-rate test. The modified SSR test technique can yield significantly more data with respect to crack initiation and crack propagation as compared to the conventional SSR test. Application of this technique to 4340 stainless steel and to 2024 T351 aluminum are described elsewhere.[2,3]

The SSR tests were conducted on a screw-driven creep/relaxation machine interfaced to an 18-bit waveform generator. The SSR specimen geometry is a 1/2T compact specimen as per ASTM E399. Crack extension in the SSR specimen is measured throughout the entire SSR test with an alternating D.C. potential drop technique which is described elsewhere.[4] The resolution of this technique is approximately ± 0.0005 in. The extension rate was 5.0 x 10^{-8} in/s. Load, displacement, and crack length were also recorded by the computer at intervals throughout the test.

RESULTS AND DISCUSSION

The objective of this investigation is to identify and quantify the role of δ phase in intergranular stress corrosion cracking for alloy 718. Super 718 material in the CHT and MHT conditions was tested with a modified SSR testing technique. As shown in Figure 2a, Super 718 in the CHT condition has grain boundaries heavily decorated with δ phase, while the MHT condition results in little or no δ phase at the grain boundaries. The results of the SSR tests for these two materials are summarized in Table III. The data in Table III was extracted from the graphs shown in Figures 4 and 5.

SSR test results for the CHT condition, which has δ phase decorating the grain boundaries, coupled with the fractographic analysis suggest that, at a stress intensity of approximately 40 Ksi \sqrt{i} n., the crack begins propagating intergranularly. The crack converts to a ductile tearing mode at approximately 50 Ksi \sqrt{i} n. Due to noise in the potential drop system, exact crack growth rate data are not available for this experiment. However, the initial and final crack lengths as measured by potential drop agree with crack lengths measured on the fracture surface. The average crack growth rate for the CHT material, calculated using the initial and final crack lengths, is 1.4 x 10^{-4} in/h.

SSR test results for the MHT condition, which does not have δ phase present at the grain boundaries, do not show any crack growth until a stress intensity of approximately 40 Ksi√in. The crack propagation mode is primarily transgranular. A few localized spots along the crack front revealed some intergranular cracking (<5% of cracked area). The average crack growth rate for the MHT material is 6 x 10^{-5} in/hr. This is significantly lower than the average crack growth rate for the CHT material. Improvements in stability of the potential drop system will provide crack length data of sufficient accuracy for development of crack growth rate versus stress intensity plots. The sudden drop in stress intensity factor, K, was due to test shutdown and restart during which an electrical load offset occurred. The large perturbation in load may have resulted with increased crack growth rate. The highest intergranular stress corrosion cracking resistance is associated with the microstructure free of $\delta\ phase.$ The increased stress corrosion cracking resistance exhibited by the MHT condition may be due to grain boundary homogeneity. Delta phase is high in niobium and chemically different from the matrix. Delta precipitates may act as local cathodes, causing the neighboring anodic matrix material to be attacked by the environment.

Tensile results for MHT and CHT Super 718 are compared with the current EPRI specification for alloy X-750 in the HTH (CIB) condition,[5] a commonly used bolting heat treatment. Super 718 in the MHT condition advertises a thirty percent increase in yield strength as compared to X-750 in the HTH condition. The MHT condition has approximately seventy-five percent of the room temperature and 360°C (680°F) yield strength of the CHT condition. The coarse grain size coupled with the increased average γ' and γ'' particle diameter in the MHT condition are primarily responsible for this loss in strength. Previous studies found that the MHT condition had approximately ninety to ninety-five percent of the room temperature and high temperature yield strength of the CHT condition.[2-4] This discrepancy may be attributed to the different thermomechanical processing history of the materials investigated.

Several previous studies of the fatigue crack propagation (FCP) behavior of the CHT and MHT conditions found the latter to have slightly lower FCP rates under continuous cycling.[6] Furthermore, the fracture toughness measured as $J_{\rm IC}$ of alloy 718 in the MHT condition is superior to the toughness of the CHT condition.[7] The increased fracture toughness of heat treatment 3 was attributed to the dissolution of δ precipitates during the high temperature solution anneal.

SUMMARY AND CONCLUSIONS

Slow strain rate tests were conducted at 360°C (680°F) in PWR primary water on two specimens of Super 718 which were heat treated to obtain different grain boundary morphologies. The conventional jet engine heat treatment (CHT) resulted in grain boundaries heavily decorated with δ phase. The modified heat treatment (MHT) resulted in grain boundaries free of δ phase precipitates. The results from the SSR tests are summarized:

- 1) Intergranular stress corrosion cracking resistance is highest for the microstructure which does not have δ phase present at the grain boundaries (Modified Heat Treatment).
- 2) The crack propagation rate is lowest for the microstructure which does not have δ phase present at the grain boundaries (Modified Heat Treatment).
- 3) The higher solution annealing temperature employed for the MHT condition dissolves the lenticular δ precipitates. The absence of the δ particles at the grain boundaries may increase the elemental homogeneity at the grain boundaries and enhance the stress corrosion cracking resistance.
- Super 718 in the MHT condition has a thirty percent increase in yield strength as compared to alloy X-750 in the HTH condition.

ACKNOWLEDGEMENTS

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REFERENCES

- [1] G. R. Smolik and G. E. Korth, Report TREE-1253, EG&G Idaho Inc. 1978 (Available from NTIS).
- [2] R. A. Mayville, T. J. Warren and P. D. Hilton, Trans ASME, Vol. 109, July 1987, p. 188-193.
- [3] K. H. Schwalbe, private communication.
- [4] K. H. Schwalbe and Dieter Hellman, J. Testing and Evaluation, Vol. 9, No. 3, May 1981, pp. 218-221.
- [5] A. A. Stein and M. S. Gennaro, Material Specification for Alloy X-750 in LWR Internal Components, EPRI NP-6202, Jan. 1989.
- [6] L. A. James, Eng. Fract. Mech., Vol 25, No. 3, 1986, p 305-314.
- [7] W. V. Mills, Trans. AIME, Vol. 102, Jan 1980, p 118-126.

Table I: Heat Treatments for Alloy 718 (wt %)

Conventional Heat Treatment	Cor	nventi	onal	Heat	Trea	tment
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Solution annealed at 1093°C (2000°F) for 1 hour for 1 hour Furnace cooled at 55°C (130°F) + 28°C (82°F)/hour to 718°C (1325°F)

Aged at 718°C (1325°F) for 4 hours Furnace cooled at 55°C (130°F) + 28°C (82°F)/hour to 621°C (1150°F)

Aged at 621°C (1150°F) for 16 hours Air cooled to room temperature

Modified Heat Treatment

Solution annealed at 1093°C (2000°F) for for 1 hour Furnace cooled at 55°C (130°F) + 28°C (82°F)/hour to 718°C (1325°F)

Aged at 718°C (1325°F) for 4 hours Furnace cooled at 55°C (130°F) + 28°C (82°F)/hour to 621°C (1150°F)

Aged at 621°C (1150°F) for 16 hours Air cooled to room temperature

Table II: Chemistry for Super 718 Material

ri Al Nb Ni Fe Mo Cr C B Si Co Mn Ta S S Cu

1.04 0.46 5.23 bal. 19.50 3.11 18.30 0.035 0.004 0.059 0.24 0.16 0.051 <0.001 0.015 0.050

Table III: Slow Strain Rate Test Results for MHT and CHT Super 718 Conditions

Condition	Extension Rate (in/sec)	Kinitial (Ksivin)	Kcrack initiation (KSivin)	$\frac{{\tt Kfinal}}{({\tt Ksivin})}$	t _{Failure}	Failure Mode	Grain Bour Condit:
CHT	5.10-8	40	4 0	100	220	IGSCC	Delta phas present.
мнт	5.10-8	40	4 0	130	470	TGSCC	No delta j present.

Table IV: Tensile Data for Super 718 and Tensile Specification for Alloy $x-750\ \text{HTH}$

	0.2% Yield	Ultimate	E 1	RA
Condition	Ksi	<u>Ksi</u>	%	<u>*</u>
Super 718 CHT	173	207	19.2	41.1
Super 718 MHT	130	185	23.0	39.4
X-750 HTH (CIB)	100	160	20.0 min.	25.0 min.

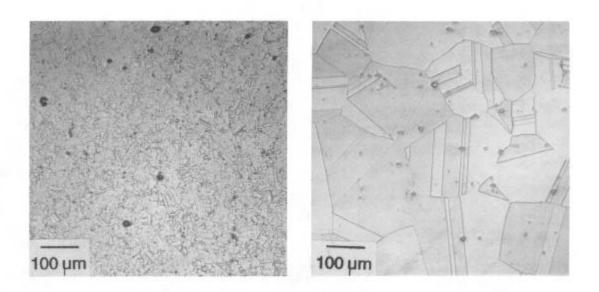


Figure 1- Optical micrographs of Super 718 in the a) CHT condition and b) MHT condition.

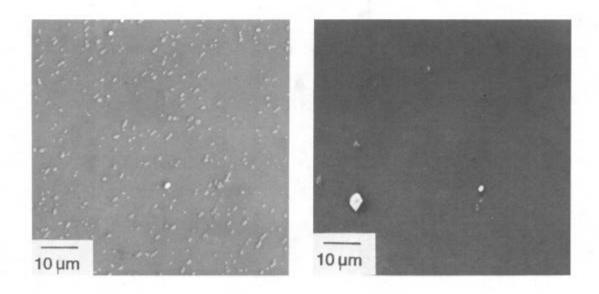


Figure 2- SEM micrographs revealing the presence of a) carbides and intergranular delta phase and b) carbides but no delta phase.

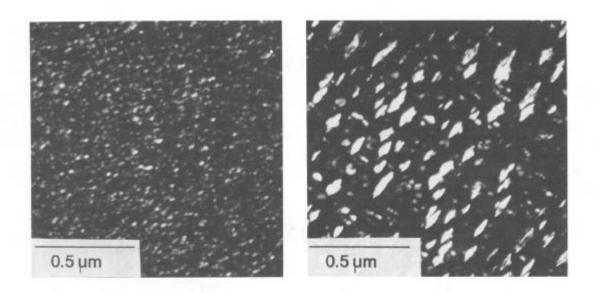


Figure 3- Dark field TEM revealing the δ ' and δ " precipitate morphology for a) Super 718 in the CHT condition and b) Super 718 in the MHT condition.

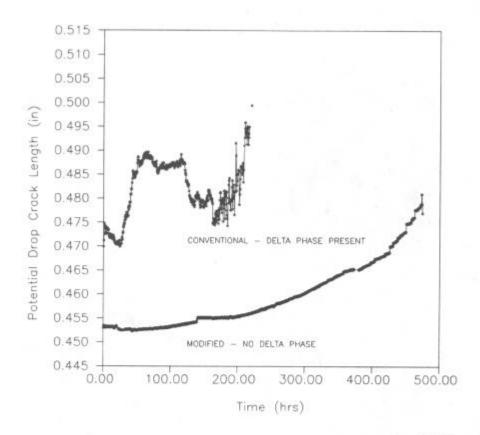


Figure 4- Potential drop crack length versus time for CHT and MHT Super 718.

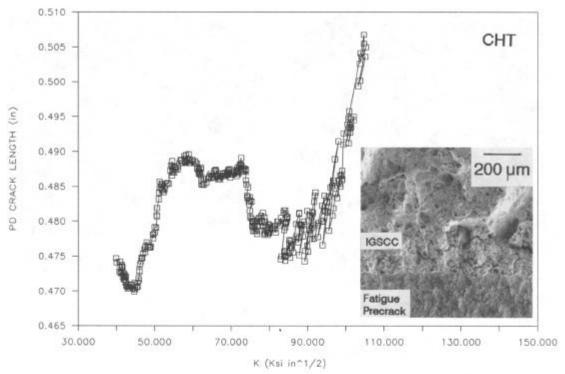


Figure 5- Potential drop crack length versus stress intensity factor, K, for CHT Super 718 and a SEM micrograph of the fracture surface.

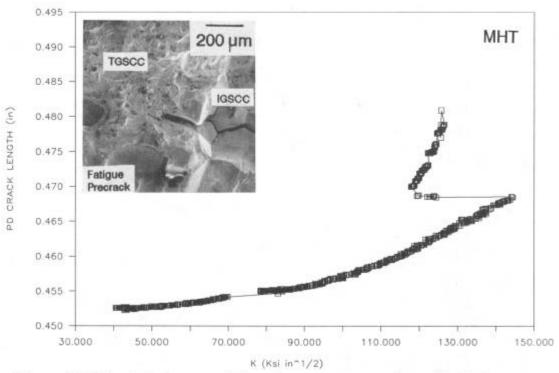


Figure 6- Potential drop crack length versus stress intensity factor, K, for MHT Super 718 and a SEM micrograph of the fracture surface.

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Table I

Conventional Heat Treatment

Solution annealed at 996°C (1825°F) for 1 hour. Air Cooled to room temperature Aged at 718°C (1325°F) for 8 hours Furnace cooled at 55°C (130°F) \pm 28°C (82°F)/hour to 621°C (1150°F) Aged at 621°C (1150°F) for a total aging time of 18 hours Air cooled to room temperature

Improving Thermal Stability of Alloy 718
Via Small Modifications in Composition
by
Encai Guo, Fengquin Xu and E.A. Loria
Page 567

Introduction, Page 568, Paragraph 2, lines 4-10, Underlined:

In a promising redesign effort, Collier and Tien^{2,3} have systematically varied the content of the three critical elements, Al, Ti and Nb. They have extended Cozar and Pineau's work¹ by modifying the alloy in the direction of a higher (Al+Ti)/Nb (0.90) ratio. In addition, they have employed high Al/Ti (1.60-1.75) ratios, coupled with 3.4 or 3.6 at pct Nb, and they were able to reduce the amount of overaging of the γ ^w particles and also to increase the amount of γ ' in order to stabilize mechanical properties. This preliminary study supplements the results obtained by Collier and Tien etc...

Results, Page 572, End of Paragraph 1, Line 5, Underlined:

composition is properly balanced towards higher (Al+Ti)/Nb and Al/Ti ratios, as shown in the initial work by Collier and Tien. 3