ALLOY 718 - ALLOY OPTIMIZATION FOR APPLICATIONS IN

OIL AND GAS PRODUCTION

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Abstract

Alloy 718 is extensively used by the oil and gas industry for Critical Service applications involving both wellhead components and downhole auxiliary equipment for corrosive/sour service. This study addresses the metallurgy and precipitation behavior of this alloy and their effects on fracture resistance. The use of heat treatment and microstructural control to tailor the alloy to the specific needs of oil and gas production applications, in contrast to aerospace applications is highlighted.

Superalloy 718—Metallurgy and Applications
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The Minerals, Metals & Materials Society, 1989

Introduction

In the last ten years, Alloy 718 has become the preferred material for manufacture of wellhead components, auxiliary and downhole tools for oil and gas wells containing corrosive and crack sensitive species such as CO_2, chlorides and H_2S. For many such applications, the alloy now replaces other corrosion resistant alloys (CRA) candidates such as K-monel, 17-4 pH and Inconel X750. This increased interest stemmed primarily from various factors, namely: (a) reported lab and field failures of production equipment made from the above CRAs(1,2), (b) ability to fabricate heavy wall and/or complex shaped components and still achieve a desired combination of strength, toughness, corrosion and environmental cracking resistance, and (c) discovery that slight modifications in composition and heat treatment can be used to tailor the alloy to the needs of specific environmental applications.

The objectives of this paper are to report typical applications of Alloy 718 in oil and gas production, and the special requirements imposed by its use in deep high pressure sour gas wells. The relative characteristics of underage, peak age, and overage heat treatments of Alloy 718 provides the basis for preferred heat treatments which optimize desired properties. The factors which affect fracture resistance are highlighted and the criteria for toughness requirements of Alloy 718 in critical applications are presented.

Application Requirements

Table I shows the typical components commonly made of Alloy 718. In general, the strength requirements fall into two classes. For wellhead equipments such as valve trims and valve bodies for American Petroleum Institute's Specification 6A PSL4 service, the minimum yield strength is 75 ksi. For fasteners, downhole and auxiliary equipments designed to connect to threaded members such as high strength tubing, and/or to contain high internal pressures, typical minimum strength is 120 ksi. In all cases involving exposure to H_2S, required compliance with National Association of Corrosion Engineer's Standard (NACE) MR-01-75 limits the maximum acceptable hardness to HRC 40.

Table I. Typical Applications of Alloy 718 in Oil and Gas Production

	Typical Minimum Yield Strength, ksi	Maximum Hardness, HRC
Subsurface Safety Valves	120	40
Packers	120	40
Hangers	75-120	40
Valve Gates, Seats, and Stems	75	40
Fire Safe Valves	75	40
Blowout Preventers	75	40
Side Pocket Mandrels	75	40
Fasteners	120	40

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Auxiliary components such as tubing hangers, packers, and subsurface safety valve body components, are considered particularly critical since they can be highly stressed with tensile loads up to 90,000 psi, and with internal pressures of 15,000 psi, while being exposed to corrosive well fluid containing H_2S, CO_2, and aqueous chlorides. Typical service temperatures can range from ambient during acidizing treatments to 350°F at normal production. Failure of such components can result in substantial monetary risks, and release of deadly toxic gases such as H_2S. Thus such concerns demand special material properties with tight process controls in manufacturing of Alloy 718 components.

For application in deep sour gas wells, the critical requirements therefore are (1) resistance to general and localized corrosion, (2) resistance to environmentally induced cracking for both sulfide stress cracking and anodic stress corrosion cracking, based on a maximum hardness of HRC 40, (3) high strength typically above 120 ksi for both tubing hangers, safety valves and packer components which must be compatible with the strength and thread design of the production tubing, and (4) fracture toughness. The goal of the toughness optimization is to ensure that a leak-before-break type of failure is achieved for pressure containment boundary components, or as a minimum, a catastrophic brittle fracture is precluded. A further consideration is that the metallurgical factors which optimize toughness at a given strength also promote corrosion and cracking resistance. These factors include specific controls on alloy composition, melting and refining practices, degree and uniformity of hot working, and heat treatment.

Metallurgical Considerations

Compositional and Microstructural Effects

The role of the various alloying elements as well as the microstructural phases in Alloy 718 have been extensively studied(3). These microstructural phases include intermetallic compounds such as γ'-body centered tetragonal structure [$Ni_3(TiAlNb)$], γ''-orthorhombic structure (Ni_3Nb), and Laves phase [$Fe_2(Ti, Nb)$]. By controlling the time and temperature at which the alloy is exposed, the amount of each phase or a combination of phases which form can be varied to meet specific requirements. The predominant hardening phase in Alloy 718 is γ'', though some γ' is also present. Carbide phases such as MC, M_6C, Cr_7C_3, and $Cr_{23}C_6$ where M refers to metallic elements like niobium or titanium can also form along with the intermetallic phases, and can affect the attainment of the desired properties. In addition, depending on oxygen and nitrogen levels in the original melt, some dirt in the form of nitride and oxide phases can be present, but to a lesser extent in vacuum processed melts. The mechanical properties as well as the corrosion resistance of Alloy 718 thus depend on the relative amount of these phases as well as their morphology (spherical, angular, film, or needle-like) and location (within grains or at grain boundaries) in the microstructure. The phase volume fractions, morphology and distribution are in turn determined by such factors as alloy composition, melt practice (air melt or vacuum melt), refining practice (vacuum refined or electroslag refined), degree of wrought structure achieved by hot working, and finally, heat treatment. Thus Alloy 718 composition used in the current application is limited to lower carbon grades (0.045% max) and higher minimum levels on Ti, Al and Nb (0.80%, 0.40%, 4.87%, respectively) compared to the aerospace material specifications AMS 5596. Also the maximum levels of the metalloid elements such as P, S, Pb, Se, and Bi were lowered, since these are known to affect toughness and environmental cracking resistance due to their influence on surface activity and grain boundary properties.

Heat Treat Response and Strengthening Behavior

Annealing Behavior. Tensile strength in Alloy 718 in the solution annealed and aged condition is controlled primarily by γ'' and γ' phases, and to a lesser degree by the carbides, nitrides, and the matrix solid solution. An annealing treatment is used prior to aging in order to: (1) dissolve various intermetallic phases in the wrought structure, (2) refine the microstructure by recrystallizing new and uniform grains, and (3) provide homogeneity in the alloy so that properties developed by subsequent aging treatment are less dependent on prior thermomechanical processing history of the alloy. Such annealing effects can be assessed by an annealing response curve which shows the effect of annealing temperature on strength (hardness).

Figure 1 shows typical anneal curves for Alloy 718. They show that as the annealing temperature increased, the hardness decreased. The decrease in hardness also depends on the rate of cooling from the annealing temperature. The hardness of the as-annealed product increases in the slow cooled condition versus water quenched condition. This effect is attributed to the reprecipitation of grain boundary secondary carbides such as $M_{23}C_6$ as the alloy is slow cooled through 1550 to 1650°F. This effect is particularly significant in mill annealed products where variability in properties have been observed. The microstructural changes associated with annealing are shown in Figure 2. It shows the mixed structure associated with low annealing temperature (<1650°F), and the coarse grain structure associated with high annealing temperature (>1950°F). An annealing temperature in the range 1825 to 1925°F provides a more homogeneous structure by eliminating the effects of prior processing history which can influence aging response. Low annealing temperatures close to 1700°F are not sufficient to wipe out the effects of prior processing history such as duplex grain structure, precipitation of deleterious phases, and chemical inhomogeneity. However, the fine grain structure associated with low annealing temperature results in increased strengthening. As a compromise between sufficient chemical and microstructural homogeneity versus strength and toughness, an annealing temperature in the range of 1825°F to 1925°F is often used.

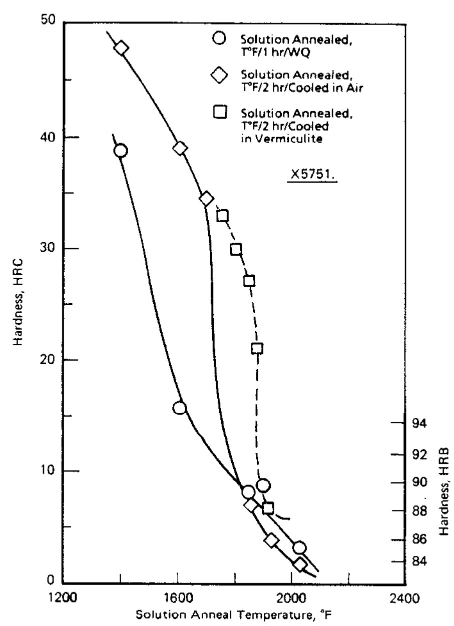

Figure 1. Softening Behavior of Alloy 718 Valve Stem Materials

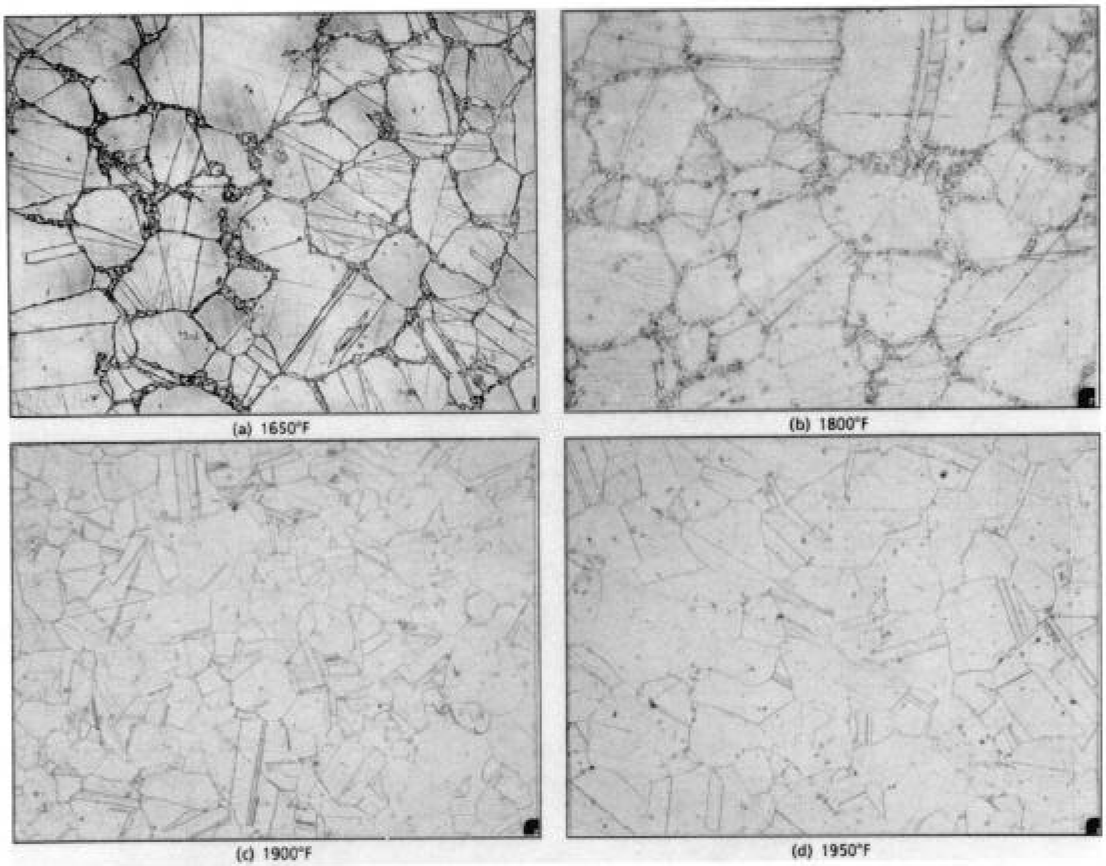

Figure 2. Microstructural Changes Associated with Annealing Treatment in Alloy 718
Annealed: Temperature °F/1hr/Water Quench; *Magnification:* 100X

Aging Behavior. For high strength or optimum creep rupture life, the common aerospace heat treatments involve 1950°F or 1725°F anneal treatments, respectively, followed by a double aging cycle. However, such heat treatments were not designed to provide high fracture toughness, and stress corrosion cracking resistance, both of which are major objectives for application to deep sour gas production.

Figure 3 shows the effect of annealing treatment on the age hardening response of Alloy 718. The data shows three conditions representing underaging (1300°F), peak aging (1400°F) and overaging (1500°F). It is evident that for a given prior hot (cold) working history, the age hardening response in the underage or overage conditions is directly affected by the anneal treatment. Increasing the anneal temperature results in softer as-aged material in the underage or overage conditions, whereas, at or near the peak aged condition, the anneal temperature has no significant influence on strength of as-aged material. The dependence on anneal temperature perhaps explains the variabilities in strength observed in underaged annealed bars, where the production furnace temperatures can vary by up to 200°F during annealing. Thus strength uniformity can be better assured by annealing between 1825 to 1925°F, followed by single aging at 1450°F ± 25°F. The proposed aging temperature range provides the desired strength and hardness requirements, without the need for the second aging cycle.

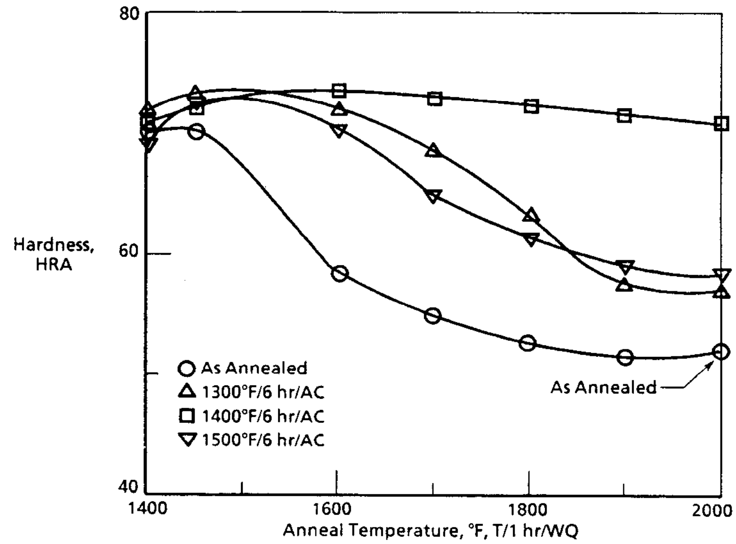

Figure 3. Effect of Anneal Treatment on Age Hardening Response in Alloy 718

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A comparison of single aging heat treatments with the "conventional" heat treatments for Alloy 718 is shown in Table II. Notice that the double aging treatments result in higher strength but reduced fracture toughness. Also the susceptibility to stress corrosion cracking is expected to be higher due to the increased hardness above HRC 40. The dependence of fracture resistance on heat treatments is evaluated in the next section.

Table II. Effect of Heat Treatment on Typical Room Temperature Properties of Alloy 718

Heat Treatment	Tensile Strength, psi	Yield Strength (0.2% Offset), psi	Elongation	% RA	Hardness, HRC	Fracture Toughness J_{IC}, psi-in.
Solution Anneal: 1875°F/1 hr/WQ Age: 1450°F/6-8 hr/AC	174	124	28	51	35	1908
Solution Anneal: 1925°F/1 hr/WQ Age: 1400°F/6 hr/AC	175	124	27	42	38	1631
Solution Anneal: 1750°F/2 hr/WQ or AC Age: 1325°F/8 hr/Cool 100°F/hr to 1150°F/8 hr/AC	193	164	23	48	42	572
Solution Anneal: 1925°F/1 hr/AC Age: 1400°F/6 hr/FC 100°F/hr to 1200°F/8 hr/AC	205	182	17	41	44	480
Solution Anneal: 1950°F/1 hr/AC Age: 1400°F/10 hr/FC 100°F/hr to 1200°F/8 hr/AC	190	161	19	NR	40	546

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Effects of Heat Treatment on Fracture Resistance of Alloy 718

To assess the effects of heat treatment on fracture resistance, Charpy impact toughness and fracture toughness based on J-integral and equivalent energy techniques were used. Impact toughness measurements comply with ASTM E23. Elastic plastic fracture toughness tests were based on methods of ASTM E813 for J_{IC} and $K_{IC}(J)$ toughness, and on ASTM E992 for K_{EE} toughness. Single specimen unloading compliance method was used so that both J_{IC} and K_{EE} were obtained from a single specimen test. Figure 4 shows a typical load-displacement record for Alloy 718. Figure 5 shows the associated crack growth J-resistance curve. In most cases fracture initiation occurred near the maximum load, and the K_{EE} toughness was close to the calculated $K_{IC}(J)$ toughness.

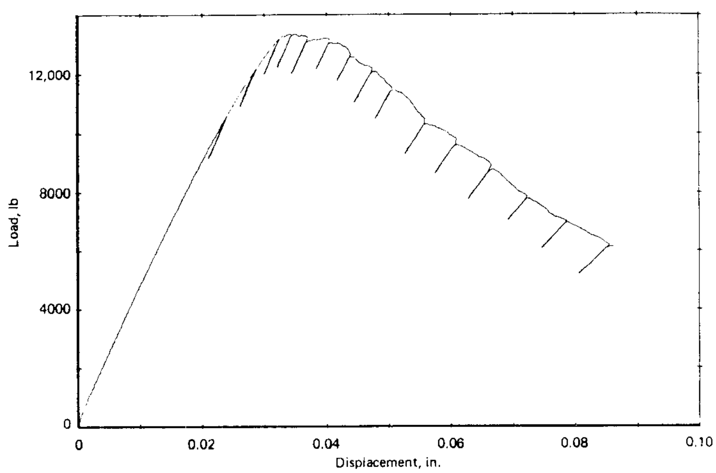

Figure 4. Computerized Load-Displacement Record for K_{EE} and J-Integral Fracture Analysis of Alloy 718 by Unloading Compliance Technique
Note the absence of a linear elastic behavior commonly observed in low toughness (brittle) materials.

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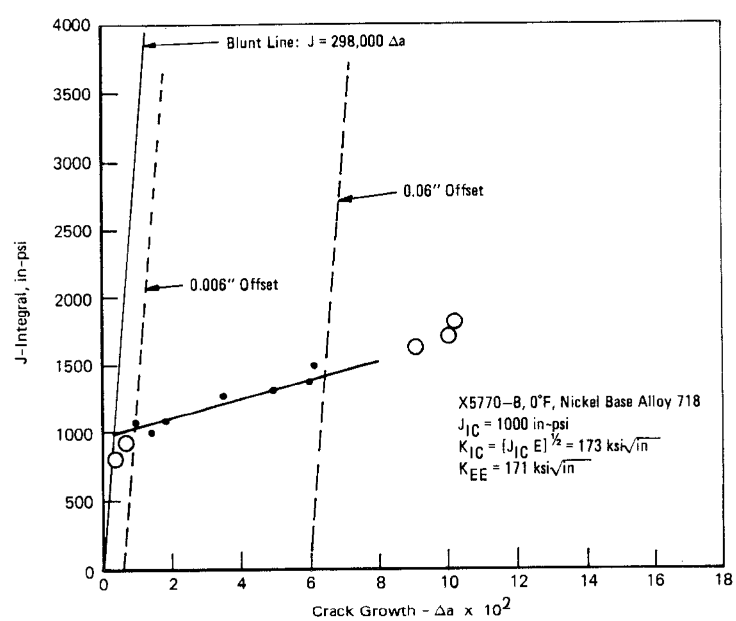

Figure 5. J-Integral Crack Growth Resistance Curve Developed for Nickel Base Alloy 718 by Unloading Compliance Technique

For the heat treat evaluation, sections of Alloy 718 billet or forging stock were heat treated in accordance to a test matrix. Compact tension and/or Charpy specimens were prepared from the samples and tested according to the above techniques. Figure 6 shows the effect of annealing temperature on the equivalent energy toughness. Notice that high anneal temperature $\geq$ 1925°F resulted in lower toughness.

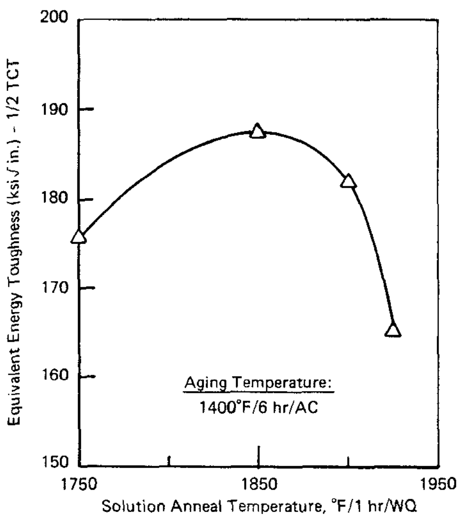

Figure 6. Effect of Annealing Temperature on Toughness of Alloy 718

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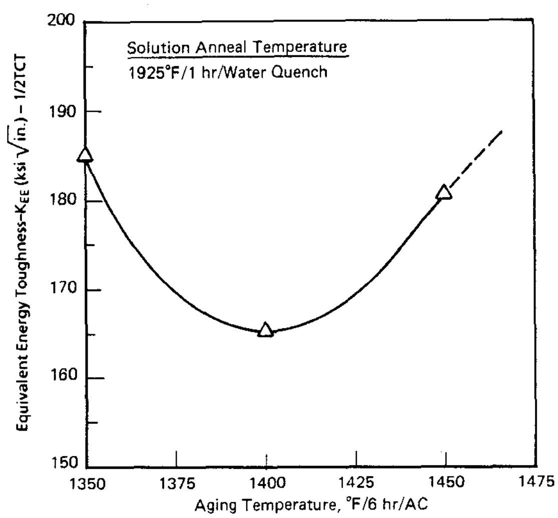

Figure 7. Effect of Aging Treatment on Toughness of Alloy 718

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The maximum fracture toughness occurs in the anneal temperature range of 1825 to 1875°F. When a higher anneal temperature such as 1925°F is used, improved toughness can still be achieved by choice of aging treatment. This is shown in Figure 7. Together these results indicate that for a given composition and alloy fabrication, the choice of specific heat treat parameters for anneal and single aging treatments can provide a optimum combination of fracture toughness >200 ksi √in, at yield strength $\geq$ 120 ksi, and hardness $\leq$ HRC 40.

Thus, from the foregoing heat treat response studies, the following inferences follow:

1. For the typical annealing temperature range of 1750°F to 1950°F, fracture resistance can vary significantly depending on the annealing cycle and the aging treatment. Improved toughness can be achieved by a relatively intermediate annealing temperature such as 1875°F $\pm$ 25°F, while satisfying the high yield strength requirements of 120 ksi minimum.

2. With a high annealing temperature (1925°F), improved toughness could be achieved with an overaging treatment, but decreased strength can be expected.

3. Single aging treatments rather than conventional double aging treatments can be used to achieve both high tensile strength and high

fracture toughness. An aging treatment of 1450 ± 25°F/6 hours/air cool is capable of meeting such requirements. This saves time, money, and complication of waivers relative to more complicated two step aging treatment.

Toughness Uniformity in Mill Production Samples

To assure that Alloy 718 purchased for Critical Service application has been processed and heat treated for high fracture toughness and environmental cracking resistance, materials heat treated as described above are required to achieve a minimum impact toughness of 40 ft-lb, in order to achieve a fracture toughness greater than 180 ksi √in. Figure 8 demonstrates that for billet sizes up to 5 1/2", this requirement can be readily met. However, for larger billet sizes or large forgings, the fracture resistance becomes significantly inferior.

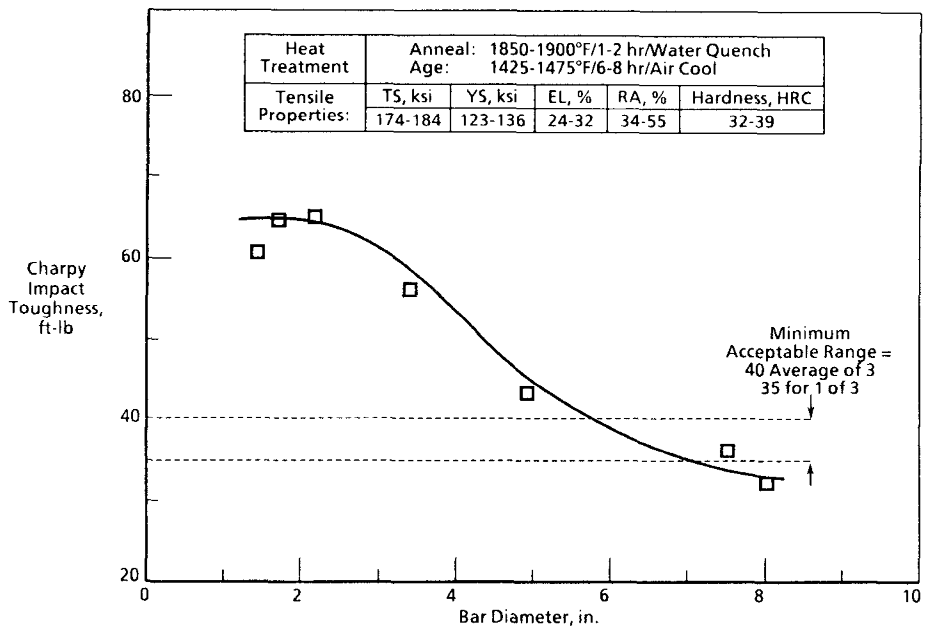

Figure 8. Effect of Heat Treat Section Size on Toughness of Production Samples of Alloy 718

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The decrease in fracture toughness with increased section size can be caused by factors such as (1) non-uniformity of hot work which affects heat treat response and microstructure and (2) density and distribution of carbide and carbonitride precipitates.

Table III compares the impact and fracture toughness values measured at the center and midwall locations of a heat treated 8" diameter stock used for tubing hanger. Here, the midwall location in the hanger component corresponds to 3/4 radius from center of the bar. Notice the center region shows inferior toughness which is less than the specified 40 ft-lb, or K_{IC} (J) of 180 ksi $\sqrt{in}$ for the component. This difference is attributed to the differences in microstructure between the two regions. Figure 9 shows the thru-wall microstructures in the bar. Compared to the mid-wall and OD regions, the center region shows more coarse grain size, higher density of NbC inclusions, and more grain boundary precipitates. Even though the fracture in the center and midwall region are both ductile, the above characteristics make void initiation and growth more favorable, resulting in low energy ductile tearing. This is supported by the slope of the J-resistance curve as shown in Figure 10. This slope is a measure of the tearing modulus of the alloy. Notice that in addition to the lower fracture toughness, the center region also shows a lower tearing modulus compared to material in the mid-wall region.

Table III. Variability in Impact and Fracture Toughness in 8" Diameter Forging for Alloy 718 Tubing Hanger

Location/ Orientation	Temp., °F	Impact Toughness		Fracture Toughness	
		Cv, ft-lb	Lateral Expansion, mils	J_{IC}, psi-in.	K_{IC}, J ksi $\sqrt{in.}$
Center/CR	0	31	20	975	170
Center/CR		32	19	900	163
Center/CR		31	20	975	170
3/4 Radius/CR	0	41	25	1560	214
3/4 Radius/CR		37	23	1662	221
3/4 Radius/CR		37	24	1885	236

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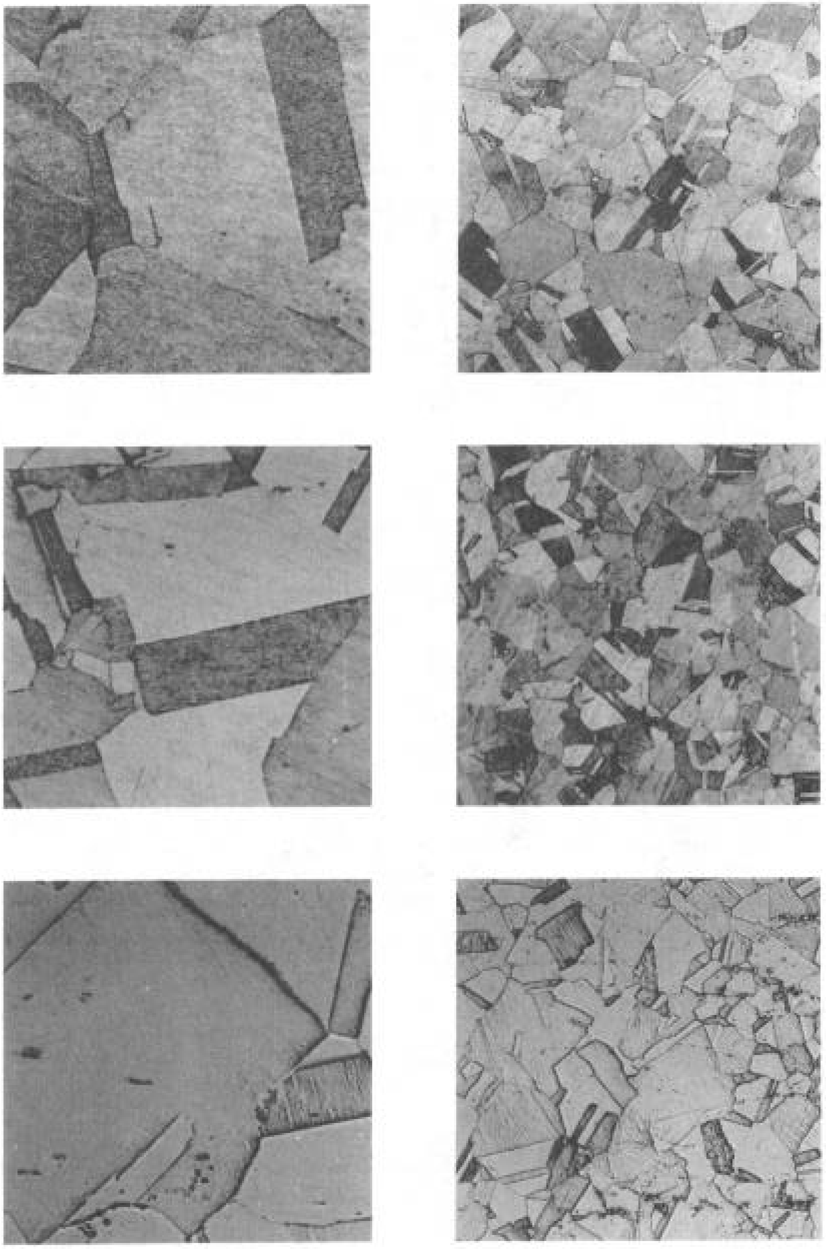
As stated earlier, differences in thru wall fracture resistance can also be caused by the density and distribution of carbide precipitates. Figure 11 shows how such precipitates can lower fracture resistance by promoting ductile fracture initiation. As the matrix deforms, local stress concentrations and plastic strain incompatibility between the matrix and the hard precipitates result in early cracking and decohesion of the inclusions. The resulting microcrack nuclei can then propagate into the matrix.

When the inclusions are aligned and occur in high volume fraction, they provide a low energy ductile fracture path. Figure 12 depicts the effect of such precipitate alignment. In this figure, the microstructure normal to the fracture plane in a compact tension specimen is shown. The top is the profile of the crack growth. Notice that when the inclusions



O.D.

Mid Radius

Center

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Magnification: 80X

Figure 9. Thru Wall Microstructure in the Longitudinal Orientation of 8" Diameter Forging for Alloy 718 Tubing Hanger

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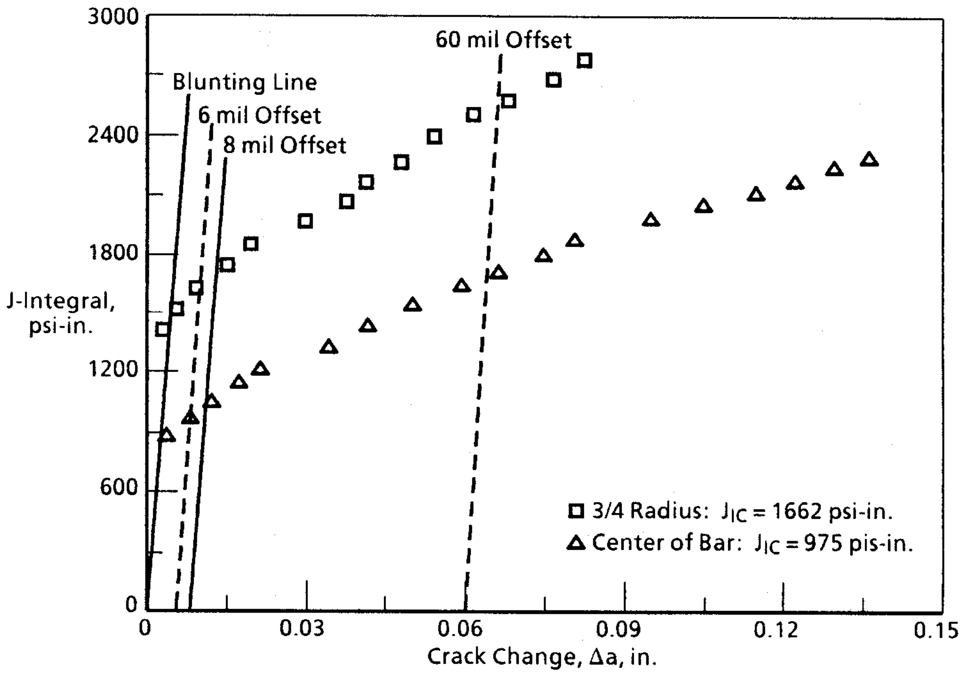

Figure 10: Crack Growth Resistance Curves in 8" Diameter Alloy 718 Tubing Hanger (X-6437) Showing Differences in Fracture Initiation and Ductile Tearing Modulus at Center and 3/4 Radius of Bar

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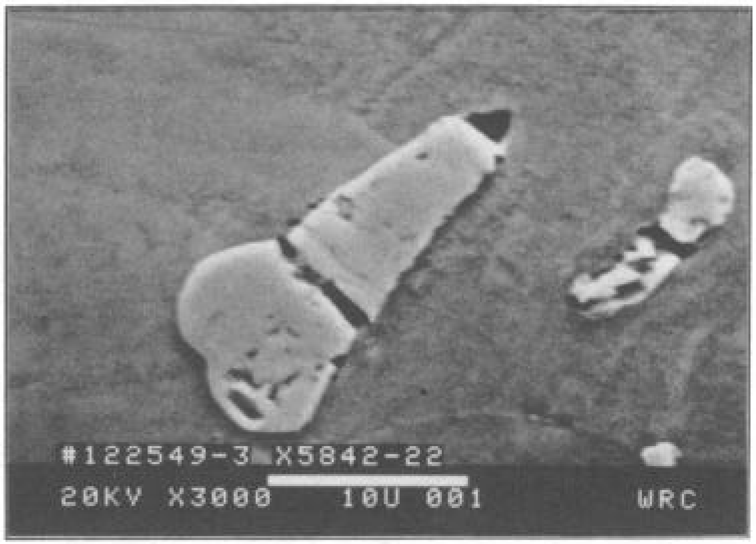
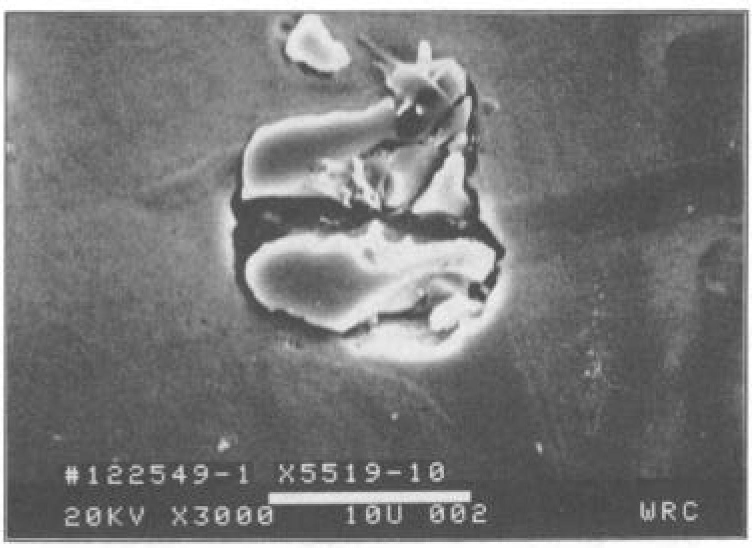


(a)



(b)

Figure 11. Micrographs Showing Role of Carbides and Inclusions in Initiating Cracks
As the matrix deforms, local stress concentration and incompatibility result in cracking of the inclusions. The resulting microcrack nuclei can then propagate into the matrix.

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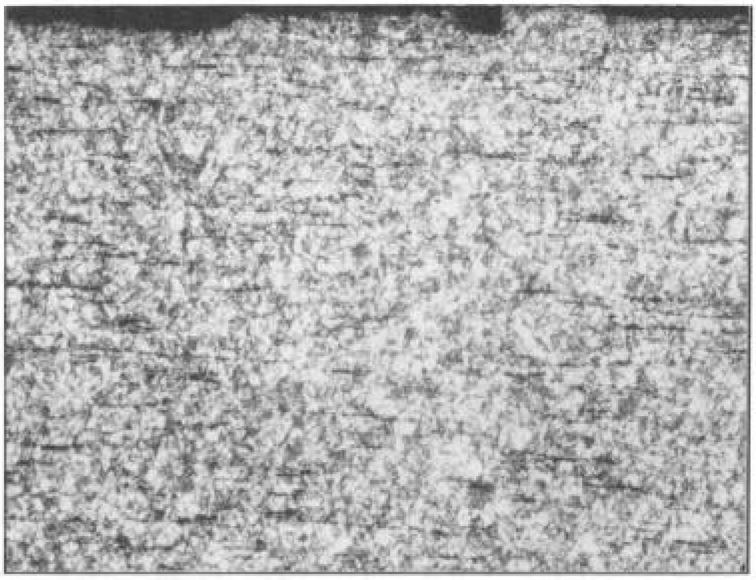
← Crack Propagation



(a) Fracture parallel to inclusion stringers (dark streaks). The alligned NbC precipitates control the fracture and cause low fracture resistance.

← Crack Propagation

(b) Uniform structure and little amount of inclusions. Note jagged texture of the crack profile indicative of high fracture resistance.

Figure 12. Fracture Profile and Microstructure in Alloy 718 Showing Effect of Alligned Precipitates

Magnification: 16X; *Etchant:* Mixed Acid

Magnification: 16X; *Etchant:* Mixed Acid

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are aligned, the crack profile is more stepped and planar. In contrast, when the inclusions are dispersed and nonaligned, the crack profile is more jagged as shown in Figure 12b. The differences in crack profile associated with precipitate alignment also shows up as differences in the fractography of ductile fracture. As shown in Figure 13 precipitate alignment results in "woody" texture and faceted ductile fracture compared to the randomly dispersed case. Thus, even though in both cases, the fracture is ductile, the differences in the micromechanics explain the difference in measured fracture toughness.

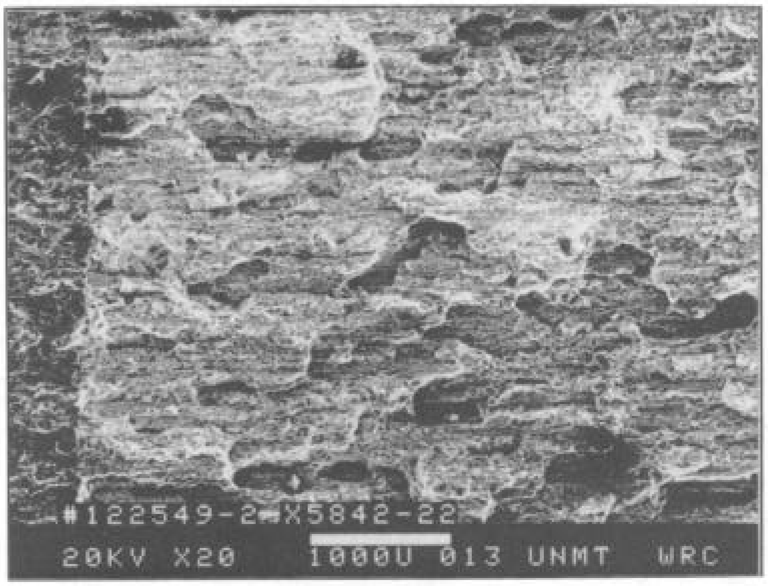
Evaluation of Environmental Cracking Resistance

Environmentally assisted cracking in production environments containing $H_2S_{(aq)}$, CO_2, and chlorides can occur by cathodic or anodic mechanisms. Though the conditions at which both mechanisms synergistically interact are unknown, it is clear that anodic cracking predominates at high temperature, and cathodic cracking occurs at lower temperature where hydrogen embrittlement predominates. Table IV shows the conditions and results of cathodic cracking experiments. Precracked double cantilever specimens (DCB) were used to measure the threshold stress intensity factor for unset of crack growth. C-rings prestressed to yield loads assess crack initiation resistance, and the slow strain rate (SSR) test evaluates the dynamic effects of plastic straining during hydrogen absorption.

In general, Alloy 718 shows good resistance to H_2S induced cracking under cathodic conditions. As shown in Table IV, even the heat treatment conditions which show low fracture toughness did not show cracking. The intergranular cracking observed in the SSR test has been attributed to the higher severity of the test. All samples heat treated in accordance to the conditions proposed in the previous sections of this paper did not show cracking.

Table V shows the conditions and results of anodic cracking experiments, using U bend specimens of Alloy 718 samples heat treated to standard aerospace specification. Samples heat treated as proposed in this paper did not crack under the test conditions shown in Table V. In addition, the results suggest that for high H_2S levels, anodic cracking susceptibility increased with yield strength. Also at high strength levels the results suggests that cracking susceptibility increases with H_2S concentration.

Fatigue Precrack ⟵|⟶ Crack Propagation



(a) Note the "woody" and flat texture of the fracture characteristic of low energy ductile tearing. No evidence of embrittlement.

Fatigue Precrack ⟵|⟶ Crack Propagation

(b) Note more dimpled, ductile fracture, consistent with the higher fracture resistance of this sample of 718.

Figure 13. Fracture Details Just Ahead of Fatigue Precrack in Alloy 718
Sample with (a) high localized coarse precipitates,
(b) fine and uniformly dispersed precipitates.
Magnification: 20X

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Table IV. Hydrogen Embrittlement Resistance of Alloy 718

Specimen ID/Type	Heat Treat	Hardness, HRC	Test Temp., °F	Condition	Result
X5322-5,2/DCB	*	37-38	RT-250°F	25% NaCl + 200 psi H_2S, C·S Coupled, 4 Weeks	No Cracking
X4140-38,41/C-Rings	*	38-39	RT-250°F	25% NaCl + 200 psi H_2S, 4 Weeks	No Cracking
X5322-1/DCB	*	38	32	NACE Solution + C·S Coupled 4 Weeks	No Cracking
X5058-5,7/C-Ring	**	38-41	RT	NACE Solution + C·S Coupled 2 Months	No Cracking
X5322-4/SSR	**	38	RT	NACE Solution + C·S Coupled 40 Hours	Intergranular Cracking

** 1925°F/1 hr/WQ + 1400°F/8 hr/AC*
*** 1950°F/1 hr/AC + 1325°F/8 hr/FC 1150°F/8 hr/AC*

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Table V. U-Bend Stress Corrosion Cracking Tests Alloy for 718
25% NaCl – 6-Day Tests

	Test #1	Test #4	Test #5
Heat Treatment	1950°F/1 hr/AC 1325°F/8 hr/FC 1200°F/10 hr/AC	1950°F/1 hr/WQ 1325°F/8 hr/FC 1150°F/8 hr/FC	1950°F/1 hr/WQ 1325°F/8 hr/FC 1150°F/8 hr/FC
Yield Strength, psi	~135,000	~156,000	~156,000
Stress	>y.s.	>y.s.	>y.s.
Temperature, °F	400	400	400
Pressure, psi	14,000	14,000	1500
H_2S, %	3.5	3.5	1
CO_2, %	10	10	10
P_{H_2S}, psi	490	490	22.5
SCC Results, # Failed/# Tested	0/2	2/2	0/2

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Conclusions

1. Alloy 718 subjected to anneal plus single age heat treat cycles provides the required properties for application in deep, corrosive oil and gas wells.

2. For optimum combination of strength, toughness, and environmental cracking resistance an anneal temperature within 1825 to 1925°F followed by water quench and an aging temperature of 1450 $\pm$ 25°F is proposed.

3. Conventional aerospace heat treatments which optimize strength and creep rupture properties are unacceptable due to decrease in fracture toughness, and environmental cracking resistance.

4. Degradation in fracture resistance is related to the amount and distribution of carbide inclusions and grain boundary precipitation, both contributions are higher in large section components.

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