HIGH TEMPERATURE CORROSION FATIGUE AND GRAIN SIZE CONTROL

IN NICKEL-BASE AND NICKEL-IRON-BASE SUPERALLOYS

M. Yoshiba and O. Miyagawa[*]

Department of Mechanical Engineering, Faculty of Technology
Tokyo Metropolitan University
Fukazawa, Setagaya-ku, Tokyo 158, Japan

[*] Tokyo Metropolitan University, Professor Emeritus

Abstract

In order to clarify an effect of hot corrosive environment both on the fatigue strength and the fatigue fracture behavior of superalloys, the rotating-bending fatigue tests were conducted for nickel-base Inconel 751, nickel-iron-base Inconel 718 and Fe-42Ni-15Cr-3Mo alloy, at 800°C in the Na_2SO_4-NaCl molten salt environment as well as in air. A grain size was controlled in a wide range from the viewpoint of its practical importance. It was revealed that hot corrosive environment brings about a significant fatigue strength degradation for all the alloys, in particular with reduced grain size, by affecting the initiation and propagation processes of the fatigue cracks. A grain size was confirmed to concern strongly in the corrosion fatigue crack propagation behavior. In particular, the detrimental effect of reducing a grain size was emphasized on the enhanced intergranular crack propagation. A fundamental criterion of the grain size control for the improved corrosion fatigue strength properties of superalloys was presented in connection with a Ni content.

Introduction

Superalloys used predominantly for hot section components of various heat engines such as jet engines, gas turbines, diesel engines and so on, are inevitably subjected to the simultaneous effects of both the mechanical damages such as creep and/or fatigue and the corrosive damage due to a hot corrosion. For the practical applications of superalloys to be successful, it should be important for the mechanical performances to be evaluated in such an aggressive environment. For the creep rupture properties, from this standpoint, a lot of useful knowledge about the corrosion-environmental effect has been accumulated recently through a variety of worldwide studies containing authors' study (1,2). However, the environmental effect on the fatigue strength and fatigue fracture behavior remains unclear, although it is also a serious problem. The fatigue fracture behavior, and hence the fatigue strength, should be more affected by the corrosive environment than the case of a creep, because a fatigue failure is apt to initiate from the alloy surface. Such a corrosion-environmental effect on the fatigue properties is predicted to be most significant at the temperature range between approximately 800 and 900°C in which both hot corrosion and mechanical fatigue damages are dominant (3).

On the other hand, it has been well known that grain size is one of the most important material factors affecting the fatigue properties of many kinds of wrought alloys. However, the grain size dependence of high temperature corrosion fatigue properties has been hardly clarified.

In the present study, then, the high cycle fatigue tests were conducted at 800°C for some superalloys with a range of grain sizes controlled, in air and in the Na_2SO_4-NaCl molten salt environment, and the corrosion-environmental effect on the fatigue strength as well as its grain size dependence were investigated in connection with the initiation and propagation behavior of the corrosion fatigue cracks.

Materials and Experimental Procedures

Materials

Three kinds of wrought superalloys were used in this study: γ'-precipitation-hardened nickel-base Inconel 751 alloy, γ''- and/or γ'-precipitation-hardened nickel-iron-base Inconel 718 alloy, and γ'-hardened Fe-42Ni-15Cr-3Mo alloy. These chemical compositions are given in Table I. The latter one is a new alloy developed by authors group for a purpose of high performances

Table I. Chemical Compositions of Superalloys Used (Mass %)

Alloys	C	Si	Mn	P	S	Ni	Cr	Ti	Al	Mo	Fe	Others
Inconel 751	0.09	0.20	0.53		0.007	Bal.	16.07	2.05	1.10	–	5.74	Nb+Ta 1.18
Inconel 718	0.03	0.11	0.22	0.011	0.005	Bal.	17.54	0.92	0.42	3.02	18.54	Nb+Ta 4.99
Fe-42Ni-15Cr-3Mo	0.05	0.26	0.52	0.010	0.002	41.91	15.02	2.82	0.87	3.05	Bal.	B 0.012

Table II. Typical Heat Treatment Conditions and the Corresponding Grain Size and Vickers Hardness Number

Alloys	Heat Treatment Conditions	Grain Size (μm)	HV (196N)
Inconel 751	1100°C×2h→WQ + 800°C×24h→AC.	44	313
	1200°C×2h→WQ + 800°C×24h→AC.	125	306
Inconel 718	1000°C×1h→AC + 720°C×8h→FC(600°C)→AC.	15	441
	1100°C×2h→WQ + 800°C×24h→AC.	145	341
Fe-42Ni-15Cr-3Mo	1000°C×2h→WQ + 800°C×24h→AC.	30	352
	1200°C×2h→WQ + 800°C×24h→AC.	130	353

in aggressive environments (4).

Different kinds of heat treatments were adopted for these three alloys in order to control mainly a grain size in a wide range. The typical heat treatment conditions adopted are listed in Table II, along with the corresponding average grain size and Vickers hardness number. For Inconel 751, a grain size has been controlled most widely between 10 and 290 μm in grain diameter, which approach is already described elsewhere in detail (5,6).

The smooth bar specimens with 8 mm in diameter and 15 mm in gage length were machined from the heat-treated rods. Furthermore, in order to examine the fatigue crack behavior in detail, the notched bar specimens also were adopted with 5 mm in notch radius and 8 mm in specimen diameter at the notch root. They were emery-polished through 500 grit, and were cleaned ultrasonically in acetone.

Experimental Procedures

The fatigue tests were conducted at 800°C using the rotating-bending fatigue testing machines (1500 rpm). In order to simulate an actual hot corrosive environment, the specimens were coated with synthetic salt mixture composed of 90% Na_2SO_4 plus 10% NaCl (m.p.=785°C). The amount of salt mixture precoated was 0.4 kg/m^2. For the prolonged fatigue tests, the coating of the same amount of salt mixture was repeated at every 3×10^5 cycles to refresh the corrosive media. The tests in static air also were carried out using the specimens without coating the salt mixture. These two types of tests will be termed as "in hot corrosive environment" and "in air", respectively.

For both the fatigue-failure and the interrupted specimens, the metallographic examinations were made mainly on the longitudinal sections by means of an optical microscope and an electron probe X-ray microanalyzer (EPMA). The observation of the fracture surface also was conducted by a scanning electron microscopy (SEM).

Results and Discussion

Fatigue Strength in Air and in Hot Corrosive Environment

Figures 1, 2 and 3 show the S-N curves (Wöhler curves) obtained from the fatigue tests at 800°C both in air and in hot corrosive environment for Inconel 751, Inconel 718 and Fe-42Ni-15Cr-3Mo alloy, respectively. In air, the fine-grained specimens exhibited longer fatigue life at the higher stress levels. However, such a superiority of fine-grained specimens disappeared as a stress level lowers. In hot corrosive environment, on the other hand, a significant fatigue life reduction occurred in all the alloys, although the degree of degradation is different dependingly on the alloy chemistry and the grain size as mentioned below. High temperature corrosion fatigue is also characterized by the disappearance of the fatigue limit (the endurance limit) along with the increased data scatter. In such an aggressive environment, the fine-grained specimens are found to yield rather lowered fatigue life or at best the same one as compared with the coarse-grained specimens.

The fatigue strengths at the representative cycles in both environments are summarized in Fig. 4, along with the corrosion fatigue strength ratio as a measure of the corrosion sensitivity of the fatigue strength. It is noted again that in air a grain size favorable for the increased fatigue strength is reversed dependingly on the cycle life: a fine grain is favored for the short life term, and a coarse one is for the long life term. As regards the alloy comparison, two nickel-iron-base superalloys were found to have the
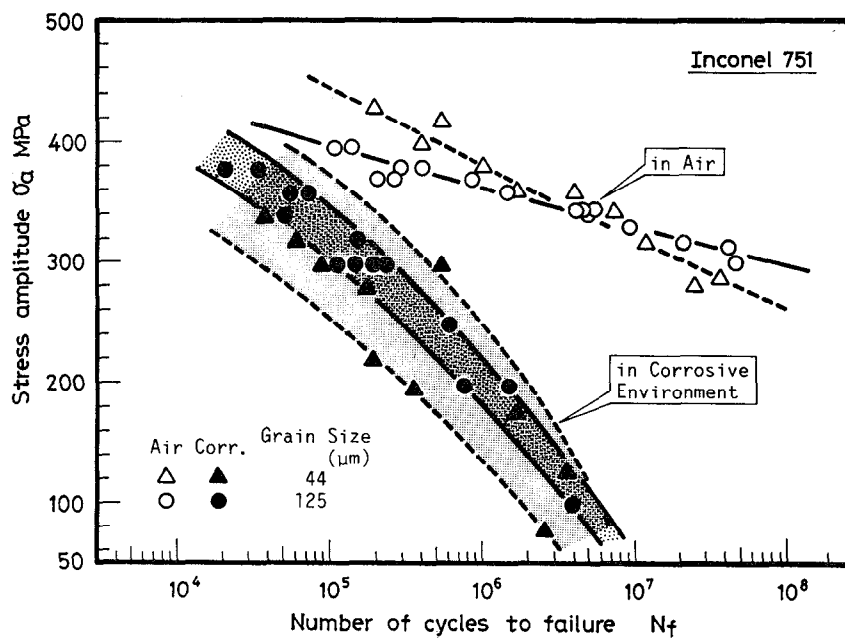
827

Figure 1 –
S-N curves of Inconel 751 specimens with different grain sizes at 800°C in air and in hot corrosive environment.
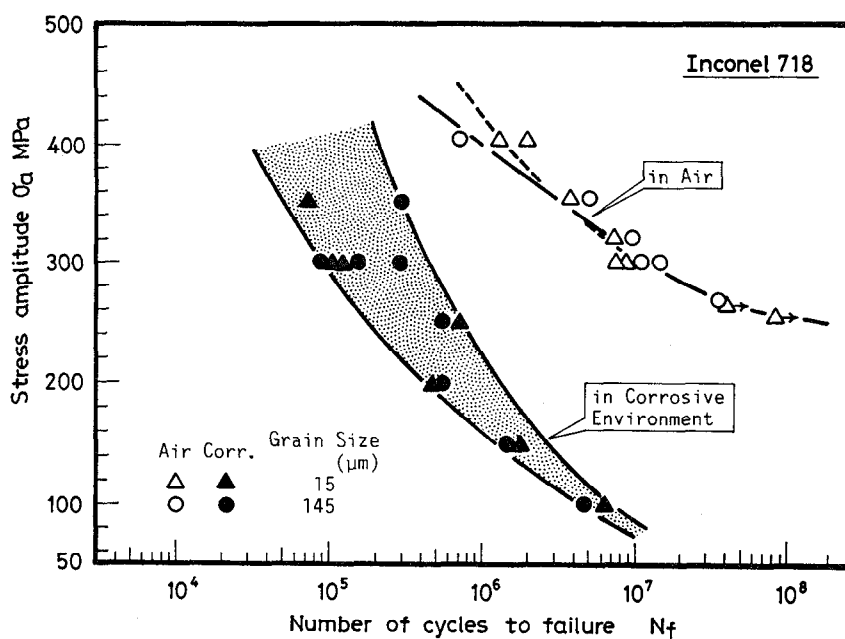

Figure 2 –
S-N curves of Inconel 718 specimens with different grain sizes at 800°C in air and in hot corrosive environment.
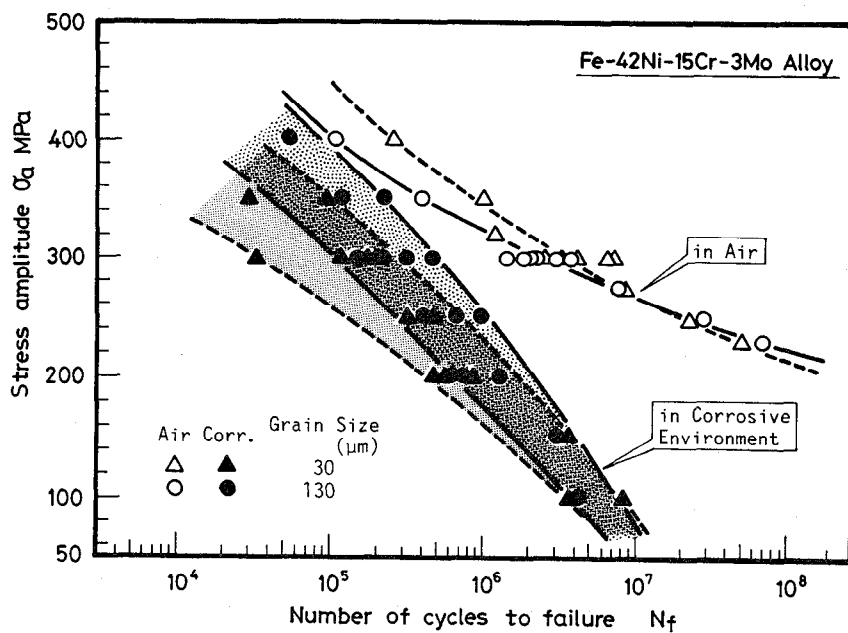

Figure 3 –
S-N curves of Fe-42Ni-15 Cr-3Mo alloy specimens with different grain sizes at 800°C in air and in hot corrosive environment.
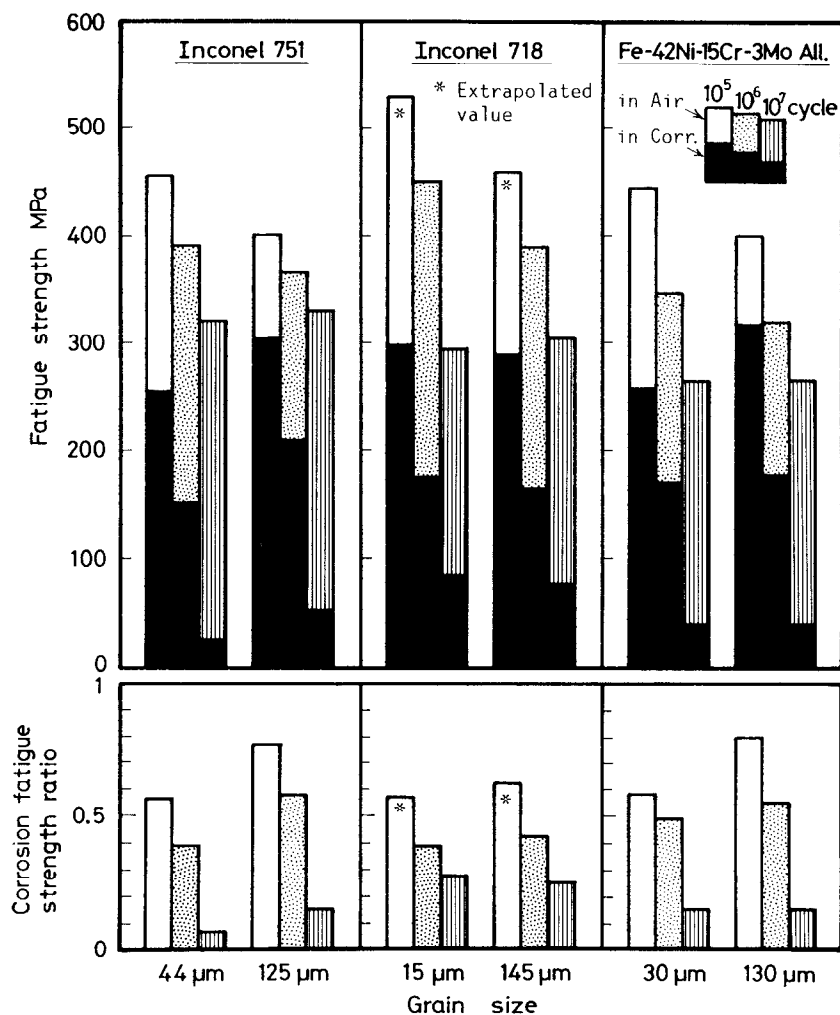
Figure 4 — Summary of the fatigue strengths in air and in hot corrosive environment along with the corrosion fatigue strength ratio.

fatigue strength comparable to Inconel 751 with the similar grain size in the short life regime, although in the long life regime their fatigue strengths were fairly lowered as compared with Inconel 751.

In hot corrosive environment, both the fatigue strength and its corrosion sensitivity were found to show somewhat different grain size dependence between nickel-base Inconel 751 and two nickel-iron-base alloys. For Inconel 751, an advantage of grain size coarsening is pronounced for the improved corrosion fatigue strength, along with the minimized corrosion sensitivity. For two nickel-iron-base alloys, on the contrary, the corrosion fatigue strength is hardly dependent on a grain size except for the short life term of Fe-42Ni-15Cr-3Mo alloy, so that an advantage of coarse grain is rather diminished.

Fatigue Fracture Behavior in Air and in Hot Corrosive Environment

From the metallographic examinations about the longitudinal sections of fatigue-failured specimens, along with fatigue-interrupted specimens in air and in hot corrosive environment, a fracture mode was found to change from a transgranular mode to an intergranular mode as a grain size decreases. Figure 5 shows the schematic illustrations of the fatigue fracture modes observed in three alloys as functions of a grain size, a stress level and an environment, along with the fracture surface morphologies of Fe-42Ni-15Cr-3Mo alloy, as a typical example. The present results about the grain size dependence of fatigue fracture mode are consistent with the previous result for Inconel 751 (5,6): an intergranular fracture becomes dominant as a grain size decreases less that about 50 μm, while a transgranular one is dominant for
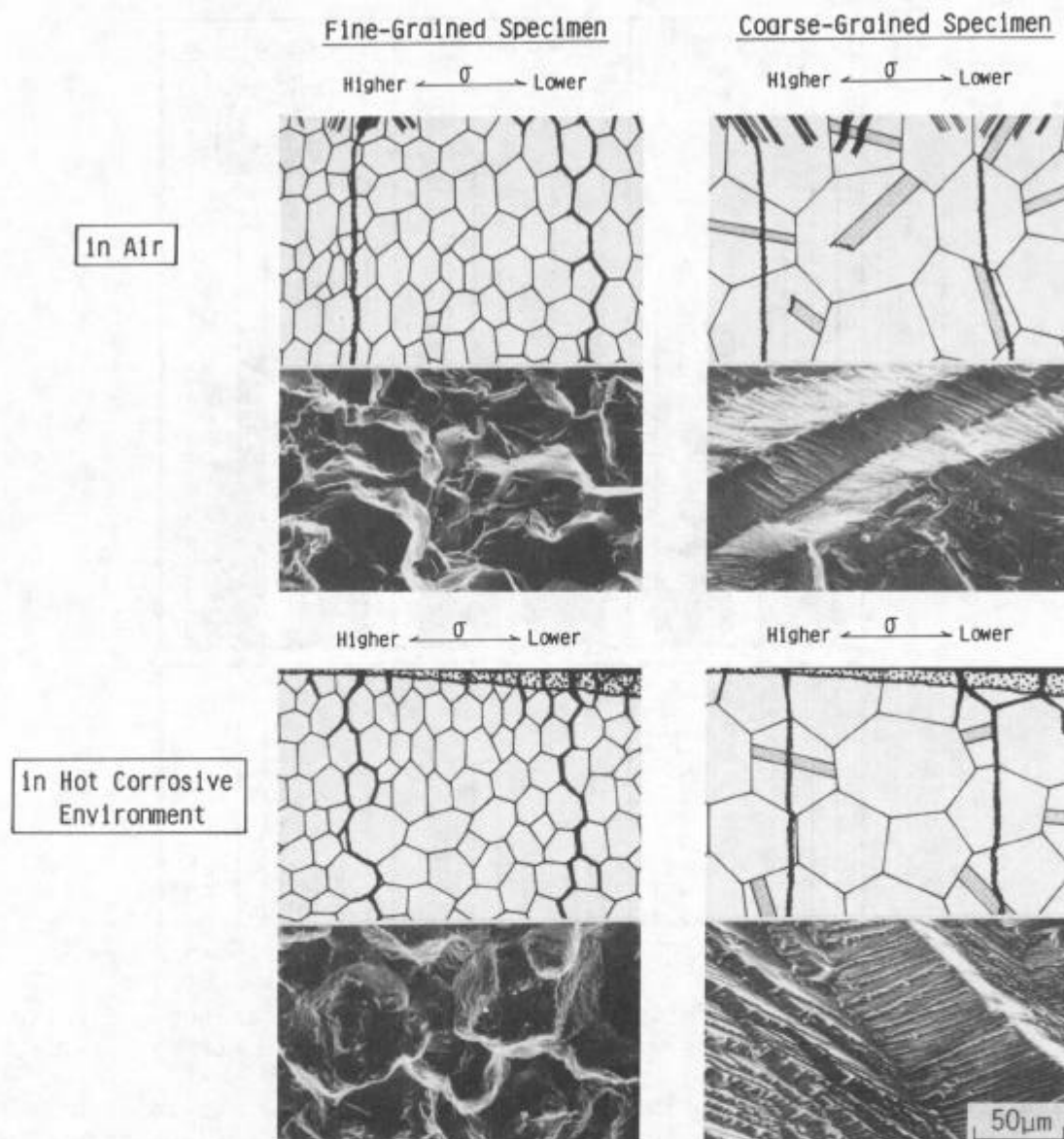
Figure 5 – Schematic illustrations showing different types of fatigue
fracture behavior and the corresponding fractgraphs of Fe-
42Ni-15Cr-3Mo alloy specimens in connection with a grain
size, a stress level and an environment.

the coarser grain size more than about 100 μm, and an intermediate grain size
between 50 and 100 μm results in a mixed fracture mode. Hot corrosive envir-
onment appears to bring about little change in the fracture mode. However,
it should be noted that in the aggressive environment the fatigue cracks al-
ways initiate at the grain boundary subjected to the intergranular penetration
of sulfides followed by oxides and so on, regardless of an alloy chemistry and
a grain size. Figure 6 shows a typical intergranular attack by sulfides etc.
occurred in the Inconel 751 corrosion fatigue specimen (5). Such a very
small corrosion pit, with a depth of approximately one grain diameter, is
possible to develop already in the early fatigue stage in which the mechanical
fatigue damage is hardly accumulated, so that it provides itself the crack
nucleation site. Therefore, the intergranular-attack-stimulated premature
crack initiation should be one of the principal causes for the marked fatigue
strength degradation, as schematically shown in Fig. 7. It should be noted
that the development of intergranular attack is affected strongly by an alloy
chemistry, i.e. the alloy composition, and is rather insensitive to a stress
level and a grain size. Then it is suggested that the alloy with the mini-
mized intergranular attack sensitivity is favored to inhibit the corrosion-
induced strength degradation. In this respect, nickel-iron-base superalloys

Grain size : 125 μm
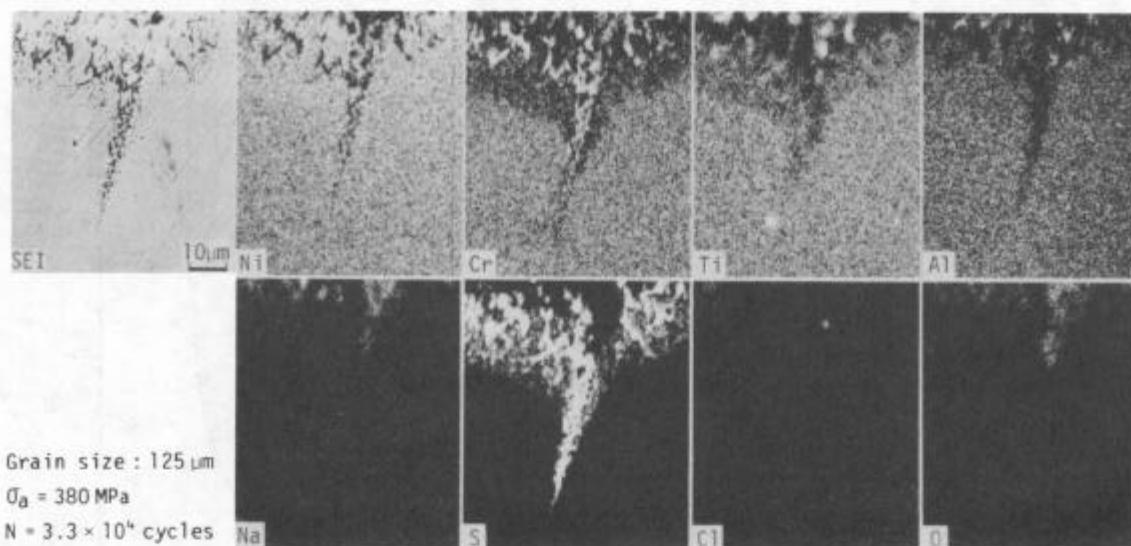σ_a = 380 MPa
N = 3.3 × 10⁴ cycles

Figure 6 – Characteristic X-ray images at the early stage of intergranular attack in a coarse-grained Inconel 751 specimen fatigue-tested in hot corrosive environment.
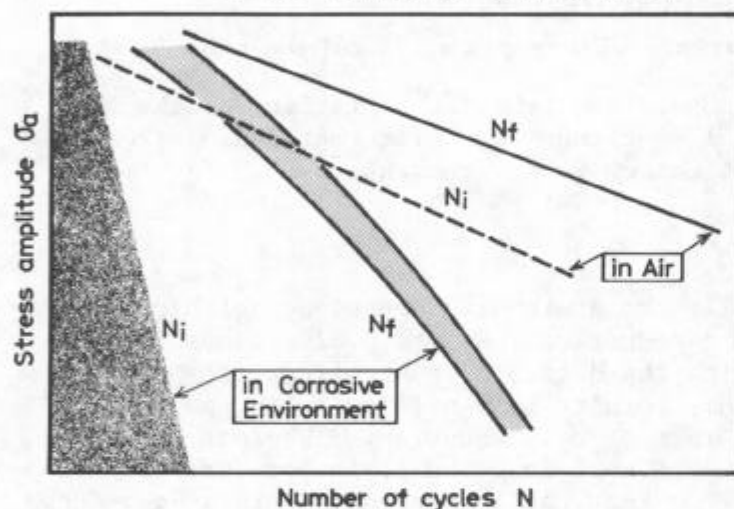

Figure 7 – Schematic representation showing the N_f and N_i in air and in hot corrosive environment. N_f and N_i denote the number of cycles to failure and the number of cycles to crack initiation, respectively.

seem to be more beneficial because of their inherent good intergranular attack resistance (7).

It has been known that the fatigue crack propagation process also is affected by the corrosive environments (8,9). Figure 8 shows the initiation and propagation behavior of fatigue cracks for Inconel 751 specimens in air and in hot corrosive environment, which was obtained from the microstructural measurement of the interrupted fatigue specimens. It is evident again that hot corrosive environment brings about a premature fatigue crack initiation. Furthermore, in the fine-grained specimens the corrosion fatigue cracks tend to propagate so rapidly almost along the grain boundary as to cause a premature intergranular fracture. This should be attributed to the combined effect of the mechanical fatigue and chemical corrosive damages concentrated to the grain boundary region (5). On the contrary, the coarse-grained specimens are apt to be subjected to the isolated damage: the fatigue damage points into the grain, while the corrosive damage prefers the grain boundary.

Grain Size Dependence of Corrosion Fatigue Strength

It has been already discussed for a nickel-base Inconel 751 alloy that in air the grain size dependence of high temperature fatigue strength is determined by two competitive factors: one is a tendency of the intergranular fracture and the other is an ability to disperse the cyclic slip deformation
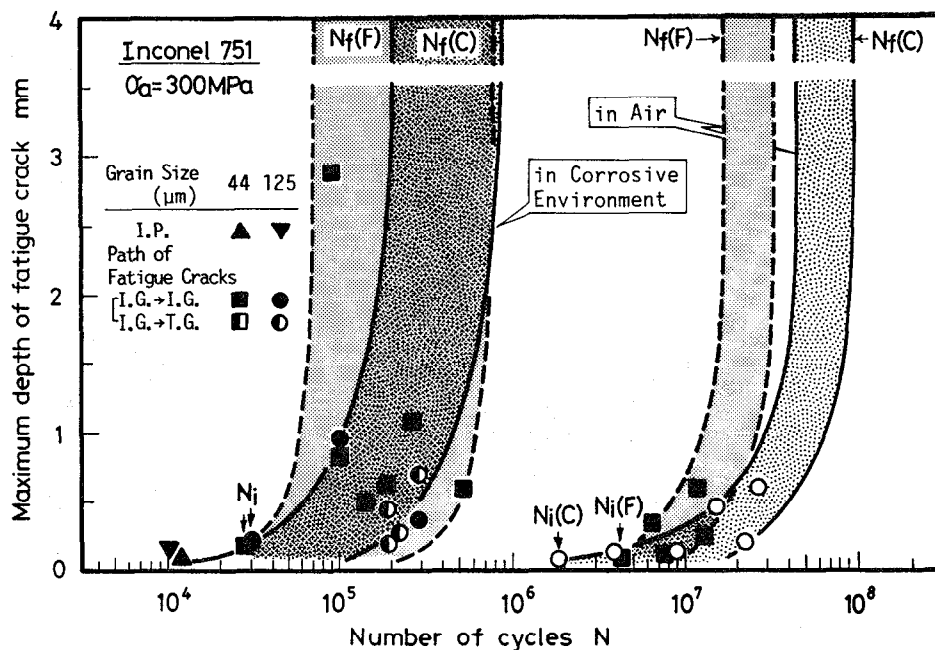
I.P.:Intergranular Penetration, I.G.:Intergranular, T.G.:Transgranular

Figure 8 – Initiation and propagation behavior of fatigue cracks
in the Inconel 751 specimens with different grain sizes
in air and in hot corrosive environment.

(6).   Figure 9 shows schematically the grain size dependence of high temper-
ature fatigue strength in air.   A reduction in a grain size tends to promote
the slip dispersion associated with the diffusion-controlled recovery process
such as a dislocation climb.   This results in a difficulty in the fatigue
crack initiation and propagation in the grain interior.   Whereas, this in
return tends to cause the fracture rather along the grain boundary with rela-
tively high strain concentration, so that the intergranular-cracking-induced
strength degradation becomes more significant as a grain size decreases (10).
Then, it should be reasonable to consider that for the increased fatigue
strength in air a grain size should be controlled rather small as far as a
transgranular fracture is dominant.

On the contrary, the grain size dependence of the high temperature cor-
rosion fatigue strength seems to be different among the alloy systems.   For
a nickel-base Inconel 751 alloy, the corrosion fatigue strength has been
found to lower monotonically with reduction in grain size, as schematically
shown in Fig. 10 (5).   Again, a reduction in a grain size in this alloy
system is very harmful to the corrosion sensitivity as well as the corrosion
fatigue strength itself, because of the corrosion-enhanced intergranular
crack propagation rate due to the combined mechanical and chemical damages
with the low melting nickel sulfide formation such as Ni_3S_2-Ni eutectic (m.p.
=637°C).   In this respect, nickel-iron-base alloys seem to be more useful
than nickel-base alloys for suppressing the nickel sulfide formation, which
should be reflected in the mitigated grain size dependence of the corrosion
fatigue strength for nickel-iron-base alloys as well as in their restrained
crack initiation and propagation behavior.   Then, it can be concluded that
for the improved corrosion fatigue strength properties of superalloys, in
particular with higher Ni content, a grain size has to be controlled appro-
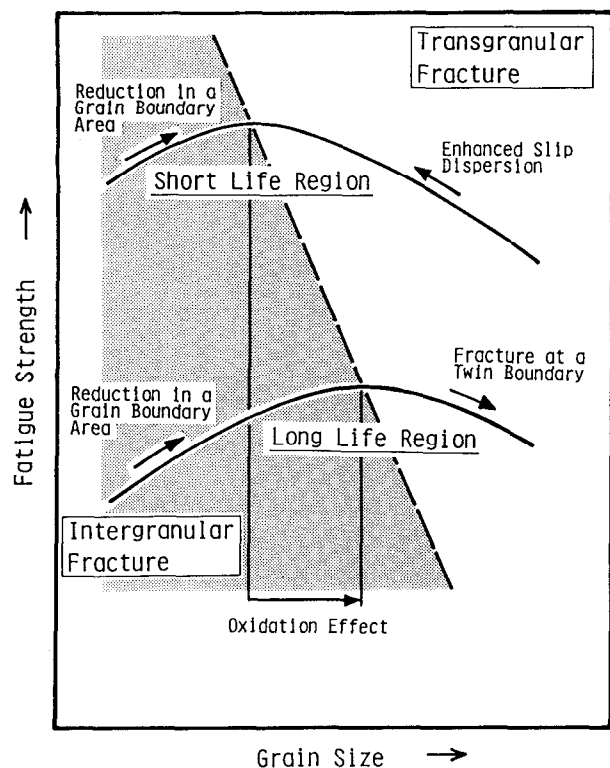priately large enough not to induce an intergranular fracture.

Figure 9 –
Schematic representation showing the grain size dependence of high temperature fatigue strength of superalloys in air in connection with various affecting factors.
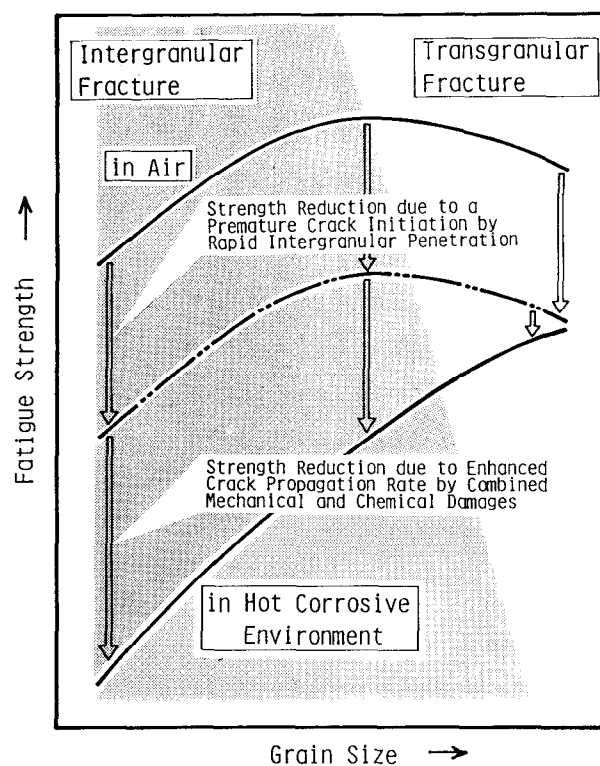
Figure 10 –
Schematic representation showing the grain size dependence of the corrosion fatigue strength of nickel-base superalloys in connection with various affecting factors.

Concluding Remarks

1) Hot corrosive environment was found to bring about a significant fatigue strength degradation for the nickel-base and nickel-iron-base superalloys. Such a marked strength degradation was confirmed to result partly from the intergranular-attack-stimulated premature crack initiation, depending mainly on a corrosion resistance, particularly on the intergranular attack resistance, of the alloys.

2) Corrosion fatigue crack propagation behavior was found to depend strongly on a grain size. In particular, a reduction in grain size results in the enhanced intergranular crack propagation rate because of the combined effect of the mechanical fatigue and chemical corrosive damages concentrated to the grain boundary, so that the corrosion-induced strength degradation is more significant than the case of a coarse grain size.

3) Hot corrosive environment caused a different grain size dependence of the fatigue strength in air. In order to improve the corrosion fatigue strength properties of superalloys, a grain size has to be controlled large enough not to induce an intergranular fracture. Such a demand becomes more serious in nickel-base superalloys with higher Ni content.

Acknowledgments

References

1. V. Guttman and M. Schutze, "Interactions of Corrosion and Mechanical Properties," High Temperature Alloys for Gas Turbines and Other applications, W. Betz et al., eds., Part I (Dordrecht, Holland: D. Reidel Publ. Co., 1986), 293–326.

2. M. Yoshiba and O. Miyagawa, "Effect of Hot Corrosive Environment on Creep Rupture Properties of Commercial Iron-Nickel-Chromium Heat Resisting Alloys," Proc. International Conference on Creep, (Tokyo: Japan Society of Mechanical Engineers, 1986), 193–198.

3. D. A. Spera and S. J. Grisaffe, "Life Prediction of Turbine Components," (NASA TMX 2664, 1973).

4. M. Yoshiba, O. Miyagawa, and H. Fujishiro, "High Temperature Performances of New Nickel-Iron-Base Superalloys in Aggressive Environments," Proc. MRS International Meeting on Advanced Materials, (Materials Research Society), to be Published.

5. M. Yoshiba, O. Miyagawa, K. Sato, and H. Fujishiro, "Effect of Grain Size on the High Cycle Fatigue Strength of a Nickel-Base Superalloy Subjected to Hot Corrosion," Trans. Japan Society of Mechanical Engineers, 50(1984) 1113–1122 (in Japanese).

6. M. Yoshiba, O. Miyagawa, and H. Fujishiro, "Effects of Grain Size and Notch on the High Cycle Fatigue Strength of a Nickel-Base Superalloy at Elevated Temperature," Trans. Japan Society of Mechanical Engineers, 50(1984), 1443–1452 (in Japanese).

7. M. Yoshiba, O. Miyagawa, and H. Fujishiro, "Hot Corrosion Behavior of Heat Resisting Alloys," J. Iron and Steel Inst. Japan, 67(1981), 996–1005 (in Japanese).

8. W. Hoffelner and M. O. Speidel, "The Influence of the Environment on the Fatigue Crack Growth of the Nickel-Base Superalloy IN 738LC and IN 939 at 850°C," Behavior of High Temperature Alloys in Aggressive Environments, I. Kirman et al., eds., (London: The Metals Society, 1979), 993–1004.

9. S. Floreen and R. H. Kane, "Effects of Environment on High-Temperature Fatigue Crack Growth in a Superalloy," Metall. Trans., 10A(1979), 1745–1751.

10. M. Gell and G. R. Leverant, "Mechanisms of High-Temperature Fatigue," Fatigue at Elevated Temperatures (ASTM STP 520), (1973), 37–67.