CONTROLLING INTERMEDIATE TEMPERATURE FATIGUE
CRACK GROWTH IN NICKEL BASE SUPERALLOYS BY
MICROSTRUCTURAL VARIATIONS

S. Floreen and R. H. Kane
Inco Research & Development Center
Sterling Forest, Suffern, NY 10901

*Increasing the grain size of superalloys markedly
reduced the 650°C fatigue crack growth rates in air and in
helium.  Grain boundary microstructure changes and overaging
also were beneficial in air, but not in helium.  Structures
optimized by testing in air may not be effective when some
species other than oxygen dominates the environment.*

INTRODUCTION

The role of microstructure in elevated temperature
fatigue crack growth is not clear.  At 1 Hz increasing the
grain size was found to reduce crack growth rates[1], but
overaging heat treatments produced no significant changes[1,2].
At lower frequencies however, overaging has been found to be
beneficial[2,3-6].  Overaging also is beneficial in creep
loading.  Homogenizing slip within the grains, and/or
changing the grain boundary microstructure have been cited
as the reasons for the improvement in properties[7].

In the present study the fatigue crack growth behavior
of a number of Ni-base superalloys was examined.  The hot
working and heat treating histories were varied to produce
changes in the grain size, grain shape and carbide, gamma
prime, and inclusion particle morphologies.  Tests were run
in air and also in high purity Helium so that the effects of
the air environment could be evaluated.

EXPERIMENTAL PROCEDURE

Table 1 summarizes the alloys examined and the processing
history used to vary the microstructures.  The variations in
structure with each alloy were as follows:

Table 1.  Materials Processing History.

| Alloy | Code | Processing History |
|-------|------|--------------------|
| ASTROLOY | A | 1095°C/1 h/AC+650°C/24 h/AC+ 760°C/8 h/AC |
|  | B | 1165°C/1 h/AC+650°C/24 h/AC+ 760°C/8 h/AC |
|  | C | 1165°C/1 h/FC to 1065°C/AC+ 650°C/24 h/AC+760°C/8 h/AC |
| NIMONIC* alloy 115 | D | 1190°C/2 h/FC to 1000°C/AC |
|  | E | 1190°C/2 h/AC+1075°C/6 h/AC |
| INCONEL* alloy X-750 | F | 1095°C/2 h/WQ+885°C/24 h/AC+ 705°C/20 h/AC |
|  | G | 1095°C/2 h/WQ+705°C/20 h/AC |
| WASPALOY-1 | H | As-forged + 845°C/24 h/AC+ 760°C/16 h/AC |
|  | J | 1095°C/1 h/WQ+845°C/24 h/AC+ 760°C/16 h/AC |
|  | K | 1175°C/4 h/WQ+845°C/24 h/AC+ 760°C/16 h/AC |
| WASPALOY-2 | L | 1095°C/Hot Roll+845°C/24 h/AC+ 760°C/16 h/AC-Longitudinal Orientation |
|  | M | 1095°C/Hot Roll+845°C/24 h/AC+ 760°C/16 h/AC-Transverse Orientation |
| WASPALOY-3 | O | 1035°C/Hot Roll+845°C/24 h/AC+ 760°C/16 h/AC-Longitudinal Orientation |
|  | P | 1035°C/Hot Roll+845°C/24 h/AC+ 760°C/16 h/AC-Transverse Orientation |

Astroloy - The solution heat treatment was varied to change the grain size, codes A and B, and a furnace cooling after solution annealing, Code C, was used to introduce serrated grain boundaries(8).

NIMONIC* Alloy 115 - Furnace cooling after solution annealing was employed to produce a blocky $M_{23}C_6$ grain boundary carbide (Code D).  Air cooling after solution annealing followed by a 1075°C heat treatment (Code E) was employed to produce a semi-continuous $M_{23}C_6$ grain boundary carbide(9).

*Trademark of the Inco families of Companies.

INCONEL* alloy X-750 - An intermediate 885°C heat treatment before aging, Code F, was used to produce extensive $M_{23}C_6$ grain boundary carbide and a grain boundary zone denuded of $\gamma'$(10). Omitting this treatment, Code F, produced a much finer carbide and less denuded zone.

Waspaloy-1 - Stock was given different solution heat treatments to produce equiaxed grains of various sizes, Codes H, J and K.

Waspaloy-2 - Stock was hot rolled at 1095°C and aged to produce a duplex microstructure of coarse and very fine grains strung out parallel to the rolling direction. Samples were cut in the longitudinal (Code L) and transverse (Code M) directions.

Waspaloy-3 - Stock was hot rolled at 1035°C and aged to produce equiaxed grains with inclusion stringers parallel to the rolling direction. Samples were again cut in longitudinal (Code O) and transverse (Code P) orientations.

Compact tension fracture toughness samples were machined from the stock materials. Test specimens were 30.5 mm x 29.3 mm x 9.2 mm thick following the dimensions used in earlier studies(12). After machining the samples were fatigue precracked at room temperature at low stress intensities so that more than 50,000 cycles were used to grow the starting cracks.

The test facility has been described in detail in an earlier paper(11). Specimens were brought to temperature and exposed to the environment, either air or 99.995% pure helium, for about one hour before loading. All tests were at 650°C under load control using sine wave loading form. Tests were run at an R ratio of 0.1 and a frequency of 0.1 Hz. After testing the fracture surfaces were examined optically and in many cases by scanning electron microscopy.


RESULTS AND DISCUSSION

Figure 1 shows the crack growth rates da/dN plotted against the stress intensity range $\Delta K$. To facilitate comparison of the results the crack growth rates at a $\Delta K$ value of 35 MPa√m are summarized in Table 2. To broaden the comparison the results from an earlier study(2) of crack growth in INCONEL alloy 718 are included in Table 2. These tests are run under identical test conditions to those used

Examination of Table 2 shows that some very significant changes in crack growth rates were achieved in the Astroloy and Waspaloy specimens in which the grain sizes and shapes were varied. Figure 3 shows a plot of the crack growth rates at $_\Delta K$ = 35 MPa√m versus the grain size. To a first approximation the data can be represented by a straight line for each environment with slope of -1 on a log-log plot. At all grain sizes the crack growth rates in air were about 2.5 times faster than in He.
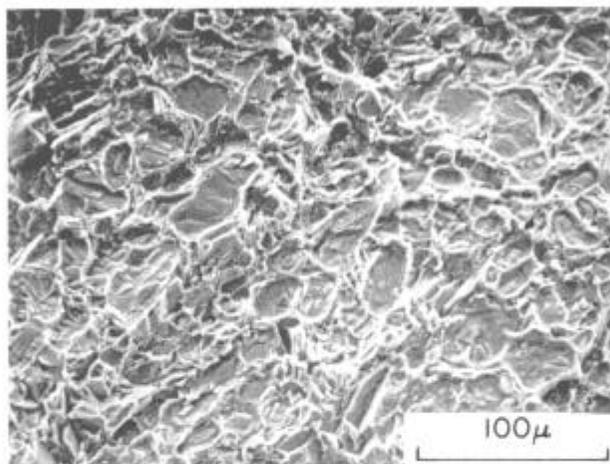


FIGURE 2:   Scanning electron micrograph showing typical facetted appearance of transgranular fractures.

Several specific comments must be made about these results. In the case of the Astroloy with serrated grain boundaries, Code C, no marked improvement in crack growth resistance was observed, compared to materials of comparable grain size. Since crack growth was transgranular this lack of any effect of the serrations is not surprising. In the case of the Waspaloy-2 samples with a duplex grain structure, the results are plotted in terms of the small sized grains. In both Waspaloy-2 and Waspaloy-3 samples there was no effect of longitudinal versus transverse sample orientations. The data for the remaining alloys in Table 2 do not line up with the Astroloy and Waspaloy data in Figure 3. Generally the crack growth rates for the other alloys are significantly faster than would be predicted from Figure 3.

Other investigators have noted that coarse grained alloys can show lower crack growth rates in ambient temperature fatigue tests. Of particular interest is a recent

study by Lindigkeit et al(12) of age hardened Al and Ti
alloys. Coarse grained materials showed lower da/dN values,
and the fracture surfaces displayed the flat, faceted
appearance that was very similar to the fractures exemplified
by Figure 2. Lindigkeit et al concluded that in alloys that
display a high degree of planar slip a coarser grain size
would minimize slip activity on secondary slip systems, thus
allowing a greater degree of reversibility of deformation
ahead of the crack tip under cyclic loading and hence a
reduced rate of crack growth. A mechanism of this type is
a plausible explanation for the present results since slip
in superalloys at intermediate temperatures is generally
planar and localized. The enhanced crack growth rates in
air versus helium might also be related to slip reversibility;
i.e., air might impair reversibility by oxidation damage in
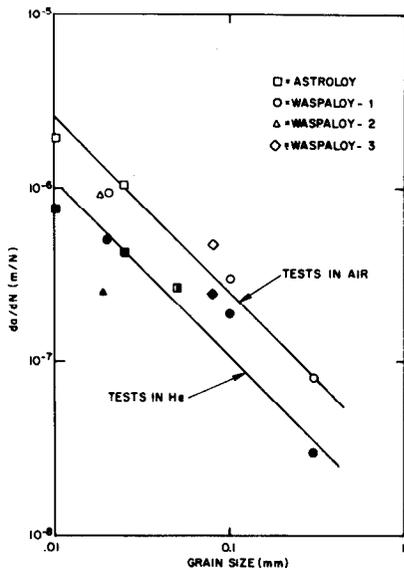the crack tip region.



FIGURE 3-CRACK GROWTH RATE AT ΔK=35 MPa√m VERSUS GRAIN SIZE.

     Other structural variables besides the grain size
influenced the behavior. In the case of Alloy X-750
the introduction of a coarse grain boundary $M_{23}C_6$ carbide
plus $\gamma'$ denuded zone by the intermediate heat treatment
(Code F) caused the crack path to change to intergranular
and produced a substantial increase in crack growth rate
over the material aged only at 705°C (Code G). Intuitively
one might expect the microstructure produced by the Code F

heat treatment to be deleterious, but it must be noted that
this deleterious effect was seen only when the samples were
tested in air. When tested in helium the two heat treatments
gave identical crack growth rates. Thus it would appear
more correct to say that the Code F heat treatment made the
alloy much more susceptible to the deleterious effects of
the air environment.

Changes in the carbide morphology at the grain boundaries
did not always affect the fatigue behavior. This lack of
sensitivity to microstructure is illustrated by the results
for NIMONIC alloy 115. In this case the two microstructures,
Codes D and E, gave identical crack rates in each environment.

The earlier tests on alloy 718 show that overaging is
beneficial. Once again it should be noted that the environ-
ment may play a significant role. When tested in helium the
alloy 718 results showed that the effects of heat treatment
were reversed; i.e., the crack growth rate was slightly
higher in the overaged condition(2). Thus here also it may
be more accurate to say that the principal effect of the
heat treatment was to change the sensitivity to the air
environment.

These results with alloy X-750 and alloy 718 indicate
that microstructural effects observed in helium can be
radically changed when the tests are conducted in air.
However, the general effects of grain size were similar in
both environments, with air producing about a 2.5 fold
increase in crack growth rate at all grain sizes. Changing
the environment evidently may produce either abrupt changes
in behavior, or fairly continuous effects. It is noteworthy
that the cases where abrupt changes occurred involved
significant amounts of intergranular crack growth, while
continuous changes in growth rate were observed under
conditions of transgranular cracking. In general one might
expect that environmental effects would be most pronounced,
and perhaps most unpredictable, when the crack growth was
intergranular.

Thus the optimum microstructure may depend upon the
environment. Examination of fatigue crack growth of alloy
718 in a number of different environments showed that
oxygen was the aggressive component in air(11). Sulfur
containing environments were more aggressive than air and,
in fact, under sulfidizing conditions, oxygen was found to
help retard the effects of sulfur. Alloys that were developed
by testing in air may perform well as long as oxygen is the
dominant species influencing cracking. It is not at all

certain however, whether microstructures optimized by testing in air would offer the best resistance to sulfurizing or other aggressive environments.

## CONCLUDING REMARKS

The present results show that the fatigue crack growth rates can be influenced significantly by the microstructure. Coarser grain sizes markedly retarded the cracking rates. This effect of grain size seems best explained by the role of grain size in promoting slip on secondary systems in the region ahead of the crack tip.

As far as tests in air are concerned, overaging has often been found to be beneficial. Changing the slip behavior within the grains, or changes in the grain boundary microstructure have been suggested as the reasons for the improved resistance to crack growth. Just what specific heat treatments or specific microstructures are best is not generally known, and considerably more work needs to be done to detail the structure-property relationships.

If the microstructure is optimized to lower the fatigure crack growth rates, other properties may be adversely affected. Fatigue crack initiation probably will be enhanced by coarser grain sizes, and the creep strength generally will be lowered by coarser grain sizes and overaged microstructures. Microstructure must be optimized for the total spectrum of properties desired in service.

The environment is a significant variable at intermediate temperatures. Microstructures that are beneficial in one environment can be detrimental in another. Structures optimized by testing in air probably will remain effective as long as oxygen is the dominant aggressive element. When other species predominate there is no certainty that such microstructures will be effective.

## REFERENCES

(1) H. F. Merrick and S. Floreen, Met. Trans. 9A, 1978, p. 1095.
(2) S. Floreen and R. H. Kane, Fatigue in Engineering Materials, in press.
(3) H. H. Smith and D. J. Michel, Naval Res. Lab., Memo Rep. 3810, Office of Naval Resarch, Arlington, VA, 1978.

(4) W. J. Mills and L. A. James, ASME Paper No. 78 -
    WA/PVP-3, ASME, NY 1978.
(5) M. Calvel and A. Pineau, Met. Trans. 9A, 1978, p.
    471.
(6) M. O. Speidel, Proc. Symp. on High Temp. Matl's. in
    Gas Turbines, Elsevier Sci. Pub. Co., New York, 1974, p.
    207.
(7) S. Floreen, AIME Symposium on Creep-Fatigue-Environment
    Interactions - in press.
(8) J. M. Larson and S. Floreen, Met. Trans. 8A, 1977, p.
    51.
(9) D. M. Ward, results summarized in R. F. Decker, Steel-
    Strengthening Mechanisms, Climax Moly. Co., CT, 1969.
    p. 137.
(10) E. L. Raymond, Trans. AIME 239, 1967, p. 1415.
(11) S. Floreen and R. H. Kane, Met. Trans. 10A, 1979, p.
    1745.
(12) J. Lindigkeit, G. Terlinde, A. Gysler and G. Lutjering,
    Actas Met. 27, 1979, p. 1717.