# Environmental Damage of a Cast Nickel Base Superalloy

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Exposure in air in the temperature range 900 to 1100 °C produces a major loss in stress rupture life and ductility of IN738. The sensitivity to this environmental damage increases with decreasing test temperature in the range 1000 to 700 °C. Oxygen is identified as the source of the damage for air exposure and indirect evidence supports grain boundary penetration of the gas to considerable depth. It is argued that oxygen segregation can lead to grain boundary immobilization and unstable intergranular fracture at intermediate temperatures. It is shown that compositional modifications, particularly boron and hafnium additions, may reduce the oxygen damage susceptibility, and that a cobalt base coating effectively eliminates the susceptibility. The relevance of these observations in understanding the effect of test environment on creep-rupture and fatigue crack propagation is considered.

AWARENESS of the important influence of test environment on the deformation and fracture of high temperature materials has evolved from at least three superficially distinct streams of investigation. The first of these has been concerned with a comparison of creep rates and rupture lives in various gaseous environments. This work has covered a broad range of nickel and iron-base alloys tested in environments ranging from vacuum and inert gases to hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, and air. Two recent reviews[1,2] of these studies reveal both the variety of gas/metal reactions which may occur and also the absence, at present, of a useful framework on which life predictions may be made.

Of special significance is the study by Shahinian and Achter[3] on creep rupture of nickel in air and vacuum. They found the effects of environment to depend strongly on stress. At high stresses, rupture lives were longer in vacuum; whereas the reverse was true at low stresses. The transition stress was similar at the two temperatures studied. Strengthening in air at low stresses (longer lives) was attributed to internal oxidation, and weakening was associated with enhanced grain boundary cavitation, although the factors controlling the transition stress were not considered in detail. Similar effects were subsequently reported in several complex alloys.[4] However, the influence on rupture lives and creep rates was not large relative to normally encountered heat to heat variations. In fact, exposures in oxidizing, carburizing, nitriding, and reducing environments, in general, have rarely been shown to influence these properties by more than a factor of 3.[1,2]

More dramatic effects have been reported on rupture life of notched bars[4] and thin sections[5] and on tensile ductility of nickel base alloys tested in air and argon.[6] In the latter study, it appeared that the magnitude of the ductility minimum observed at about 850 °C was sensitive to the slip mode of the alloy; a heat treatment which produced fine $\gamma'$ and promoted planar slip accentuated the ductility minimum in air tests.

In oxygen containing environments, the frequent observation of enhanced cavitation implies either that actual penetration of the gas in some form is involved or that internal voids grow in response to external oxidation. A time-dependent process of damage penetration is supported by tests in which the oxygen partial pressure was suddenly increased; the associated increased creep rate did not occur instantaneously but required a lapse of several hours.[7] This observation implies that the generally small effects of environment reported in laboratory creep tests may not give an accurate indication of the effect on service lives if damage is time dependent.

A similar problem exists in the second stream of investigation of environmental effects which has studied the influence of environment on fatigue. A review of some of the extensive literature on this subject is given in Ref. 2. As with creep, both air strengthening and vacuum strengthening have been reported, although generally, as with Snowden's work on lead,[8] a dramatic loss in endurance results when testing in air or oxygen relative to vacuum. Reducing the oxygen partial pressure during fatigue crack propagation in stainless steel at 800 °C was shown by Smith *et al*[9] to lead to a lower crack growth rate after about 1 h. During this time, the crack grew about 80 $\mu$ which was taken to imply oxygen penetration to this depth ahead of the crack tip.

Detailed studies of the influence of cyclic frequency on fatigue lives of a nickel base superalloy by Solomon and Coffin[10] and Woodford and Coffin[11] at 538 °C showed that most of the loss in fatigue life with decreasing test frequency was due to environmental influence rather than time dependent creep damage processes. The major unanswered question is whether the hold-time/frequency effects saturate, which might be expected on an oxygen penetration model. Indirect support for saturation derives from thermal fatigue testing[12] and from air *vs* vacuum tests over a range of temperature.[13] Saturation of the frequency effect due to test environment might be expected when damage has penetrated to a depth equal to the maximum crack growth

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Manuscript submitted May 22, 1980.

ISSN 0360-2133/81/0211-0299$00.75/0

increment possible on the next cycle for crack propagation through the damaged material.

The third stream of investigation is the least extensive but may well be that which leads to a confluence since it addresses directly the question of penetration of the damaging species. Douglass[14] and Hancock[15] have shown that the creep rupture life and tensile ductility of nickel are drastically reduced by prior exposure in air. Hancock[15] has attributed this damage to vacancy injection and associated internal void formation. He also reported an initial decrease in creep rate following prior oxidation. Thus, there was a strengthening effect due to either the presence of an oxide film or to internal oxidation, together with an embrittling effect supposedly resulting from enhanced cavitation at preexisting voids. Although a time dependent oxidation damage occurred, in this work it was not attributed directly to internal penetration of oxygen.

The practical importance of these observations was confirmed by the detailed study of Chang[16] on the effects of prior air exposure on tensile ductility of a nickel base superalloy, René 80. He found a pronounced embrittlement for prior exposure at 982 °C with a maximum loss in tensile ductility at about 850 °C in subsequent tensile testing. It was shown that grain boundaries were necessary for embrittlement by comparison with results for the same alloy in the directionally solidified condition, that the depth of damage was an order of magnitude greater than the oxide scale thickness, and that protective coatings substantially reduced the embrittlement. These effects have been confirmed in several other conventionally cast superalloys.[17]

For air exposure, therefore, it appears that a time dependent damage results from either direct penetration of a gaseous species or indirect void formation in response to the growing oxide scale. Characterization of the phenomenology of this damage and identification of the mechanism from a study of preoxidized specimens should therefore provide a basis for a framework on which to interpret and extend studies of fatigue and creep crack propagation in gaseous environments.

This paper reports the results of an extensive study of the effects of prior exposure in various gases on creep rupture of a cast nickel base alloy, IN738. The results are typical of those obtained on a variety of similar alloys.[18,19] The main emphasis is on the phenomenology, although some important observations on the mechanism of damage are reported. A more detailed mechanistic study is currently in progress on a number of simpler alloys. Some of these results and their implications, where applicable to the phenomenology of environmental embrittlement of IN738, will be incorporated in the discussion.

## EXPERIMENTAL PROCEDURE

Most of the specimens were taken from a 2.5 cm × 12.5 cm × 20 cm casting of IN738. Prior to machining, the slab was heat treated at 1120 °C for 2 h in vacuum followed by an aging treatment at 840 °C for 24 h in vacuum. Specimen slugs were electrodischarge machined and ground to size with a reduced gage section of 9.53 mm and gage diam of 2.54 mm. For the prior exposures, specimens were heat treated in flowing air, oxygen or nitrogen, or they were vacuum encapsulated. The specimens for vacuum exposure were first baked out at about 350 °C before sealing the capsule in a vacuum of $1.3 \times 10^{-2}$ N/m$^2$.

Small experimental castings of several modifications of IN738 were also tested in this study. These included IN738 with additions of yttrium and hafnium, and one heat with boron replacing the carbon. In addition, to examine the effect of purity, a special heat was made with higher purity Cr, C, Ti, Zr and B additions, and this heat was repeatedly purged with argon prior to final pouring in argon. These heats were poured into 20 mm × 60 mm × 75 mm graphite molds and then heat treated as before. Twelve specimens were prepared from each of these ingots for testing in the as heat treated condition and after exposure in air and vacuum. The chemical compositions of all the heats and identifications are given in Table I.

A final series of experiments studied the effect of several surface coatings. The coatings were deposited on the specimen gage length, after grit blasting and cleaning the surface, using a vacuum plasma spray technique initially developed by Muehlberger.[20] With proper processing control, this technique can be used to deposit highly dense (98 to 100 pct), nearly oxide-free coatings. The coating compositions in weight percent were Co-29Cr-6Al-1Y, Ni-20Cr-10Al-2Hf-0.1C, and IN738. The first two were selected as typical cobalt and nickel-base environmentally protective coatings, and the IN738 powder was included to examine the effect of surface microstructure on embrittlement. Coating thicknesses were between 100 and 150 $\mu$. However, all stresses were based on the uncoated cross section area.

Most of the rupture tests were performed in air because of machine availability, although some tests

**Table I. Chemical Composition, Wt. Pct**

| Alloy | Ni | Co | Cr | Al | Ta | C | W | Mo | Ti | Nb | Zr | B | HF | Y |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| IN738* | Balance | 8.3 | 15.78 | 3.43 | 1.69 | 0.1 | 2.6 | 1.74 | 3.49 | 0.81 | 0.06 | 0.011 | — | — |
| IN738 + Hf | Balance | 8.5 | 16 | 3.5 | 1.8 | 0.1 | 2.6 | 1.8 | 3.5 | 0.9 | 0.05 | 0.01 | 1.5 | — |
| IN738 + Y | Balance | 8.5 | 16 | 3.5 | 1.8 | 0.1 | 2.6 | 1.8 | 3.5 | 0.9 | 0.05 | 0.01 | — | 0.5 |
| IN738 + B | Balance | 8.5 | 16 | 3.5 | 1.8 | 0.1 | 2.6 | 1.8 | 3.5 | 0.9 | 0.05 | 0.1 | — | — |
| IN738-HP† | Balance | 8.5 | 16 | 3.5 | 1.8 | 0.1 | 2.6 | 1.8 | 3.5 | 0.9 | 0.05 | 0.01 | — | — |

* The standard heat was chemically analyzed. All others are nominal compositions.

† IN738-HP is high purity heat with iodide Cr, Ti and Zr, spectroscopically pure C, and 99.8 pct B.

were performed in argon. Except at the lowest temperature, the test environment had little effect on rupture lives during the period of the test. In fact, as discussed subsequently, this observation helped to identify a basic conceptual error in the interpretation of previous studies of environmental influence on creep rupture behavior.

Environmental embrittlement was manifested both by loss in rupture life and reduction in ductility. The former was found to be affected in a more systematic manner and is emphasized in this paper. However, considerable scatter does occur in rupture life, as might be expected for an embrittling phenomenon. This scatter was not studied specifically for all the exposures but should be kept in mind when examining curves drawn through a limited number of data points.

## RESULTS

### i. Effect of Prior Exposure on Stress-Rupture

Stress rupture tests were conducted at 700, 800, 900 and 1000 °C at stresses of 620, 448, 255 and 100 MPa respectively. The results could therefore be represented conveniently on a Larson-Miller parametric plot, as shown in Fig. 1. The rupture times for the individual test points are given in the figure to indicate the dramatic loss in life associated with prior exposure in air for 200 h at 1000 °C. It is also clear that the sensitivity to the damage increases with decreasing temperature in the range studied.

Figure 2 shows the effect of exposure for 200 h at 1000 °C in oxygen, nitrogen, and vacuum. The data points are shown in comparison with the curves reproduced from Fig. 1. It is clear that exposure in oxygen has the same effect as air exposure. By contrast, nitrogen and vacuum exposure produce only a modest life reduction relative to the unexposed reponse. This degradation is reduced for specimens which were exposed in vacuum before machining. It is believed, therefore, that the degradation is partly due to microstructural changes associated with the thermal exposure and partly due to the vacuum and nitrogen not being totally inert. It does seem probable, however, that oxygen is responsible for the dramatic loss in life in air exposed specimens.

Figure 3 illustrates the effect of all these exposure conditions at the four temperatures. This figure also demonstrates that testing in argon did not significantly change the rupture lives of the unexposed specimens, except at 700 °C.

To examine the kinetics of embrittlement, specimens were exposed in air for various times at temperatures of 900, 1000, and 1100 °C, and then tested at 800 °C and 400 MPa. These were not the most sensitive test conditions, but were chosen to ensure some life after embrittlement. The results in Fig. 4 show a progressive and reasonably systematic life reduction from an average of 200 h in the unexposed condition. Of special note is the substantial scatter after some embrittlement. For example, rupture lives between 0.24 and 106 h were measured after exposure at 900 °C for 100 h. This scatter is also reflected in the measured elongation as shown in Fig. 5, and, as stated previously, the time to rupture is a more sensitive indicator of damage. For a given prior exposure, there was no correlation between ductility and rupture life.

An attempt was made to determine the depth of damage after air exposure by testing specimens with diameters reduced by machining off progressively greater amounts after air exposure at 1000 °C for 100 h. These tests were run in argon at 700 °C and 620 MPa, and are compared with a series of unexposed specimens. Unfortunately, significant losses in rupture life (and probably increased scatter) were encountered in the unexposed control samples. This is, in part, due to the well known reduction in rupture life of conventionally cast superalloys with decreasing section size.[21] However, it is also important to recognize that the rupture life at these test conditions is extremely sensitive to environmental damage. It is likely, therefore, to be particularly sensitive to the presence of metallurgical defects and to surface preparation. This is reflected in the variability in rupture lives depicted in Table II. Nevertheless, the results are consistent with damage having penetrated beyond a depth of 0.75 mm. This conclusion

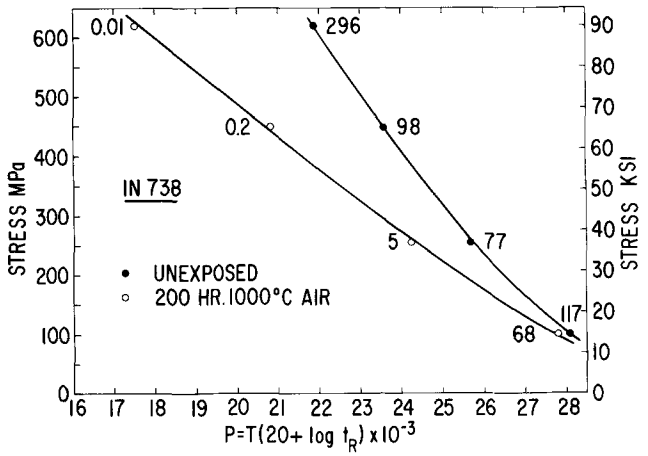



Fig. 1—Effect of air exposure on rupture life. Individual lives are indicated in hours for tests at 700, 800, 900 and 1000 °C.

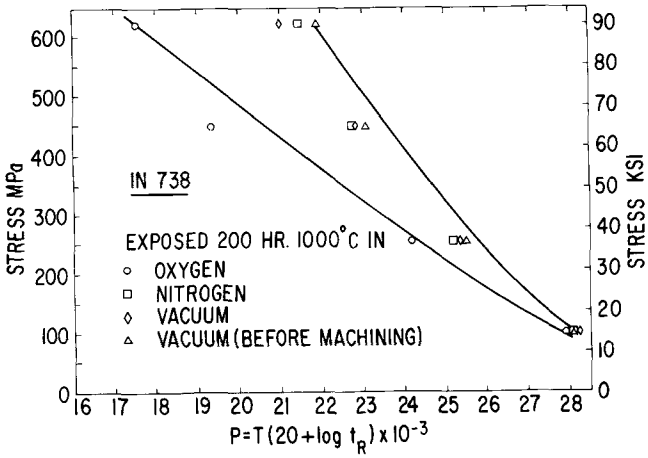



Fig. 2—Effect of exposure environment on rupture. Curves are reproduced from Fig. 1.

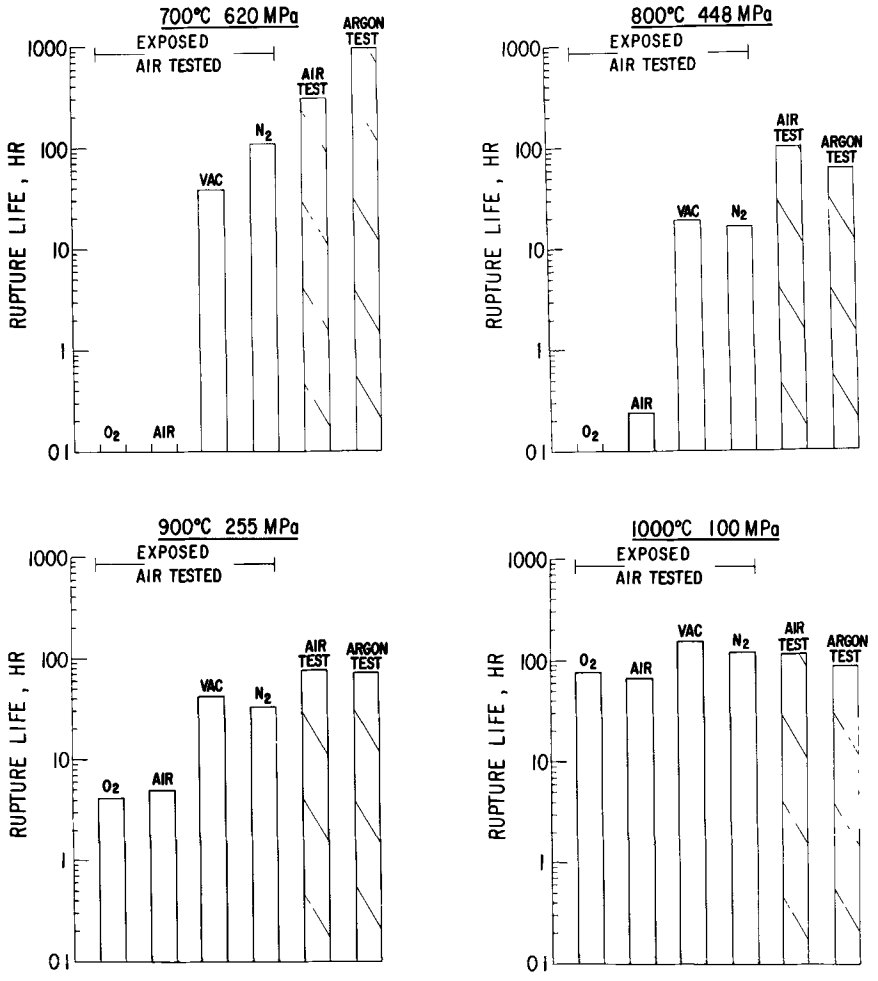



Fig. 3—Effect of exposure environment on rupture life at the four test conditions. All exposed specimens were tested in air.

is probably very conservative since exposure at 900 °C for 100 h in pure nickel damages at least to a depth of 1.25 mm.[22]

### ii. Composition Modifications

For the various composition modifications, the effect of air exposure for 200 h at 1000 °C is shown on the parameter plot of Fig. 6. The curves for the standard heat from Fig. 1 are included for comparison. Figure 7 shows the measured elongations for all these data plotted on a stress scale to allow an overall assessment of the major effects on ductility. There are several important points to note in relation to these two figures:

1) At the higher stresses in the unexposed condition, there is a significant increase in ductility for the boron, hafnium, and high purity modifications. This is in part responsible for the increased rupture life at 620 MPa and 700 °C.

2) All modifications show appreciable loss in life after exposure, but the boron and hafnium modifications are least sensitive to this damage and some of their loss may be due to microstructural changes associated wih the heat treatment.

3) The high purity heat in the unexposed condition may have a shorter rupture life than the standard IN738 at high temperatures, and the improved ductility at lower temperatures (higher stresses) is not retained after air exposure.

4) The yttrium addition decreases the high-temperature rupture lives in specimens with and without prior air exposure.

The beneficial effect of boron and hafnium in decreasing the environmental embrittlement has been confirmed in several other alloys.[18,19] It is apparent that, although both modifications produce higher rupture

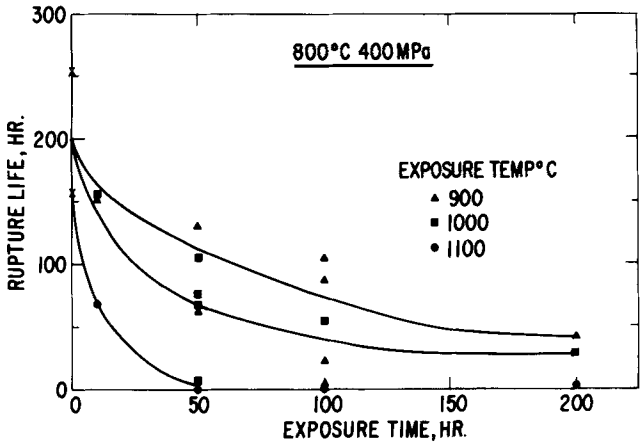



Fig. 4—The effect of time and temperature of air exposure on rupture life. Crosses are rupture lives of duplicate unexposed specimens.

**Table II. Effect of Surface Removal on Stress-Rupture in Argon at 700 °C and 620 MPa**

| | As Heat Treated | | | Exposed 100 h −1000 °C | | |
|---|---|---|---|---|---|---|
| Gage Diameter (mm) | $t_R$ | El Pct | RA Pct | $t_R$ | El Pct | RA Pct |
| 2.41 | 223 | 6 | 5.5 | 2.9 | 4.0 | 4.4 |
| 2.03 | 622 | 4.4 | 3.9 | 13.7 | 3.8 | 8.6 |
| 1.52 | 2.3 | 2.2 | 2.3 | 16.5 | 2.4 | 5.6 |
| 1.02 | 143 | 4.7 | 2.1 | 0.6 | 2.7 | 7.8 |

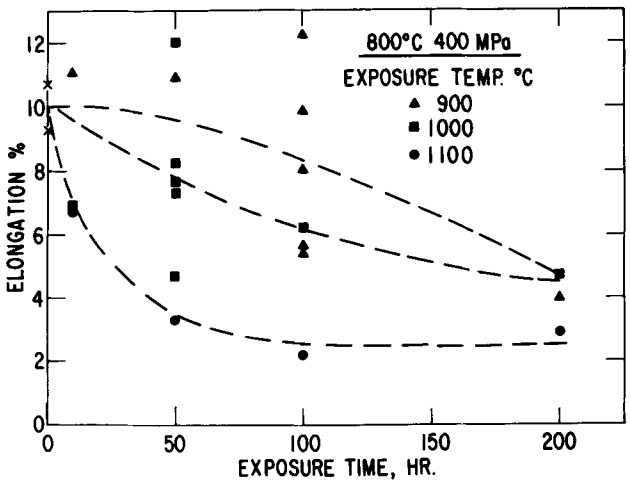



Fig. 5—The effect of time and temperature of air exposure on elongation at the indicated test conditions.

ductility in the unexposed condition, an intrinsic increase in ductility cannot alone explain the reduced environmental embrittlement. The high purity IN738, despite its impressive unexposed ductility, has rupture lives and elongations comparable to those of the standard IN738 after exposure.

### iii. Effect of Coatings

Figure 8 shows that the rupture lives of specimens coated with Co-29Cr-6Al-1Y and then exposed at 1000 °C for 200 h are not affected by air *vs* vacuum exposure; the damage is prevented by this coating. The small loss in life relative to the curve for bare unexposed specimens is, based on Fig. 2, attributed to microstructural changes associated with the temperature exposure.

The results for the other two coatings are compared in Fig. 9 after air exposure. Although vacuum-exposed control tests were not included, the data do indicate that there may be some environmental embrittlement of specimens coated with the nickel-base alloys. There is too much scatter in the ductility results shown in Fig. 10 for them to lend real support to this. However, there is little reason to believe that a coating of IN738 on IN738 should prevent embrittlement. The fine grain size of the coating may, however, ameliorate the situation either by its intrinsically higher ductility or by serving as a sink for oxygen atoms because of the higher grain boundary surface area.

It is worthwhile noting in Fig. 10 that the Co-29Cr-6Al-1Y coated specimens have very similar elongations for air and vacuum exposure.

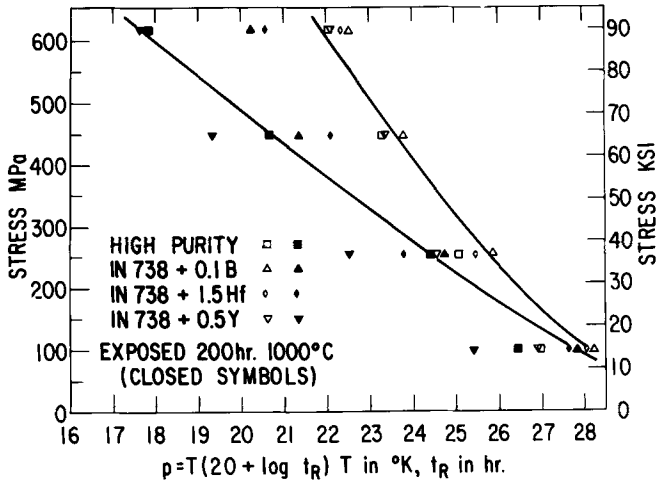



Fig. 6—Effect on rupture life of exposure in air at 1000 °C for 200 h for the four alloy variations. The curves are reproduced from Fig. 1 for the standard IN738.

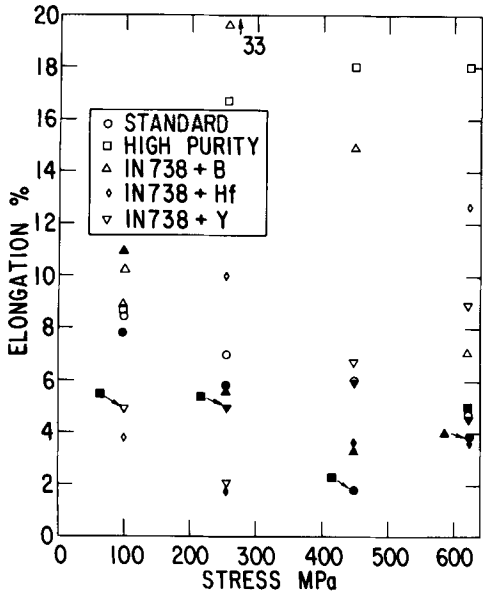



Fig. 7—Effect on elongation of exposure in air at 1000 °C for 200 h as a function of applied stress. Filled symbols are exposed specimens.

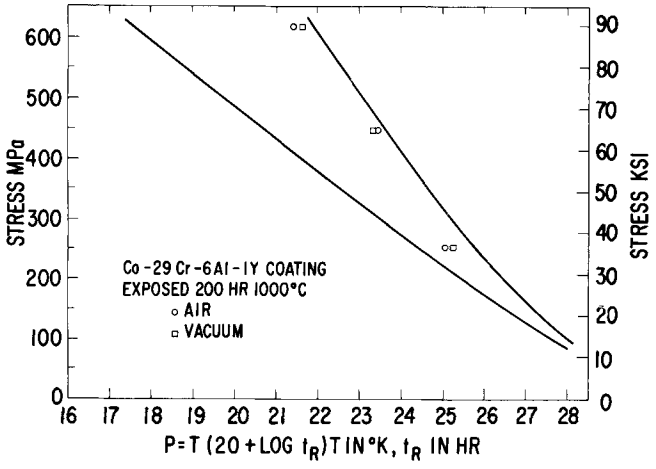



Fig. 8—Elimination of environmental damage by application of a Co-29Cr-6Al-1Y coating. Curves are reproduced from Fig. 1.

### iv. Metallography

Extensive metallography on these alloys was not especially helpful in clarifying the mechanisms of environmental damage. Fracture of these cast superalloys is principally intergranular (or interdendritic transgranular, which is not readily distinguishable) even in the unembrittled state. However, for the standard IN738 there were generally some regions of flat crystallographic fracture in the undamaged state. For example, Fig. 11(*a*) shows a near surface transgranular segment in a specimen exposed at 1000 °C in vacuum prior to testing at 800 °C, compared in Fig. 11(*b*) to a totally intergranular fracture and secondary intergranular cracking for a specimen exposed in oxygen. These differences were confirmed in scanning electron microscopy of the fracture surfaces. For example Fig. 12 compares the fracture appearance of two undamaged

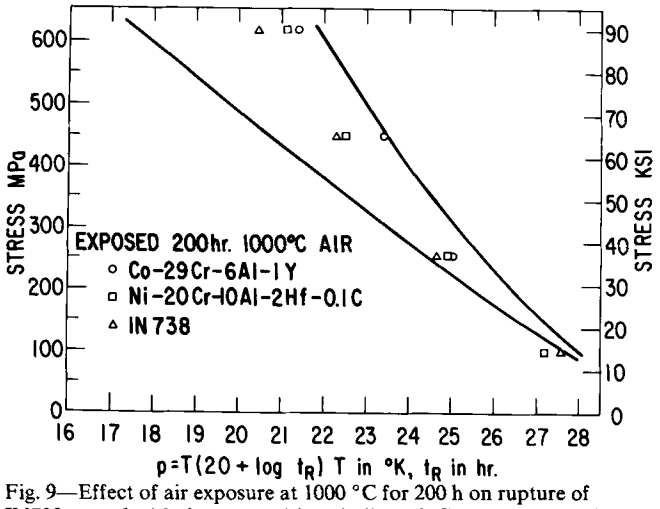



Fig. 9—Effect of air exposure at 1000 °C for 200 h on rupture of IN738 coated with the compositions indicated. Curves are reproduced from Fig. 1.

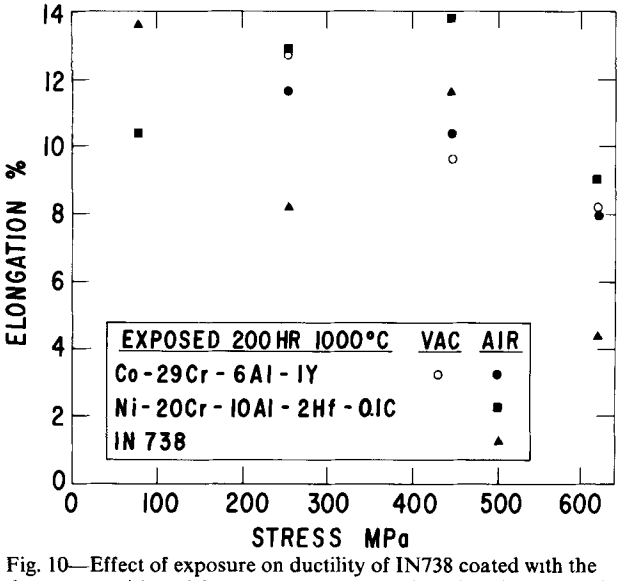
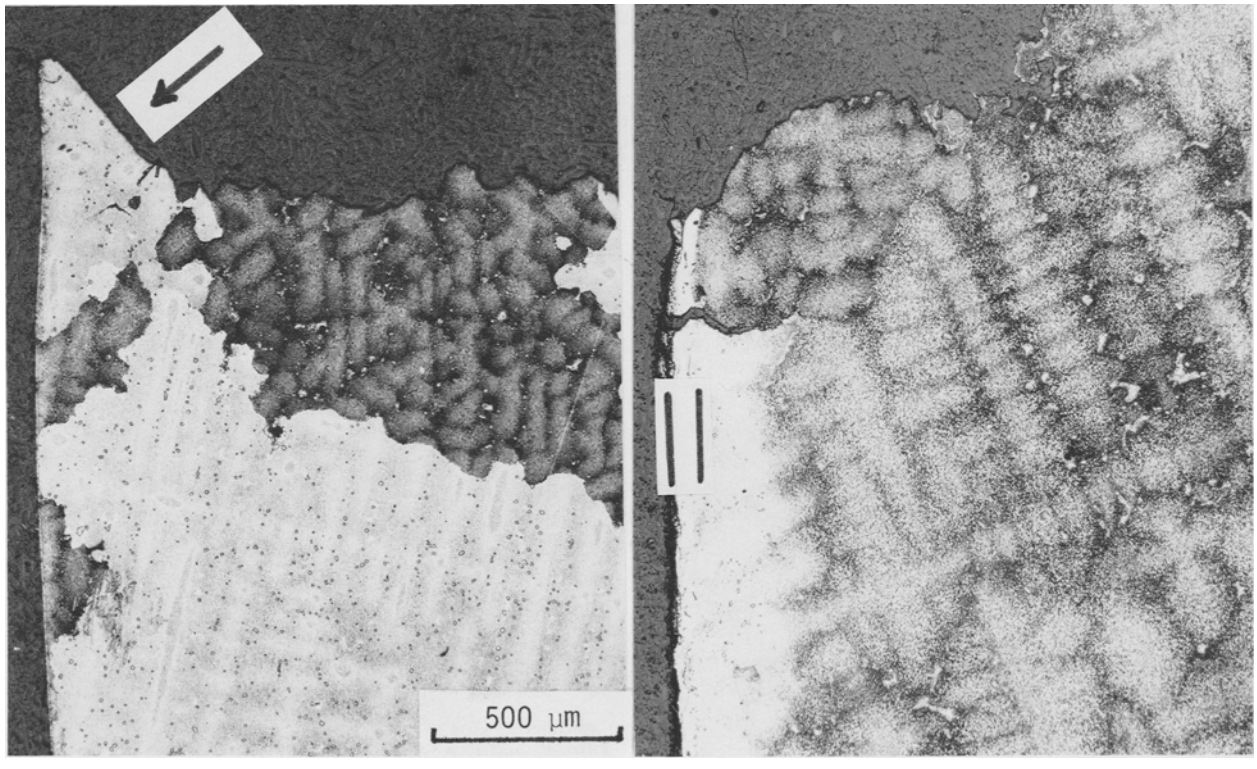
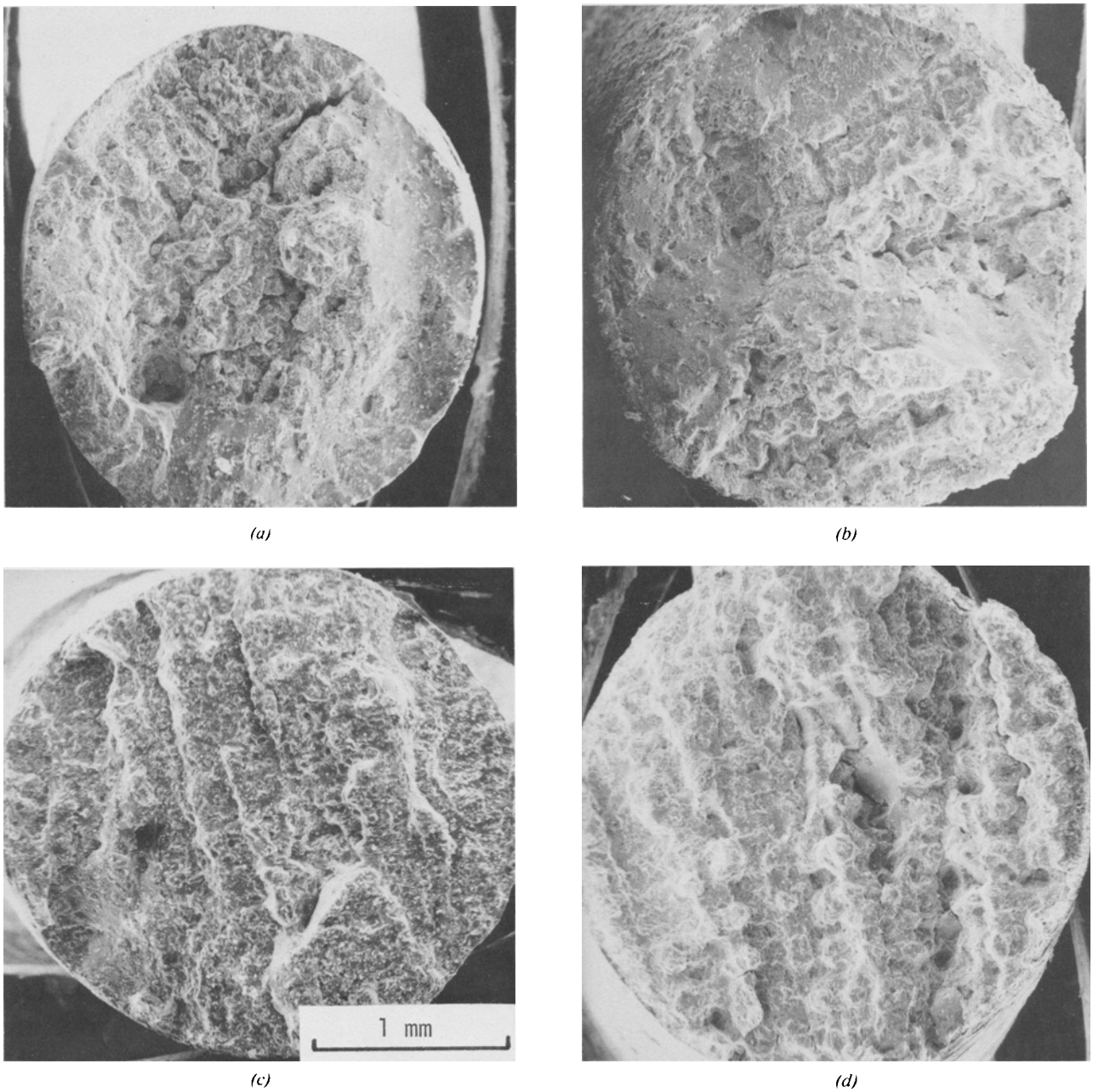



Fig. 10—Effect of exposure on ductility of IN738 coated with the three compositions. The measurements are plotted against applied stress.




(*a*) (*b*)

Fig. 11—Longitudinal sections of standard IN738 after rupture at 800 °C and 448 MPa. (*a*) vacuum exposed at 1000 °C showing a region of transgranular failure; (*b*) air exposed at 1000 °C. Fracture is entirely intergranular. Note also narrow precipitate-denuded region beneath oxide scale.




(a) (b) (c) (d)

Fig. 12—Scanning electron fractographs of standard IN738 after rupture at 800 °C and 448 MPa following exposure at 1000 °C for 200 h, (*a*) vacuum exposure showing some transgranular regions; (*b*) coated with Co-29Cr-6Al-1Y and air exposed showing some transgranular regions; (*c*) air exposed—intergranular; (*d*) oxygen exposed—intergranular.

specimens (vacuum exposure and air exposure of coated specimen) with that of two damaged specimens (air exposure and oxygen exposure). In the first two specimens some flat transgranular regions are visible, whereas the other two have totally intergranular (or interdendritic fractures).

The general connection between embrittlement and fracture appearance is confirmed in Fig. 13. In the unexposed condition, extensive smooth transgranular fracture is apparent in the boron and hafnium modified heats. Some of this is even present in the boron alloy after exposure. The material modified with yttrium is totally intergranular for both conditions, reflecting the generally deleterious effect of yttrium (Figs. 6 and 7).

Apart from a thin oxide scale and a shallow surface region (approximately 45 $\mu$m for 200 h air exposure at 1000 °C) denuded in $\gamma'$ precipitate, there were no discernible differences in the microstructure between damaged and undamaged specimens for exposures at 1000 °C. To determine whether the damage might be on too fine a scale at this temperature, some specimens were examined after exposure at 1100 °C. After 200 h in air there appeared to be some voids near the surface similar to those reported by Hancock[15] and attributed to vacancy injection from the growing oxide scale. To confirm this observation, one specimen was held for 1000 h in air at 1100 °C and, indeed, as shown in Fig. 13, voids clearly developed to a depth of approximately 400 $\mu$m.

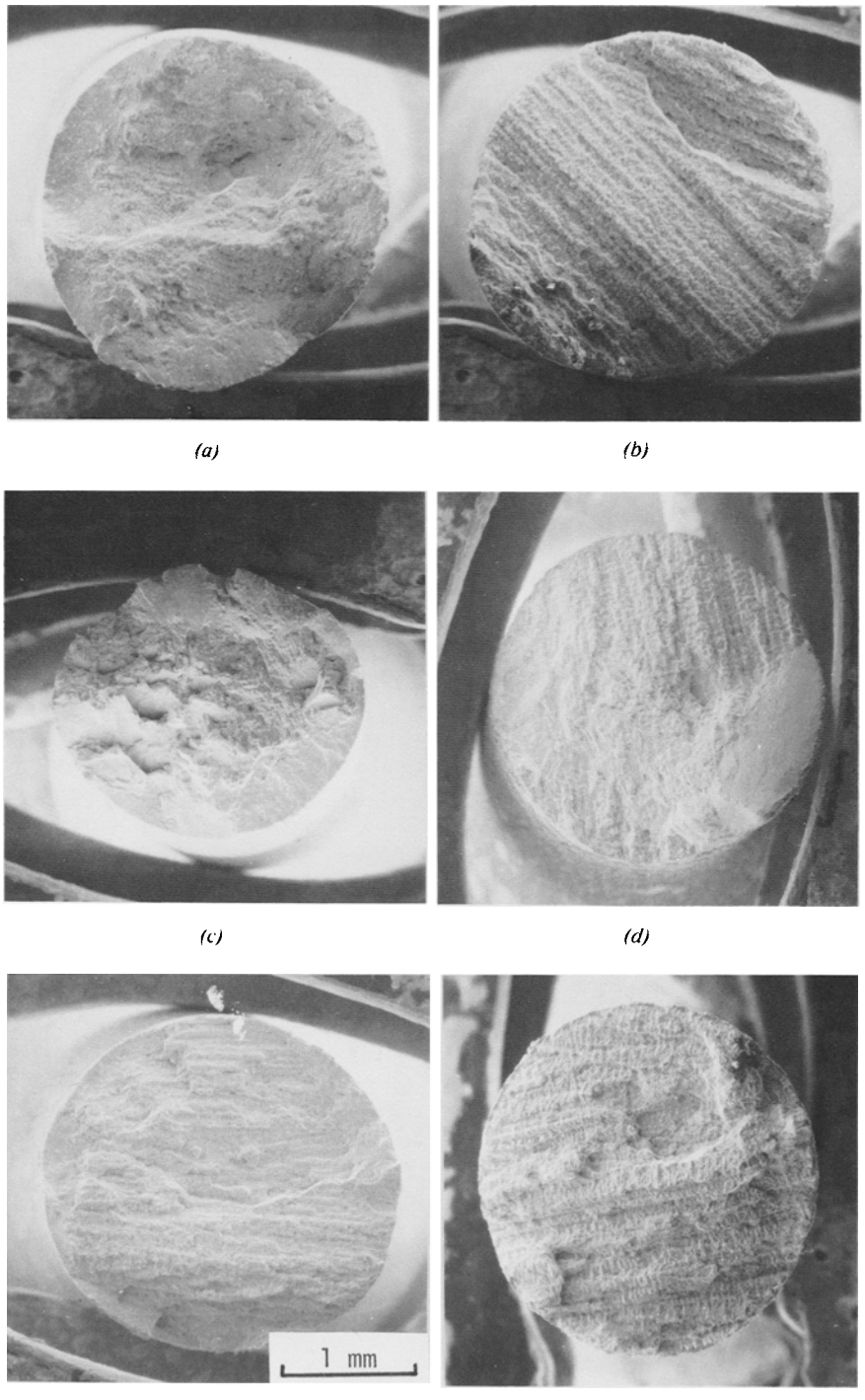



Fig. 13—Scanning electron fractographs of IN738 modifications after rupture at 700 °C and 620 MPa. IN738 + Hf: (*a*) unexposed; (*b*) air exposed, 1000 °C. IN738 + B: (*c*) unexposed; (*d*) air exposed, 1000 °C. IN738 + Y: (*e*) unexposed; (*f*) air exposed, 1000 °C.

## DISCUSSION

Although the loss in rupture life after exposure in oxygen containing environments is dramatic, it should be recognized that the exposure and testing sequence reported here is not simulative of any current or proposed work cycle for cast superalloys. Thus, although some loss in rupture strength and ductility may occur after long time service, there is no evidence that environmental damage in this sense is a cause for alarm. However, there are a number of important consequences of the reported observations related in general to environmental effects on fracture.

Figure 4 shows that, at least in the case of air exposure, increasing temperature and increasing time result in increased damage. By contrast, the sensitivity to this damage increases with decreasing temperature between 1000 and 700 °C as illustrated in Figs. 1, 2, 3, and 6. Thus in tests of undamaged specimens in various environments, some damage would occur at high temperatures, but sensitivity to the damage is low and the measured effect should be small; at low temperatures,

the sensitivity to damage may be high, but the extent of damage during the period of a laboratory creep test would be low, and again the measured effect should be small. In fact, Fig. 2 suggests that the test environment (air *vs* argon) for the undamaged state does have a significant effect only at the lowest test temperature.

The important point is that the failure to recognize this result could mean that a major environmental effect on creep and creep rupture would only show up in a long time service exposure at lower temperatures. It is necessary, therefore, to recognize that the time dependence of environmental damage may be quite different from the time dependence of creep. Extrapolation of short time data could be seriously in error.

A second important consequence of this work is that, at moderate temperatures where low cycle and thermal fatigue are design limiting phenomena, small scale damage penetration could be expected ahead of a growing crack. As described in the introduction, many studies have demonstrated the importance of test environment on fatigue crack growth. Smith *et al*,[9] in particular, showed that the crack growth rate could be changed intermittently by changing the oxygen partial pressure. The difficulty in quantifying these environmental effects on fatigue is the sensitivity of the growth rate to the subtleties of the imposed cycle in terms of temperature, hold time, frequency, etc. In addition, there may be synergistic effects between environmental damage ahead of the crack and local creep deformation. Nevertheless, it is clear that the extent of damage needs to be far less for it to influence fatigue crack propagation than to affect creep-rupture.

A possible alternative approach to extrapolation, based on empirical rules currently used to predict time-dependent fatigue lives in aggressive environments is to consider crack growth in a predamaged specimen. This should ensure that the crack growth rate is a maximum for the particular material. There is, then, the possibility of using a lower bound fatigue crack growth rate in design. An additional intriguing aspect relative to fatigue is that alloys which show reduced sensitivity to environmental damage, such as the boron and hafnium modifications, may offer improved high temperature fatigue resistance. These concepts are currently under investigation.

The immunity to oxygen damage imparted by the Co-Cr-Al-Y coating is of major significance since this is a typical cobalt base coating used for protection against hot corrosion in gas turbine applications. It has been shown that this coating also prevents oxygen embrittlement in commerically pure nickel[22] and correspondingly prevents internal oxidation which otherwise occurs. Thus, prevention of embrittlement coincides with elimination of oxygen penetration. It is not yet known whether the nickel base coatings prevent oxygen penetration. However, on the basis of other work[18,19] it appears that cobalt base alloys may be intrinsically immune to oxygen embrittlement. Some damage indeed appears to have penetrated both nickel-base coatings, according to Fig. 9. Additionally, it should be recognized that the actual stresses were somewhat lower than indicated for the coated specimens because of the load-bearing cross section of the coating.

Although very long time air exposures cause voids to develop below the oxide scale (as shown in Fig. 14), this is not believed to be the principal cause of the oxygen damage. Careful scanning electron microscopy of grain boundary fracture surfaces of embrittled nickel[22] does not show voids extending anywhere near the known depth of damage. The most likely explanation is that oxygen, in atomic form, is present at the grain boundary at distances far in excess of those calculated on the basis of oxygen diffusion in nickel. This subject will be considered in detail in a subsequent publication.

The major remaining question relates to the mechanism of embrittlement by oxygen penetration of grain boundaries. Although grain boundary voids can clearly lead to the type of damage described here,[23–25] embrittlement by oxygen segregation is more difficult to explain. Although arguments related to grain boundary cohesion have been made for similar phenomena (*e.g.* Tipler and McLean[26] for antimony and oxygen embrittlement of copper), it is very difficult to relate fracture resistance quantitatively to cohesive strength.

An alternative idea stems from observations by Wood[27] on copper, and Martin and Parker[24] and Chaston[28] on silver. These authors observed that grain growth was suppressed when their specimens were exposed at high temperatures in air compared with observations of normal grain growth for vacuum exposure. They suggested grain boundary oxygen penetration to be the cause, and the depth of boundary immobilization was consistent with the depth of oxygen damage believed to be occurring in the present work. In fact, further work has shown that recrystallization is suppressed after air exposure of nickel.[22] Details will be reported subsequently.

A tentative hypothesis for environmental damage is, therefore, that grain boundary mobility is effectively reduced by solute segregation[29] such that stress concentrations associated with grain boundary sliding cannot be relieved by migration. This readily explains why, above a certain temperature, the embrittlement is reduced,[16] and also why fatigue life in air passes through

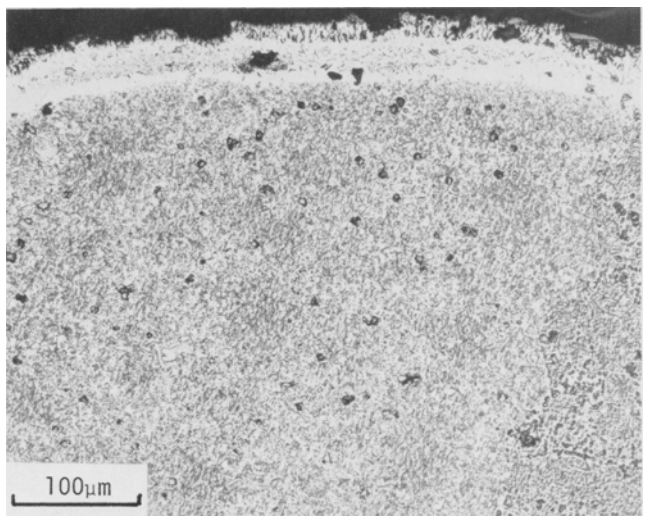



Fig. 14—Micrograph showing internal voids in standard IN738 after exposure in air for 1000 h at 1100 °C.

a minimum with increasing temperature;[13] even in the damaged condition, the grain boundary mobility is restored at very high temperatures. Clearly, this mechanism requires considerable development and elaboration. It does, however, have several attractive features. All currently available experimental findings are consistent with it, although it may prove difficult to make measurements of boundary mobilities in cast superalloys. In a general sense, it offers an interpretation of intrinsic embrittlement without environmental exposure. Grain boundaries may be immobilized by solute segregation from the interior or by precipitation. In such cases there may be no additional effect of environmental damage. Oxide dispersion strengthened alloys may fall in this category.[18]

## CONCLUSIONS

The phenomenology of rupture life degradation following high temperature exposure in air has been reported for a cast nickel base alloy, IN738, and some modifications thereof. The principal findings and deductions are:

1) Exposure at temperatures of 900 and 1100 °C in air leads to severe losses in rupture life and ductility with more degradation at lower temperatures in the range 700 to 1000 °C. This has important implications relative to extrapolation of creep and rupture lives for service applications.

2) The embrittlement is associated with oxygen penetration through grain boundary diffusion.

3) Chemistry modifications, in particular hafnium addition and boron substitution for carbon, substantially decrease the susceptibility to oxygen damage.

4) A cobalt base coating eliminates oxygen damage. This is believed to be a unique characteristic of cobalt alloys relative to oxygen penetration; nickel base coatings were less effective.

5) It is suggested that the mechanism of embrittlement may be related to decreased grain boundary mobility leading to premature intergranular fracture in the intermediate temperature regime.

6) The processes and phenomena described in this paper will occur on a much smaller scale at lower temperatures. They will have important effects on fatigue crack intiation and propagation. A possible approach to eliminate the need for extrapolation of fatigue lives in complex cycles with hold times is outlined. This may provide a lower bound high temperature fatigue design life.

## ACKNOWLEDGMENTS

Many helpful discussions have been held with M. G. Benz, M. R. Jackson, J. H. Wood and, in particular, R. H, Bricknell. The rupture tests were performed by C. Palmer and C. Canestraro, optical metallography by J. Methé, and scanning electron microscopy by M. Gill. Finally, I should like to thank J. R. Rairden for assistance with the coatings study.

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