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ABSTRACT

Evidence is presented that the hot-zone attack of nickel-base alloys in contact with fused fluorides occurs by a grain-boundary diffusion process. Predictions based on the grain-boundary diffusion model are shown to compare favorably with existing data. Certain anomalies are pointed out with regard to the corrosion of Inconel by Fuel No. 30 and tests which would lead to a better understanding of fused-salt corrosion are outlined.

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REFLECTIONS ON THE HOT-ZONE ATTACK OF INCONEL BY FUSED FLUORIDE

J. L. Scott

Scott and Prados¹ have recently shown that the distribution of attack in a fused-fluoride system under a temperature gradient is a function of the relative rates of the hot- and cold-zone mass-transfer processes. Thus, even in those cases where the mass transfer is cold-zone controlled, i.e., Fuel No. 30^x in Inconel^{xx} it is of importance to understand the mechanism and rate of the hot-zone process. In systems where the deposition of chromium occurs in the cold zone the hot-zone process is believed to be the rate-controlling step and is of obvious importance. This report gives evidence that the hot-zone attack occurs by a grain-boundary diffusion mechanism. Certain anomalies in existing data are described and a discussion of the future research needed to better understand the mass transfer process in fused fluorides is given.

Qualitatively, the mechanism of hot-zone attack of Inconel by fused fluorides is well understood. Manly et al² have shown that attack occurs by the preferential removal of chromium from the surface of the Inconel by the reaction:

The removal of chromium from the interface establishes a concentration gradient and chromium diffuses toward the interface by a vacancy mechanism. Since the rate of diffusion of nickel away from the interface is not as high as the rate of chromium diffusion toward the interface, an excess of vacancies develops in the matrix. When the lattice becomes supersaturated with respect to vacancies, the vacancies precipitate to form voids. This mechanism accounts for the observations that (a) voids are observed, (b) the metal adjacent to the interface becomes ferromagnetic, indicating the depletion of chromium, (c) chemical analyses show that chromium depletion occurs, and (d) tests have indicated that the voids are not interconnected. This type of diffusion, when observed by

^x 50 NaF-46 ZrF₄-4 UF₄ in mol per cent

^{xx} 17 Cr-5 Fe-bal Ni in weight per cent

previous investigators in other systems,^{3,4} has been interpreted on the basis of volume diffusion, but the theory does not preclude preferential diffusion at the grain boundaries.

Evidence for Grain Boundary Diffusion

Specimens which have been examined metallographically show that void formation has occurred preferentially at the grain boundaries. One can observe this effect in Fig. 1 which shows a sample of Inconel exposed to Fuel No. 30 for 1000 hr at 900°C. Note that the total depth of attack under these conditions is about nine mils.

The depth of attack which is computed on the basis of a volume diffusion mechanism is much less than the observed attack. For volume diffusion out of a semi-infinite medium, the concentration is about 99% of its original value when the depth, d , is equal to $2\sqrt{Dt}$. D is the diffusion coefficient and t is the time. Values for D for volume diffusion are obtained by extrapolation of data from higher temperatures where the volume diffusion mechanism is known to predominate. Values for $2\sqrt{Dt}$ for Inconel, which have been computed from the data of Gruzin and Fedorov,⁵ are shown in Table I. Note that the computed depth of attack at 900°C is much less than that observed for Inconel and Fuel No. 30. The

TABLE I
DEPTH OF VOLUME DIFFUSION IN INCONEL

T°C	t-hr	$D - \frac{\text{cm}^2}{\text{sec}}$	d-mils
1000	1000	1.1×10^{-11}	4.9
900	1000	1.6×10^{-12}	1.9
800	1000	1.5×10^{-13}	0.6
700	1000	0.9×10^{-14}	0.1

difference, though marked, is in a conservative direction because the depth for one per cent depletion of chromium could be expected to be several mils greater than the nine-mils depth to which voids are observed. Additional proof is the fact that with impurities in the salt or when Fuel No. 107^x is held in contact with Inconel for 1000 hr at 900°C, a depth of voids greater than 35 mils is

^x 11.2 NaF-45.3 LiF-41.0 KF-2.5 UF₄ mol per cent

Fig. 1 (Y-12142) T14798 Hot-Zone Attack of Inconel by Fuel No. 30 in 1000 Hr at 900°C. Etchant: Modified Aqua Regia.

sometimes observed. Since d varies with the square root of D , the D value would have to be in error by more than 100-fold to account for this depth of attack. Such an error is well beyond experimental uncertainty. Thus, it is evident that the removal of chromium occurs by a grain-boundary diffusion process.

Quantitative Interpretation Based on Grain-Boundary Diffusion

It is possible to make quantitative predictions on the interrelations between the concentration driving force, the time, and the depth of attack assuming a grain-boundary diffusion mechanism. Fisher⁶ derived the following equation for grain-boundary diffusion for a system in which the grain boundary is perpendicular to the liquid-solid interface:

$$\frac{C_1 - C}{C_1 - C_f} = \exp \left[- (4D_l / \pi t)^{1/4} y / (\delta D_B)^{1/2} \right] \left[1 - (4/\pi)^{1/2} \int_0^{x/2(D_l t)^{1/2}} \exp(-Z^2) dZ \right] \quad (2)$$

In the above expression

C = solute concentration at point (x,y)

x = distance from grain boundary

y = distance from liquid-solid interface

C_i = initial concentration of solute

C_f = final or equilibrium concentration of solute

D_l = lattice diffusion coefficient

t = time

δ = grain boundary width

D_B = grain boundary diffusion coefficient

The second term in equation (2), i.e.,

$$1 - (4/\pi)^{1/2} \int_0^{x/2(D_l t)^{1/2}} \exp(-Z^2) dZ$$

is the well-known solution for volume diffusion in a semi-infinite medium. This term has a value of one at the grain boundary ($x = 0$). Since the voids observed in Inconel occur at the grain boundaries, this term has the value, one, when the depth of void formation is being considered. Thus the term, y , corresponds to the depth of voids, d .

Equation (2) may be used to relate the depth of attack with time under a given set of temperature and concentration conditions. According to equation (2):

$$y = kt^{1/4} \quad (3)$$

A plot of $\log y$ versus $\log t$ should give a straight line with slope $1/4$. Data for the depth of voids in the Inconel-Fuel No. 30 system at 925°C as a function of time are shown in Fig. 2. The line connecting these points were purposely drawn with a slope of $1/4$. If the attack process occurred by a volume diffusion mechanism, the slope of the line in Fig. 2 should be $1/2$. The data cannot be made fit this type of curve. It is interesting that the correlation given by equation (3) agrees with the observed corrosion data at very short, as well as very long times, so that one does not have to assume a two-stage corrosion process to explain the observed data.

In view of the fact that the CrF_2 content in the fused fluoride increases markedly at the beginning of the corrosion tests, the above correlation is surprising at first. Further study of equation (2) shows that the depth of attack varies with the logarithm of $C_i - C_f$. Thus moderate changes in C_f due to a shift in reaction (1) would be expected to have a small effect on d . In those cases where large changes in d are observed, i.e., with high impurity contents in Fuel No. 30 or when Fuel 107 is used, large changes in C_f would be expected. Such changes would be reflected in the solute content of loops run under different conditions so that careful sampling of loops during runs would be expected to yield valuable information which would explain the differences in depth of hot-zone attack under different conditions. To date a good method of sampling loops has not been devised.

The above analysis does not depend on the idea that the rate of mass transfer is controlled by the hot-zone process. Since the mass transfer occurs by a series of steps, the CrF_2 content of the salt adjusts itself so that the sum of the rates of the hot-zone processes is equal to the sum of the rates of the cold-zone processes. At steady state, any one of the steps may be used to describe the process and the investigator generally picks that step which is most easily described for study.

Fig. 2. Depth of Voids in Inconel Exposed to Fuel 30 at 900°C.

The Amount of Mass Transfer

Since the total mass absorbed by the cold zone equals the total mass removed from the hot zone, the amount of mass transfer may be described by the hot-zone process alone. For combined volume and grain-boundary diffusion processes, the total amount of material, A , which has diffused out of the latter per unit area is

$$A \cong \frac{2}{\sqrt{\pi}} (C_i - C_f) \sqrt{D_\ell t} \left\{ 1 + 4L \cdot \exp \left[-(4D_\ell / \pi t)^{1/4} / (\delta D_b)^{1/2} \right] \right\} \quad (4)$$

In equation (4), L is the length of grain-boundary intersections with the diffusion interface and the other factors are as previously defined. Except for very fine-grained material the exponential factor probably can be neglected so that A would be expected to vary approximately with the square root of time if the change in C_f with time were not too great. At each point in the loop the temperature would be different, making D_ℓ and the equilibrium C_f different, so that A would vary with the position in the loop but A for each point would still vary approximately with the square root of time. Thus the total amount of material leaving the hot zone A_t would be:

$$A_t = \frac{2}{\sqrt{\pi}} \sqrt{t} \int_{\text{Hot zone surface}} (C_i - C_f) \sqrt{D_\ell} \, dS \quad (5)$$

where S is the surface area of the interface. Except for t very close to zero the integral in equation (5) would be expected to be approximately constant for the non-steady-state case as well as steady-state for systems where C_f does not approach C_i .

In view of the above discussion it is interesting to plot the increase of chromium in Fuel No. 30 as a function of time. Such a plot is shown in Fig. 3. The line through the points was computed assuming that the analysis was correct for the 1000 hr sample, and that the slope of the curve is 0.5. The fact that the data do not fit the curve too well except for three points may be due to 1) the method of sampling the loop, 2) the change of C_f with time at every point in the loop, 3) errors in chemical analysis, or 4) the attainment of steady state in the loop.

Fig. 3. CrF₂ Build Up in Fuel 30 Exposed to Inconel. Maximum temperature 900°C, $\Delta T = 200^\circ\text{C}$, $Re = 10,000$.

This last supposition appears to be refuted by equilibrium data on the Fuel 30-Inconel system. Steady state could not be achieved until the CrF_2 content of the salt exceeded the value required for equilibrium in the coldest point in the loop.

Since the horizontal line at 1320 ppm CrF_2 is the value which would be expected to be in equilibrium with the 700°C cold zone according to Grimes,⁷ these data indicate that the loop has not reached equilibrium and that solution is occurring at every point in the loop. However, in view of the inaccuracies of the chemical analyses for UF_4 and UF_3 , which also govern the CrF_2 content, this correlation must be considered as highly questionable and is presented only for the sake of discussion.

If the steady-state CrF_2 content in the fused fluorides was not reached in the Fuel No. 30-Inconel tests whose data were used to plot Figs. 2 and 3, two conclusions could be made. One is that attack would occur at every point in the loop, although the depth of attack would vary with the temperature. Figure 4 shows the temperature and attack profiles in one of the Inconel-Fuel No. 30 loops. It is interesting that attack is observed at every point in the loop. Unfortunately this observation does not prove conclusively that the steady state had not been attained, for the voids in the cold zone may also be explained to be due to the rapid attack which occurs in the early stages of the test before the steady-state composition is achieved.

The second conclusion that could be reached is that the mass transfer is hot-zone controlled. This could not be the case since it is difficult to explain why high-impurity levels would increase the depth of attack. It is also impossible to explain the differences in the behavior of the loop when Fuel No. 107 is used instead of Fuel No. 30. It can be concluded that there is contradictory evidence regarding fused-salt corrosion and that careful studies should be made resolve the anomalies.

On other point about equation (4) is in order. It has been suggested that values of the diffusion coefficient in Inconel be obtained by following the amount of CrF_2 built up in a salt bath in contact with the sample. Since most of the material is supplied by volume diffusion and only a small fraction of the chromium is supplied by grain-boundary diffusion, the value of D so obtained cannot be used to compute the depth of attack, and extrapolations to other temperatures will be of questionable value. An understanding of the hot-zone process can only

Fig. 4. Pattern of Attack and Temperature Along Loop Length ~

come from very careful metallographic studies of the corroded Inconel together with careful chemical analysis of the chromium as a function of the depth of attack. However, chemical studies will be of the utmost importance in establishing the relationship between the CrF_2 content of the salt and the Cr concentration at the surface of the Inconel.

Relationship Between Temperature and the Depth of Attack

It is very difficult to predict the effect of temperature on the depth of void formation on the basis of equation (2). The quantities C_F , D , and D_B are known to be functions of temperature and it is possible that δ is also a function of temperature. In addition, no values of D_B or δ are available. As a matter of fact, the relationship between depth of attack and temperature would have to be determined experimentally in order to get D_B and δ .

There is an alternate method of predicting the effect of temperature on the depth of attack. Upthegrove and Sinnott⁸ have measured the depth of grain-boundary self-diffusion of nickel as a function of temperature and time. If it is assumed that the effect of temperature on the rate of grain-boundary diffusion in Inconel is the same as its effect on pure nickel, then the data of Upthegrove and Sinnott may be used to predict the depth of attack in Inconel.

Some assumptions must be used in relating the data of Upthegrove and Sinnott to the Inconel-Fuel No. 30 system. Upthegrove and Sinnott made measurements on bicrystals of varying relative orientations and found that the maximum depth of grain-boundary diffusion varied with the difference in orientations. The depth of attack was found to be greater, the greater the disregistry of the two grains. Since in a polycrystalline aggregate all relative orientations would be expected to be more closely related to the bicrystals with the highest degree of disregistry.

Figure 5 shows the maximum depth of grain-boundary diffusion observed by Upthegrove and Sinnott for a misfit angle of thirty-one degrees as a function of time at a series of temperatures. Since one would expect the depth of diffusion at any given time to increase with increasing temperature, it is quite surprising that all of the data fit the same curve. This would indicate that the depth of grain-boundary diffusion is practically independent of temperature, although the amount of material which had diffused would be expected to vary markedly. In terms of fused salt corrosion these data would indicate that the maximum depth at which voids are observed would be expected to be only slightly

Fig. 5. Maximum Depth of Grain Boundary Self-Diffusion of Nickel at a Series of Temperatures (8).

dependent on the temperature, although the total volume of voids would be expected to increase markedly as the temperature increases. This does not mean that the total number of voids will increase with increasing temperature, since at the higher temperatures certain voids grow at the expense of others to reduce the surface free energy. The total void volume is equal to the total number of voids times the average void volume. At the higher temperatures this latter quantity would be expected to be higher and might be sufficiently high to reduce the total number of voids while exhibiting an increased void volume.

In general the conclusion that the depth of grain boundary attack should be relatively independent of the temperature is refuted by the experimental observations. Manly et al.² showed that in a given loop the depth of voids varied approximately linearly with the wall temperature. Thermal convection and pump loops of Inconel containing modified Fuel 30 (one mole per cent UF_4) at a maximum temperature of $732^\circ C$ have shown about one mil attack in tests exceeding a year in duration. Although this fuel was not tested at higher temperatures, barren ZrF_4 -NaF salts showed about 5 mils attack in thermal convection loops operated for 1000 hr. Modified Fuel 30 would be expected to show a slightly greater depth of attack under such conditions, perhaps 7 mils.

One other point should be made about the results of Upthegrove and Sinnott. The slope of the curve, log depth of attack versus log time, is 0.432. Since equation (2) would predict the slope to be 0.25, the results are not in good agreement with Fisher's predictions and the solution for the grain-boundary diffusion problem given by Fisher is open to question. Obviously, more work is needed on grain-boundary diffusion of both an experimental and a theoretical nature.

Factors Affecting the Degree of Void Formation

Much work has been done on the mechanism of the formation of voids in alpha brass due to the unequal rates of diffusion of zinc and copper. Balluffi¹⁰ and Resnick and Seigle³ have shown that a vacancy concentration of 100 to 1000 times the normal thermal equilibrium concentration would be required for the homogeneous nucleation of voids at a reasonable rate and that experimental observations preclude such excess vacancy concentrations. Thus it is clear that the process of void formation must occur by heterogeneous nucleation, i.e., nuclei are already

present on which vacancies condense. These nuclei could be certain dislocation configurations, already existing voids produced during solidification of working, gas pockets, or inclusions in the metal. Resnick and Seigle³ showed that the number of voids increased markedly as the ZnO content increased in diffusion samples and that brass which has been freed of void nucleation catalyst by a high degree of purification exhibited a considerably reduced tendency for grain boundary cracking during creep and an increased stress-rupture life. Hopkin¹¹ showed that the presence of antimony increases the number of voids observed in the diffusion process.

The above observations indicate that the number of voids observed in Inconel exposed to Fuel 30 is probably a function of the chemical and mechanical history of the material. Thus variations in the depth and distribution of voids would be expected from test to test run under supposedly identical conditions. In fact, it is surprising that the agreement from test to test is as good as it is.

It is likely that with the proper control of the melting practice and working operations the number of voids in Inconel could be reduced. The reduction or elimination of voids would increase the stress-rupture life of the tubing, but not as much as one would desire, because the major strengthener in Inconel is the chrome carbides and these are removed by the depletion of chromium with or without the formation of voids. In the case of Inor 8 the strength is governed by the molybdenum content instead of the chromium carbides so that the formation of voids could be relatively more detrimental. Under the molten salt reactor conditions, however, no voids are observed in Inor 8 so that special control of the melting practice to eliminate voids is not required.

Further Studies Needed

If one considers all of the information which has been obtained on fused salt corrosion of nickel-base alloys, certain anomalies arise. In view of the anomalies it is still uncertain whether a) steady state is reached in times up to 3000 hr, and b) if it is, whether the mass transfer process is hot zone, or cold zone controlled. It is apparent that an experimental program should be devised which would answer these questions conclusively. Since the same oxidation-reduction reaction is operative regardless of the composition of the fused fluorides, namely reaction 1, a thorough understanding of any one system would be equally applicable to others of interest. Because the data are more complete for the Fuel 30-Inconel system, it is suggested that further work be done on this type system.

Studies to determine whether steady state is attained and what is the rate controlling step in the mass transfer process should be of two types 1) pot type isothermal tests to determine rate constants for mass transport and 2) thermal convection loops to correlated actual loop performance with the behavior computed on the basis of isothermal data. Thermal convection loops are suggested because they are cheaper and easier to operate, and because the flow rate is not an important factor in the fused salt corrosion process.

One type of apparatus which could be used for the isothermal tests will be described. Small samples of Inconel would be placed in isothermal pots of molybdenum containing Fuel No. 30 with the volume of salt sufficiently high that there is insufficient Cr in the sample to saturate the salt. The relationship of d and t would then be studied and careful analyses of the salt and of the sample would be made. Additional tests would be made with Fuel No. 30 containing various quantities of CrF_2 and at other temperatures. The data would then be analysed to obtain the relationships between ΔC , d , t , and T . Such studies would answer the question of whether d varies linearly with t , with $t^{1/4}$, or with some other function of t . Similar studies would be made for the case where the CrF_2 content exceeds the saturation value. The pickup of chromium in the samples would be followed to determine if the process occurs by the same or a different mechanism from the hot-zone process. In these pot type studies it is suggested that the oxidation reduction reaction should be

instead of reaction (1), because the chemical species could be much more easily monitored.

In conjunction with the pot type studies carefully controlled thermal convection loop studies should be carried out. Again, in these tests the oxidation reduction reaction should be reaction (6). A method of continually monitoring the hydrogen pressure would be included as well as a method of taking samples of the fused salt as a function of time. In order to eliminate transient effects attempts should be made to pre-equilibrate the loops. At the conclusion of each test the distribution of attack would be determined throughout the loop and the chromium profile in the solid at various points would be measured. These results would then be compared to calculations based on the static pot type tests.

More work should be done to determine the effects of impurities on the rate of corrosion of Inconel. Attempts should be made in isothermal pots to duplicate the thirty-five mils attack of Inconel in Fuel 107. It would also be of interest to carefully examine the solid-state diffusion process which leads to such a depth of attack. A better understanding of the hot-zone corrosion process would give experimental corroboration of the rate controlling steps determined by chemical studies on the fused salt.

Finally, much work is needed to understand the effects of fused fluoride corrosion on Inor 8. Chemical analyses of successive layers of corroded samples should be made to determine if the chromium distribution corresponds to that observed in Inconel. If discrepancies arise, experiments should be carried out to determine the effects of molybdenum on the rate of diffusion of chromium in nickel base alloys. Mechanical tests should also be run to show if exposure to fused salts decreases the stress-rupture life of Inor 8 tubing. Any effects on mechanical properties would then be correlated with changes in the composition and microstructure of the best samples.

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