OCT 11 1988

Dr. Charles G. Interrante, Program Manager
Metallurgy Division - Corrosion Section
National Bureau of Standards
U.S. Department of Commerce
Gaithersburg, MD. 20899

Dear Dr. Interrante:

We have reviewed NBS' Monthly Letter Report for July 1988 for FIN A-4171, "Evaluation and Compilation of DOE Waste Package Test Data." Comments on the MLR are presented below in Attachment 1.

Actions resulting from this letter are considered to be within the scope of FIN A-4171. No changes in costs or delivery of contracted products are authorized. Please notify me immediately if you feel this letter will result in additional costs or delay in delivery of contracted products.

Sincerely,

Charles H. Peterson
Technical Review Branch
Division of High-Level Waste Management
Office of Nuclear Material Safety
and Safeguards

Enclosures: Att. 1

cc: w/Att. 1:

Dr. Neville Pugh, Director
Metallurgy Division, NIST

Dr. David Anderson, Group Leader
Metallurgy Division, NIST

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ATTACHMENT 1

COMMENTS ON MONTHLY LETTER STATUS REPORT
JULY 1988 (FIN A4171)

1. Pg 1, ¶1

   State what the improved capabilities of Advanced Revelation over Revelation are.

2. Pg 2, Database Searches

   Provide more information on Compendex Plus. Who is Engineering Information Inc., what is the size of their database, and what areas do they attempt to cover?

3. Pg 3, ¶1

   The Bazan report deals with leaching of glass samples in Teflon dishes. We have recently been advised that long term leaching of Teflon results in leaching of fluoride ions. Was there any mention of this in the report?

4. Pg 3, ¶2

   Study of the corrosion of copper in HCl solutions seems to be departing from expected environments. Is there any indication that the work described would add to understanding of copper behavior in tuff?

5. Pg 3, ¶3

   The Yow report on possible tests to characterize water flow through pores and fractures is certainly of interest, but we would prefer that NIST not spend time with this and concentrate on documents dealing with materials performance.

6. Pg 6, Task 2

   Statements like "work is continuing" are not sufficiently informative. Acceptable reporting would be one of the following:

   a. No work was done in this area during this reporting period.
   b. No new data or tests needed for demonstration of compliance with performance objectives were identified.
   c. Two tests and one data gap were added this month to the list of tests and data considered necessary to demonstrate compliance, bringing the total to five tests and seven data gaps. A summary statement is in preparation.

7. Pg 7, Task 3

In the interests of brevity, please delete the lead paragraph in future MLRs. It would be sufficient to label each section as A, B, etc. Also, the purpose of the MLR with respect to test work is to report what was done, were any problems encountered, what steps are being taken to deal with these problems, and what is planned for the next reporting period.

A. SCC Propagation

Please quantify "a considerable improvement in the sensitivity of the measurement system": was it by a factor of 1.5? 10? 100?

Is any preliminary estimate available yet of the minimum detectible rate of crack propagation?

What is the nature of the test planned for August?

B. Resistivity Effects

Two problems were noted with respect to the tests involving agar: bacterial growth and moisture loss. What is proposed to deal with these problems, or should some other medium be substituted?

C. Pitting Corrosion in Steel

Delete the first sentence on the basis that readers of the MLRs have already been informed of this fact.

Where will the proposed paper be presented?

The statements on corrosion rates found are informative. Can the writer offer any rationale, however preliminary, for the lower rate in concentrated J-13 water based on differences in other variables such as chloride content, oxygen content, or test time?

We presume uA means microamperes. How is the conversion from uA to mils made?

Since thick films apparently reduce diffusion rate, it would appear inaccurate to say they are non-protective. Perhaps non-adherent would be better.

See Comment 3 above re the potential impact of leaching of fluoride from Teflon.

D. Corrosion of Zircaloy

Delete the first sentence as noted above.

The shift from -362 mV to +204 mV in one day appears to be a substantial shift in potential, yet the corrosion rate is essentially zero in both cases. Was this expected? Is there a tentative rationale?

How are the welded areas characterized prior to testing?

DRAFTS OF DOCUMENT REVIEWS

A. Delany report on modeling tuff/J-13 interactions

1. CONTENTS

From the viewpoint of reducing the number of words that must be entered into these document reviews, typed, proof-read, read by the NIST, read by the NRC, and read by ultimate users, it does not appear necessary to report the information under CONTENTS in sentence form. See previous comments on format.

2. AMOUNT OF DATA

Again, delete the first sentence since this has already been stated under CONTENTS. As above, further consideration should be given to summarizing the information content of the tables and figures. For example:

> Tables 1 - 4 give composition data for J-13 water and tuff, and characterizations for test specimens.
>
> Figures 1 and 2 give log activity for potassium and sodium vs log activity of silica. Figures 3 to 5b (6 figures) show simulations of concentration of various anions vs time for tuff wafers and crushed tuff. Figure 6 shows a simulation to 100 years.
>
> Three appendices provide input data.

However, rather than merely stating what is plotted in each figure, it would be more useful to the user of the review to know what was found by each plot. Perhaps only the important figures should be discussed rather than every figure in a report.

3. CONCLUSIONS

What is "a reasonable approximation"?

The conclusion that the simulation does not work as well at 250°C as at 150°C implies that EQ3/EQ6 is not yet a satisfactory tool for modeling

the tuff environment. It is recommended that more data on mineral compositions must be obtained and several rate laws for precipitation kinetics must be added to the code.

Were there any other conclusions found by the reviewer?

B. Westerman report on corrosion of 304L SS in tuff groundwater

1. Pg 16, CONTENTS

Please provide a breakdown for the other 29 pages. How much, for example, was devoted to an analysis and discussion of the results?

2. Pg 17, MATERIALS/CONPONENTS

The difference between heat A and heat B is not identified. Also, the purposes of this section might be adequately covered by stating:

> Test materials included 3 stainless steels (304, 304L, and 316L) in two thicknesses (1.52 and 6.35 mm).

3. Pg 19, METHODS OF DATA COLLECTION

If sensitization is due to carbide precipitation at grain boundaries, what occurs when an electric charge is applied to the surface of the metal that results in repassivation?

4. Pg 20, ¶5

The report states that annealing 304L at 600°C for 10 hours resulted in a sensitized microstructure whereas annealing another sample for 24 hours did not. TEM examination showed a continuous layer of grain boundary precipitates for the 10 hour sample and none for the 24 hour sample.

Comment: This is an example of the contradictory results one finds in the literature. The authors attribute these results to some irregularity in the processing of the steels, which actually came from different heats. Such results tend to reduce the confidence in conclusions reported by various authors on SCC.

5. Pg 21, CONCLUSIONS

Since specific conclusions are drawn by the authors, we wish to again point out that providing a reviewer critique for each conclusion is probably the most useful part of the review. The comments provided here

illustrate the kind of critical review desired.

(1) "Solution treated 304L SS can exhibit transgranular corrosion cracking."

Comment: Was this expected? How does this differ from the behavior of 304 SS? Was this true for both thicknesses tested? What is the behavior of 304 L that has not been solution annealed? Is the stress at which TSCC occurs higher than that for ISCC?

(2) "It is likely that the cracking was chloride induced and accelerated by additional oxidizing power resulting from the gamma irradiation."

Comment: Was transgranular cracking shown for only irradiated samples? What was the level of irradiation? What was the level of oxygen or oxidizing species in the test environment? What control specimens were included?

(3) "Most of the failures observed in the gamma flux test occurred in the vapor phase region of the 90°C autoclave."
"Test conditions differ from the anticipated repository conditions."

Comment: Were there no results for 90°C, 150°C, or 200°C? How much is "most"? All but 2 of the results? 90%? Did any of the specimens in the liquid phase fail? What was the oxygen content of the liquid phase? What type of failures were observed for the vapor phase samples: intergranular or transgranular?

How much do the test conditions differ from repository conditions? Why were not repository conditions included? Do the test conditions differ enough so that none of the results apply to understanding of the effects of using repository conditions?

(4) "Type 304 SS is more susceptible to SCC than 304L."

Comment: Does this mean that some of the 304L specimens failed? This conclusion is well-known, and should be stated as one of the confirmations that the experimental technique was satisfactory rather than as a new conclusion.

(5) "When gamma flux was present, the sensitized 304 exhibited ISCC in slow strain rate (SSR) tests while the sensitized 304L did not."

Comment: Sensitization apparently has been explained on the basis of carbon migration to grain boundaries. Does unsensitized 304 show ISCC?

Is there some level of carbon below which no sensitization occurs in 304 SS? How was it determined that the 304L had indeed been sensitized? The description should probably be: "No ISCC was observed for 304L subjected to sensitizing conditions."

Solution annealing is apparently done at 1050°C for 15 minutes whereas sensitization is done at temperatures below 700°C for periods of hours. What happens in the steels under these two sets of conditions?

What are the results when gamma flux is present? The reviewer should include a matrix of results for the variables solution annealing/sensitization, gamma/no gamma, 304/304L, no cracking/ISCC/TSCC, vapor phase/liquid phase, air cool/water quench, and U-bend/SSR tests.

(6) "Susceptibility to ISCC in SSR tests done in 150°C J-13 water was shown for 304 SS sensitized at 600°C for 24 h."

Comment: What about steels sensitized at 550°C and then air cooled, or at 700°C and then water quenched?

(7) "The susceptibility of 304 SS to SCC in SSR tests was correlated with the formation of grain boundary precipitates. Cracking was intergranular in all cases."

Comment: Again this should be stated as consistent with previous information, not as a new conclusion. The term "correlated" seems inappropriate; "associated with" seems more accurate. Also, does this conclusion apply to <u>all</u> SSR tests?

(8) "Neither 304L nor 316L SS was found to be susceptible to SCC in SSR tests using J-13 well water."

Comment: Was this for both solution annealed and sensitized specimens? How does this relate to Conclusion (1), above?

6. GENERAL COMMENTS OF REVIEWER

(1) Irradiation Corrosion Experiments

The first paragraph reads like a series of results, rather than as comments on the work of the authors. Did the authors note any of these results? In any case, even though it has been agreed that author's material, if cited, will be in quotation marks, it will aid clarity for the reviewer to state that supplementary results are being noted. Because of the number of variables involved, it is difficult to grasp the relationships among the various statements being made. This material needs to be better organized, as for example with the aid of a matrix as suggested above.

(a) Type of degradation

Does the statement, "Specimens in the rock/water region of the autoclave showed no obvious evidence of attack (by general corrosion)", mean all specimens?

Does the statement, "No evidence of pitting was found on any of the samples", mean "any of the samples in the rock/water region" or "none of the samples, including those in the vapor region"?

How many specimens were in each cell of the matrix? For 2 Temperatures x 2 Gamma levels x 3 Zones x 2 Metals x 2 Replicates, there would be 48 samples. But there were 6 Times. Did the 15 of 48 U-bend samples fail at different times due to SCC? How is the variable of solution annealed vs solution annealed and sensitized accounted for? What was the material for the 15 failed specimens? Were they all 304 SS? If so, why did only 15 of 24 specimens of 304 SS fail?

Failures of 10 of 24 sensitized 304 SS U-bend specimens occurred regardless of the region. Thus, although more failures occurred in the vapor region, there were also failures in the liquid region. Thus, the role of the vapor region is left unclear.

The first paragraph on page 22 states:

- One sample each of solution annealed 304 and 304L failed in the vapor region by transgranular cracking.

- One sensitized 304L sample failed in the vapor region by transgranular cracking and one by mixed transgranular and intergranular cracking.

This indicates that low carbon, to the level involved in the actual specimens does not protect 304 from SCC. Would a lower level protect 304? Or should some other variable be adjusted?

(b) Simulation of repository conditions

The reviewer concludes that the test conditions are not unrepresentative of repository conditions, contrary to the authors' statement. The rationale in this section should be discussed further with the NRC.

(2) Boildown tests

Here again the reviewer merely presents a result: what is needed is a critique of this result. For example, it has been argued that evaporation within from tuff matrix will result in concentration of solutes and hence eventually a more corrosive environment. This result indicates that 304L is resistant to more concentrated solutions. Would these test conditions be considered more representative of repository conditions? Is this result conistent with other results in this report?

(3) SSR Experiments

SCC was observed only for sensitized 304 samples. In view of the number of variables, this result should include the conditions for which it is valid.

(4) Sensitization Studies

Here the reviewer states an important observation: the sensitization studies are rather preliminary. This statement should appear prominently at the beginning of the Conclusions section of the authors' report. The authors speculate that the observed differences in behavior of 304 and 304L may be due to precipitate morphology and chemical differences, respectively. How about the amount of precipitate? What chemical differences? Carbon content? Something else?

C. Van Konynenburg report on Carbon-14 in spent fuel waste packages

1. CONCLUSIONS

(1) "Published measurements of C-14 in U.S. spent fuel are inadequate and deal mostly with Zircaloy-clad fuel (Westinghouse PWR fuel)."

Comment: Why are the published values inadequate? What other types of spent fuel are there?

(2) "The chemical form of the isotope in the fuel is not known. The carbon probably exists as interstitial carbon or zirconium carbide in the cladding."

Comment: Other sources state that carbon is present as carbon dioxide, and that perhaps half of the carbon is in this form. If present as carbon or carbides, perhaps the release of C-14 on penetration of a fuel rod is negligible. This point should be flagged by the reviewer for resolution by further information.

(3) "A negligible amount of C-14 is released from heated, intact spent fuel in nitrogen or helium."

Comment: Why should any C-14 be released from intact spent fuel? How much is a negligible amount?

(4) "In heating an intact PWR fuel assembly, C-14 on the surface was oxidized to CO_2 ...The isotope may have...been adsorbed from the ractor cooling water?"

Comment: How did carbon get on the surface of the spent fuel? If the origin was the cooling water, how did it get into the water?

(5) "In less than one year, more than (1 part in 100,000) of the C-14 inventory can be released from the spent fuel."

Comment: Under what conditions? Heating? Oxidation?

(6) "C-14 released by pressurized gas escaping when fuel rod cladding ruptures may be about (1 part in 10,000) of the calculated total rod inventory."

Comment: What units are used for inventory?

(7) No comment.

(8) The first statement in this conclusion is not a sentence. What is it that must be modeled: the oxidation process?

(9) No comment.

The reviewer notes that the authors concede that their estimates of the C-14 inventory are based on calculations and should be confirmed by measurements. Was there any estimate of the probable error in the inventory estimates?

COST STATEMENT

1. Expenditures for Pay Period 15 are more than double those in the two previous periods. Large changes and unusual outlays should be explained.