QA: NA

Analysis of Alloy 22 Weight Loss due to General Corrosion

3-5-2008

Yucca Mountain Project

LSN Relevant

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Sandia National Laboratories

Background Introduction

- The corrosion rate used in the TSPA-LA is based on corrosion test data from 5 year test specimens and generated in ~FY01
- The 9.5 year test specimens are being readied to provide new data that would presumably validate the TSPA-LA corrosion rate
- In a technical review of the 5 year data and a review of the methodology, a vulnerability was uncovered

Issue Summary

- There are documented, repeated and potentially significant excursions from the ASTM standard for corrosion test specimens
- The cleaning of 5 year corrosion coupons from the LTCTF may have been incomplete, leaving samples in an indeterminate state
- Complete cleaning of the 5 year coupons could result in larger weight loss values than are currently used for the general corrosion model
- Complete cleaning of 9.5 year coupons might show an increase in corrosion rate compared to the existing 5-year data
- While corroborative data exist in the literature, almost all corrosion studies reviewed to date reported corrosion rates greater than the 5 year data from the LTCTF
- Net Effect
  - Our TSPA-LA corrosion rate may not be conservative and if not, could significantly affect the results for the EIS and the peak dose results
  - Basic defensibility of the TSPA-LA corrosion rate is jeopardized without some confirmation, in essence the rate we now use is an assertion
  - A CR will be initiated

Path Forward

- Initiate a CR
- Corrective Action
  - Initiate and accelerate new scope to collect the data that will confirm or refute the conservatism in the TSPA-LA corrosion rate before docketing
    - Regenerate and re-evaluate the 5 year data and generate 9.5 year data correctly
      - ROM estimate is ~650K, refined estimate pending
    - Expert judgment is <50% chance the TSPA-LA rate is invalid, but given the critical nature of this parameter is must be confirmed
- Prevent Recurrence
  - Transition the corrosion test samples, testing, and data generation to a fully ASTM compliant methodology with qualified technicians and equipment
- Conclusion
  - Does not effect going forward with the LA if we generate the confirmatory data prior to docketing

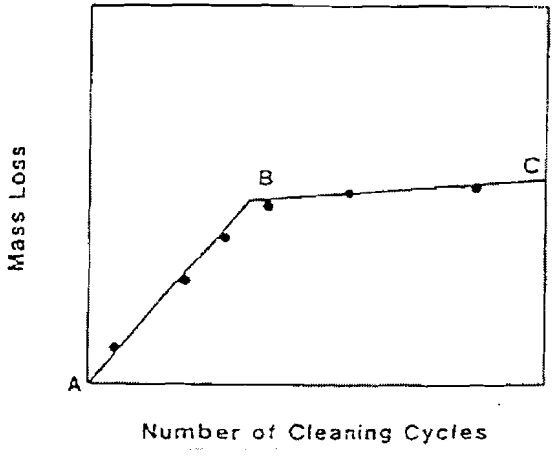
Supporting Information

The cleaning procedure used on the 5 year samples does not appear to meet the intent of the ASTM guidelines

ASTM G1-90: Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens (reapproved 1999).

ASTM G1-90	TIP-CM-51/Lab notebooks
(Pre-exposure) 6.3.1 Degrease in organic solvent or hot alkaline cleaner; 6.3.2 Pickle in an appropriate solution if oxides or tarnish are present	Samples in the LTCTF were tested in the as-received condition from the supplier (Notebook 241, p16) or samples were degreased in acetone (McCright 1998 [DIRS 114637], p2.2-11)
(Post-exposure) 7.1.1 ... By weighing the control specimen before and after cleaning, the extent of metal loss resulting from cleaning can be utilized to correct the corrosion mass loss.	TIP-CM-51, Appendix A. Weight loss on control foil samples was shown to be on the order of 0.1 mg (resolution of balance). No data from Alloy 22 coupons was located. No corrections made to weight loss data.
(Suggested practice) 7.1.2.1 The cleaning procedure should be repeated on specimens several times.	TIP-CM-51, 7.2.3.1 ... If the weight is at or below the initial starting weight, cease cleaning the specimen and proceed to step 7.2.5 below.
(Suggested practice) 7.1.2.2 The mass loss should be graphed as a function of the number of equal cleaning cycles ... the mass loss due to corrosion will correspond approximately to point B [see graph next slide].	TIP-CM-51, 7.2.5 Selected specimens will be checked by scanning electron microscopy (SEM) to ensure that all the corrosion products are removed. If corrosion products remain on the specimen repeat cleaning procedure.

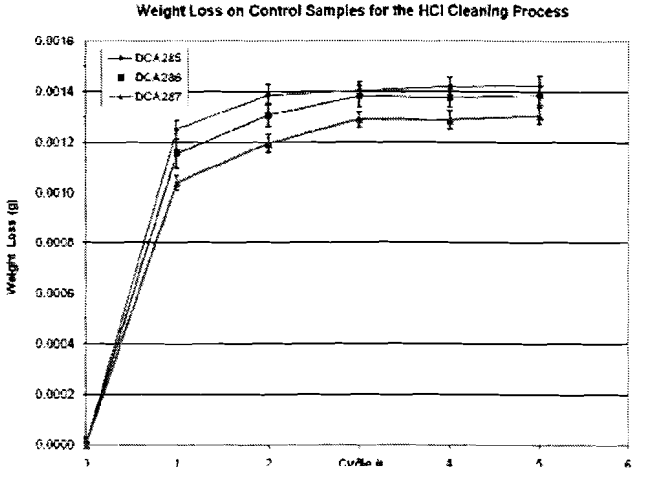
ASTM guidelines, previous control samples and recent scoping tests suggest that the weight loss per cycle should be negligible on a thoroughly cleaned sample

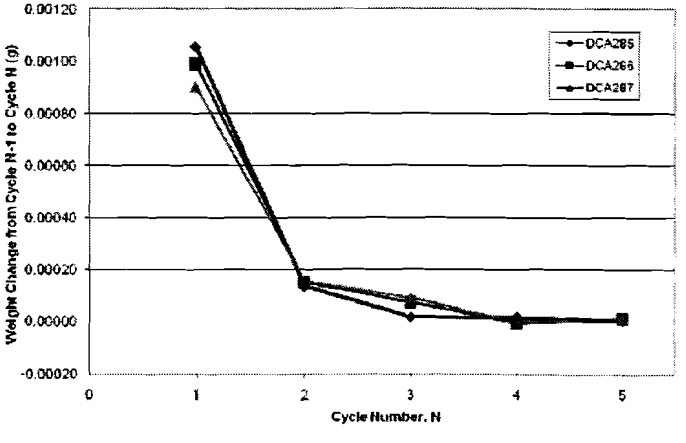


ASTM G1-90: 7.1.2.2 ... the mass loss due to corrosion will correspond approximately to point B

Foil	Weight Loss (g)
DFA01	0.0000
DFA02	0.0001
DFA03	0.0001
DFA020	0.0000

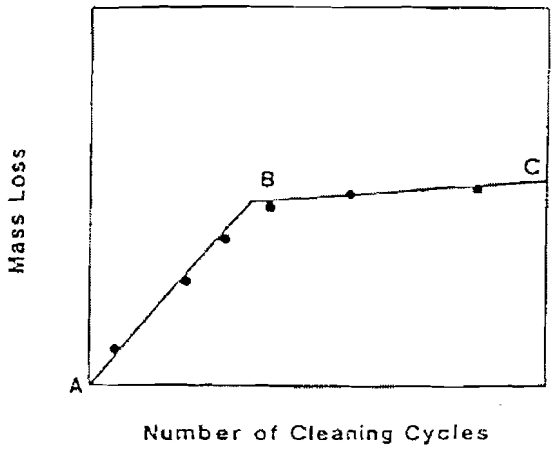
TIP-CM-51, Appendix A: foil samples (controls) exposed for 3 min in HCl bath.





Feb 08 Scoping tests: Alloy 22 coupons in HCl bath.

With Low Weight Losses, Technique and Equipment Sensitivity and Precision are Vital



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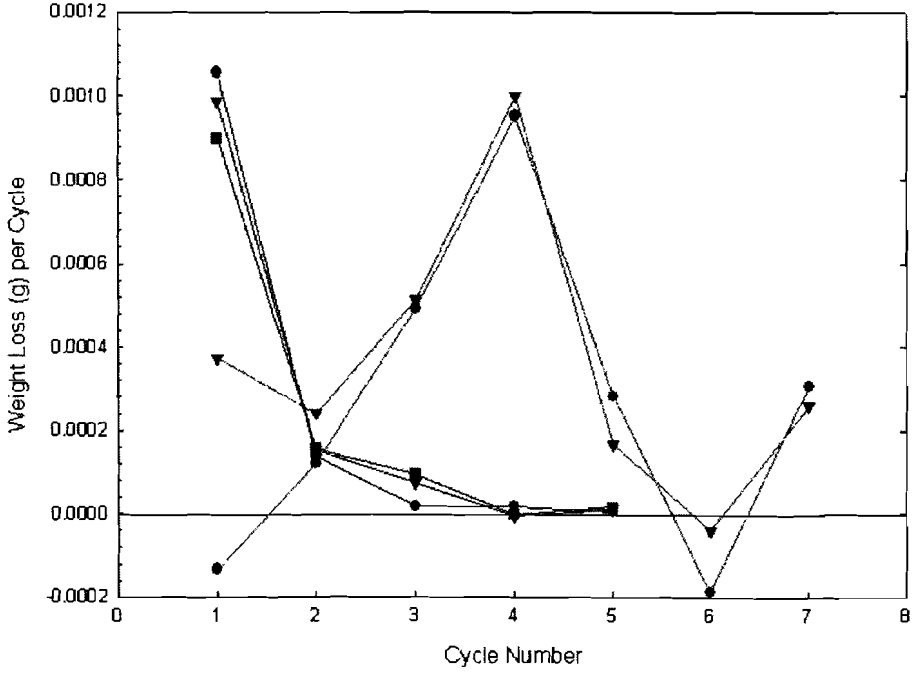
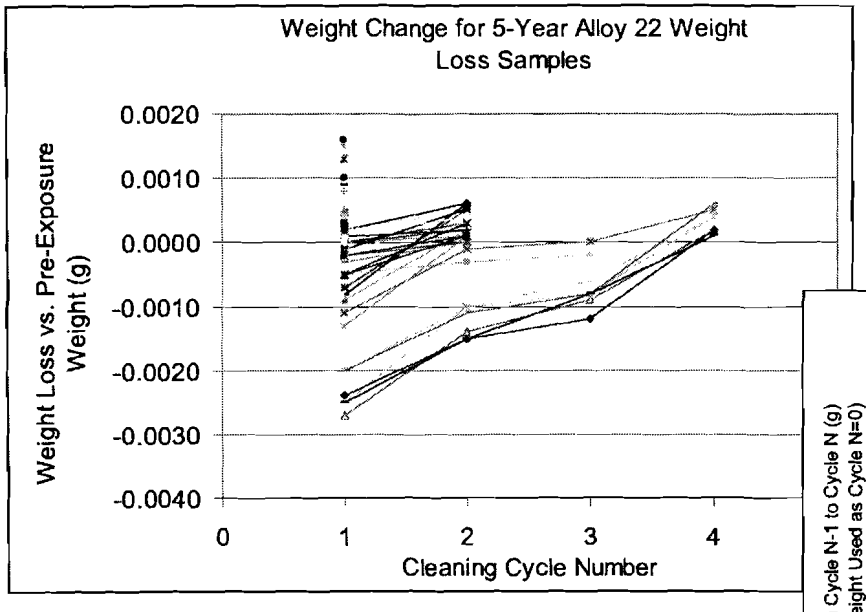


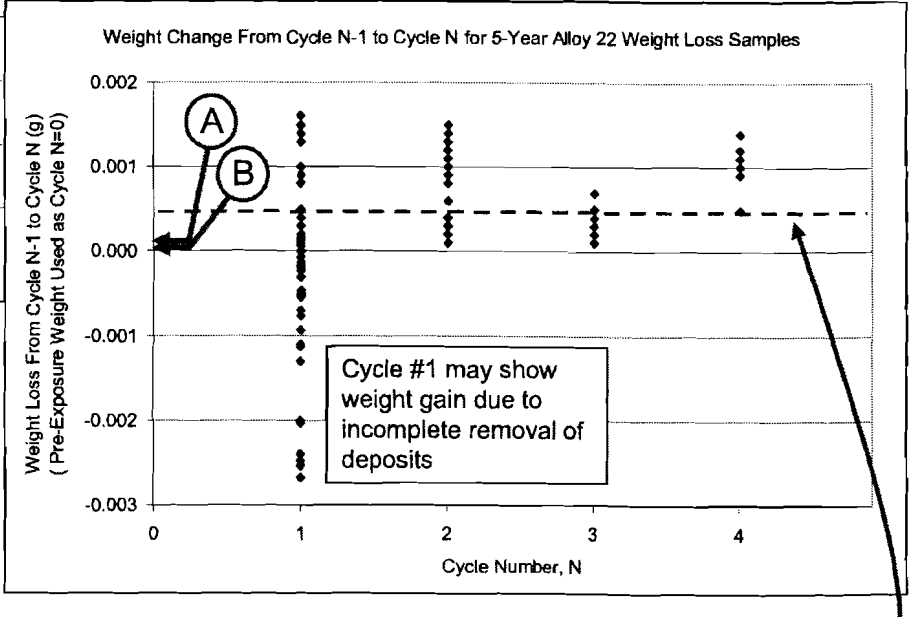
In practice, much care must be taken to perform the cleaning properly to obtain meaningful results. Blue and Red curves represent different cleaning techniques on identical blank samples

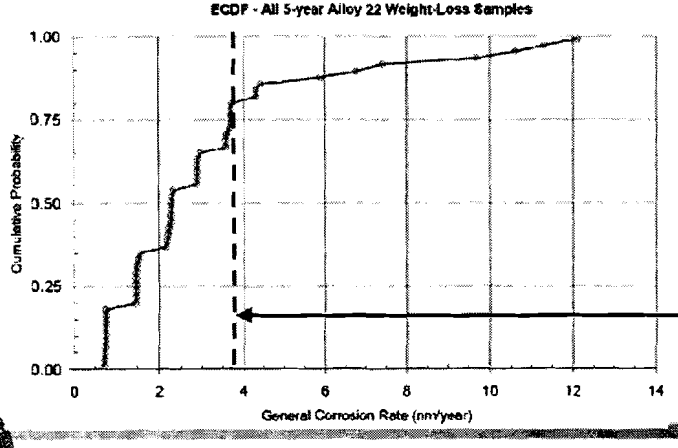
The process used to clean the 5 year weight loss samples likely left the samples in an indeterminate state



(A) Approximate weight change from control experiments in TIP-CM-51, Appendix A

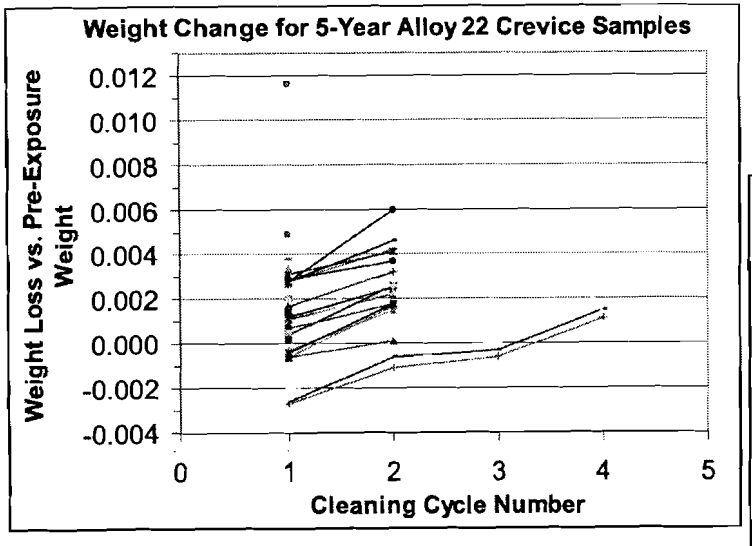
(B) Approximate cycle to cycle weight change from scoping tests Feb-08 (for cycles 4 and 5)





Corrosion rate corresponding to 0.0005 g weight loss on 5 year coupons – this mass loss was often exceeded in a single cleaning cycle.

The process used to clean the 5 year crevice samples likely left the samples in an indeterminate state



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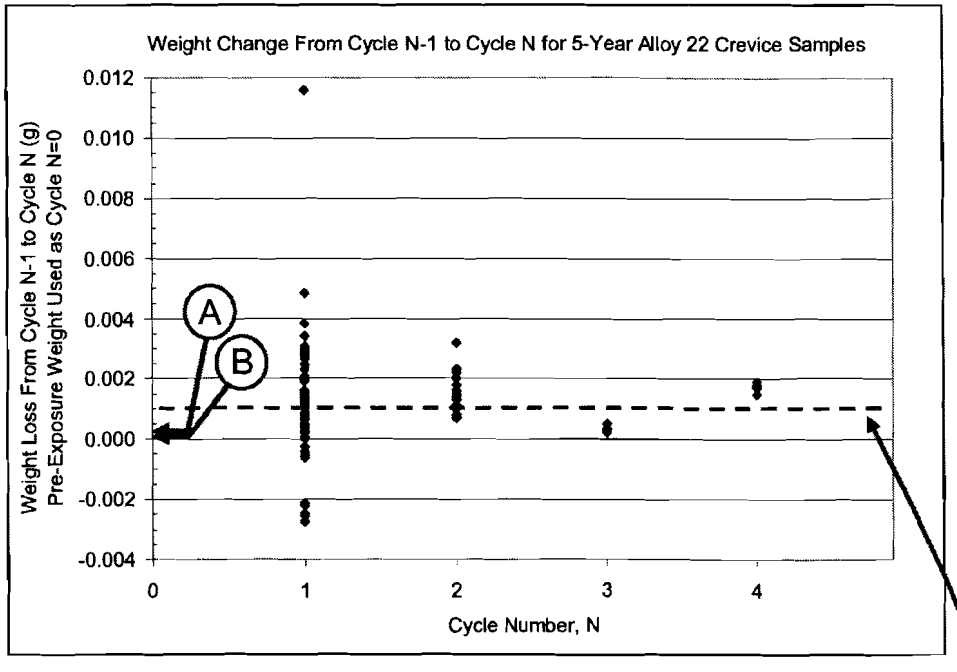
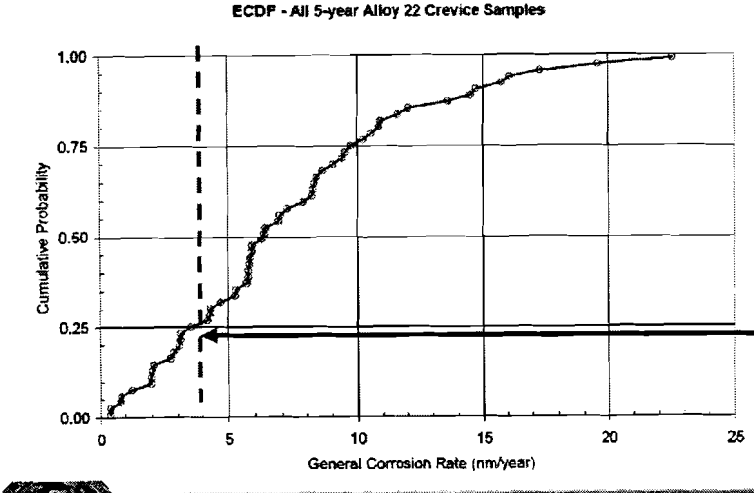




Corrosion rate corresponding to 0.001 g weight loss on 5 year coupons – this mass loss was often exceeded in a single cleaning cycle.

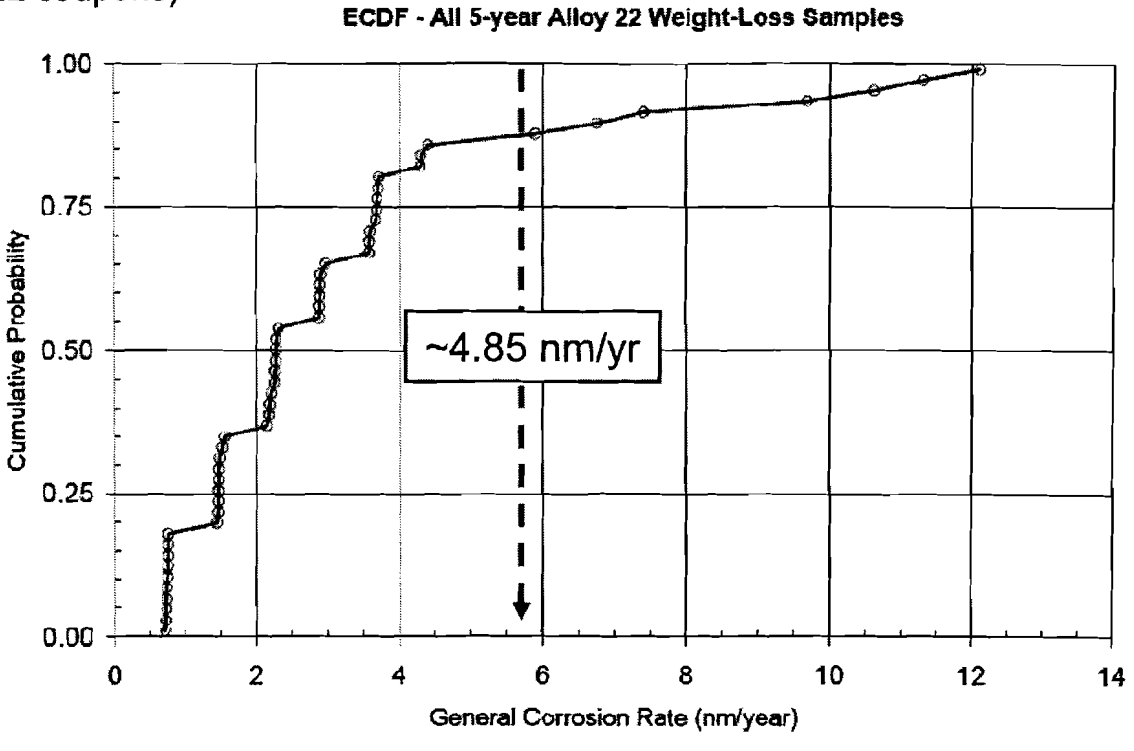
Complete cleaning of the 9.5 year Alloy 22 weight-loss samples may incorrectly indicate an increase in corrosion rate

Example: 9.5 year exposure resulting in 1.2 mg of observed weight loss

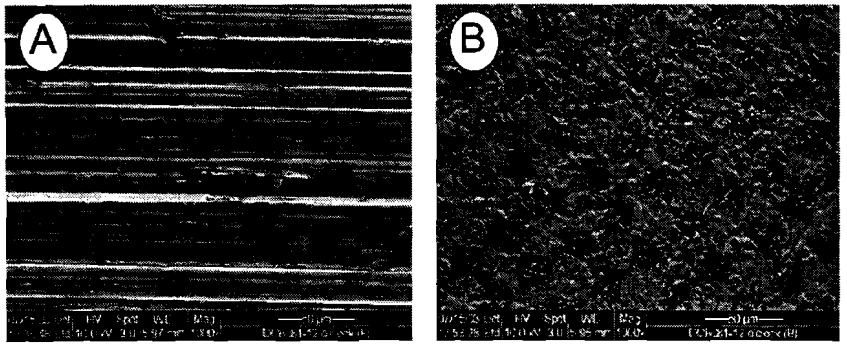
(1.2 mg is the approximate weight loss observed on recently cleaned unexposed Alloy 22 coupons)

$$\frac{87.6\times10^{9}\cdot 0.0012g}{8.69g/cm^{3}\cdot 30cm^{2}\cdot 83220hrs}=4.85nm/yr$$

*equation taken from ANL-EBS-MD-000003 Rev 03, Eq. 6-7, p6-27.



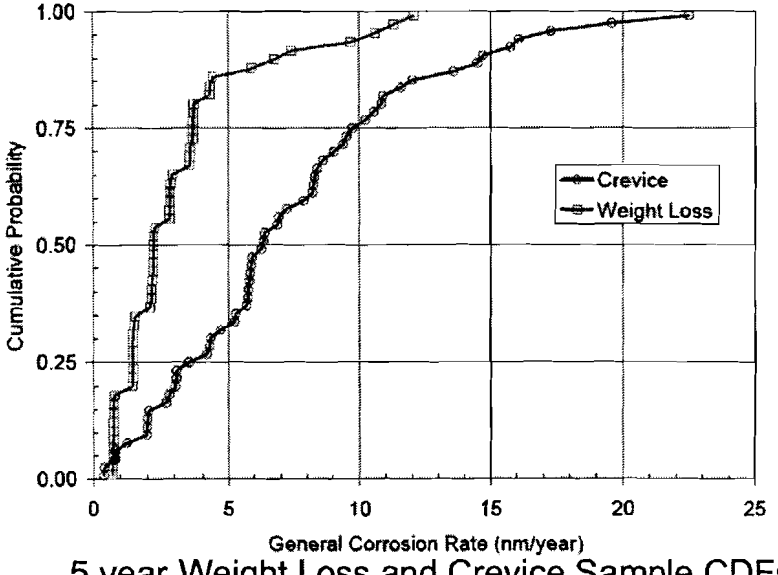
Complete cleaning of the 9.5 year Alloy 22 crevice samples may have indeterminate results as no control data have been collected for samples with the crevice sample surface finish



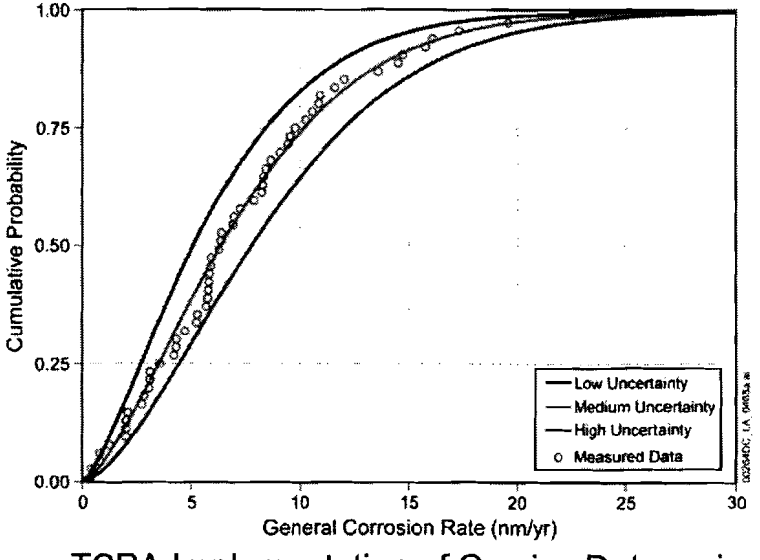
(A) Typical mechanical polish for weight loss coupons and one surface of crevice coupons

(B) Surface finish on one face of crevice samples.

* Images from L. Wong (2004) [DIRS 174800], Figure 4, p.15



5 year Weight Loss and Crevice Sample CDFs



TSPA Implementation of Crevice Data, using unbounded Weibull distributions

Corroborative corrosion rate data typically exhibit rates higher than the 5 year data (at shorter exposure times)

Reference	Corrosion Behavior Reported
Baker 1998 [DIRS 154510]	Alloy 600 under atmospheric conditions: 8 nm/yr after 36 years of exposure
McCright 1998 [DIRS 114637]	Alloy C-4 in marine atmospheric environment: no visually apparent degradation after 50 years
Smailos 1993 [DIRS 168164]	Alloy C-4 at 150C in NaCl-rich brine: 70 nm/yr at 12 months and 60 nm/yr at 18 months

Exposure Time		Mean Rate	Sources
(years)	(days)	(μm/yr)	
0.0027	1	0.460	Lian et al. 2002 [DIRS 164856], Table 3, potentiostatic polarization technique at 100 mV versus SSC applied potential in SAW, 90°C, N_2 purge.
0.0027	1	1.250	Lian et al. 2002 [DIRS 164856], Table 3, potentiostatic polarization technique at 100 mV versus SSC applied potential in SCW, 90°C, N_2 purge.
0.019	7	0.100	Evans and Rebak 2002 [DIRS 164857], Figure 2, polarization resistance technique, after one week in open-circuit potential in SAW, 90°C, air purge.
0.154	56	0.182	Hua and Gordon 2004 [DIRS 171013] calculated from regression fit for 56-day weight-loss in BSW, CR (MPY) = 31.3*exp(-25300/RT).
0.50	183	0.076	LL990610605924.079 [DIRS 104994] s99359_032 DATA REPORT (weight-loss) and s99359_031 DATA REPORT (crevice)
1.00	365	0.025	LL990610605924.079 [DIRS 104994] s99359_005 DATA REPORT (crevice data) and s99359_006 DATA REPORT (weight-loss data)
2.00	730	0.014	LL000112205924.112 [DIRS 141284] S00041_005 DATA REPORT (Combined crevice and weight-loss data)
5.00	1,825	0.007	Section 6.4.3.3, Table 6-6 (Crevice data only)

Validation DTN: MO0706WPOBVALR.000, file: *Figure 7-1.xls*.

The Project weight loss data are of similar magnitude to recent control data and observed cycle-to-cycle weight change from the analysis of the 5 year data

Time (years)	Corrosion Rate (μm/yr)	Equivalent Weight Loss (g) (based on crevice sample geometry)
0.154	0.182	0.0015
0.5	0.076	0.0020
1	0.025	0.0013
2	0.014	0.0016
5	0.007	0.0018

Feb-08 scoping test on Alloy 22 control coupons	0.0012 g weight loss after 2-3 cleaning cycles
Cycle to cycle weight change for 5 year weight-loss coupons	Significant number of trials resulted in weight loss between 0.0005 g and 0.0015 g
Cycle to cycle weight change for 5 year crevice coupons	Significant number of trials resulted in weight loss between 0.001 g and 0.003 g

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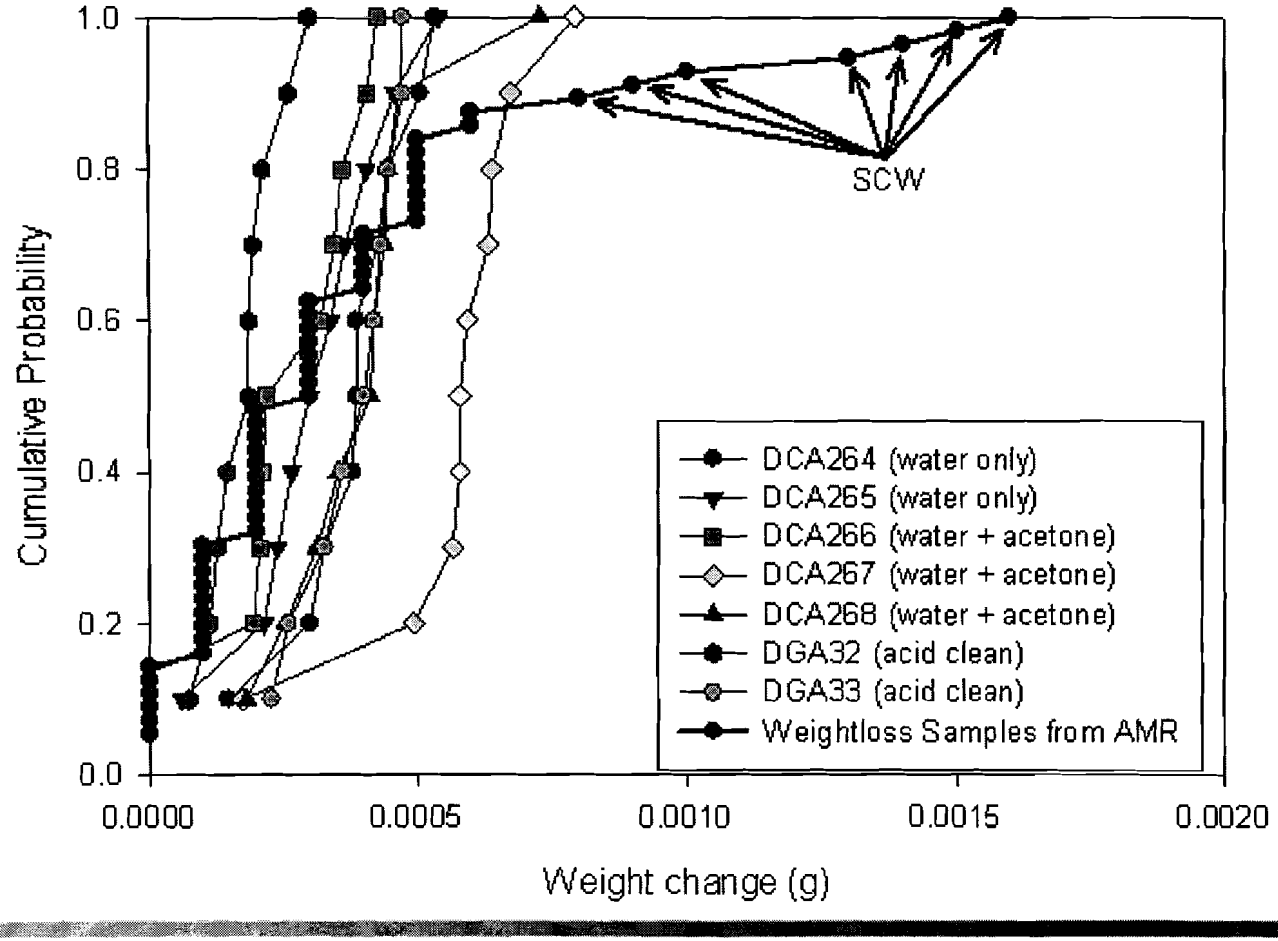

Proposed approach to clarifying the disposition of the 5 year weight loss data

- A subset of the previously analyzed 5 year samples could be cleaned using multiple cleaning cycles to determine if additional weight loss is observed compared to the existing data set
  - It is proposed that samples from the SCW and SDW aqueous environments be analyzed as the cleaning procedure for these exposure conditions has been characterized using control samples
  - It is proposed that six weight loss samples and six crevice samples from each environment be analyzed initially: 24 total samples represents approximately 20% of the previously analyzed 5 year samples