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## CONSTANT EXTENSION RATE TESTING OF TYPE 304L STAINLESS STEEL IN SIMULATED WASTE TANK ENVIRONMENTS (U)

by

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## **CONSTANT EXTENSION RATE TESTING OF TYPE 304L STAINLESS STEEL IN SIMULATED WASTE TANK ENVIRONMENTS**

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### **ABSTRACT**

New tanks for storage of low level radioactive wastes will be constructed at the Savannah River Site (SRS) of AISI Type 304L stainless steel (304L). The presence of chlorides and fluorides in the wastes may induce Stress Corrosion Cracking (SCC) in 304L. Constant Extension Rate Tests (CERT) were performed to determine the susceptibility of 304L to SCC in simulated wastes. In five of the six tests conducted thus far 304L was not susceptible to SCC in the simulated waste environments. Conflicting results were obtained in the final test and will be resolved by further tests. For comparison purposes the CERT tests were also performed with A537 carbon steel, a material similar to that utilized for the existing nuclear waste storage tanks at SRS.

**Key words:** Nuclear waste tanks, stress corrosion cracking, constant extension rate tests, 304L stainless steel

### **INTRODUCTION**

Environmental concerns over the disposal of radioactive wastes has resulted in the development of new technology for waste processing and storage. At the

Department of Energy's (DOE) Savannah River Site (SRS) two such facilities were designed to accept a variety of high level and low level wastes. The first facility mixes processed waste with glass frit which is melted to encapsulate the waste in borosilicate glass.<sup>1</sup> Other processed wastes are mixed with a cement-like material known as saltstone.

An important step in these processes is the preparation of the feed to the facilities. Figure 1 illustrates the proposed waste processing flow loop. Inorganic salt slurries will be processed by either In-Tank Precipitation (ITP) or the Extended Sludge Process (ESP). As part of the processing, the salt slurries will be washed to remove the insoluble salts. Two large tanks will be necessary to hold these wash waters until they are recycled back to the processing tanks. These wash waters will have a low level of radioactivity and be more dilute than the salt slurry fed to the processing tanks. A third small tank will be used to feed the cleaning solution wastes from the Receiving Basin for Off-site Fuels (RBOF) to the cesium removal column. The available facilities for holding these low level radioactive wastes are single-walled carbon steel tanks.

Laboratory tests and in-service observations have revealed that carbon steel is susceptible to localized attack in dilute, uninhibited waste solutions. Coupon tests performed in simulated wastes were susceptible to pitting in the wetted film region above the water line.<sup>2</sup> The attack was induced by nitrate and resulted from the depletion of hydroxide inhibitor which occurred due to the absorption of CO<sub>2</sub> into the wetted film. Sodium nitrite was determined to be an effective inhibitor for nitrate corrosion. Excess nitrite, however, was shown to decrease the amount of organic material that could be removed during the glass making process. The presence of these organic compounds have detrimental effects on both the quality of the glass product and the operation of the melter equipment.<sup>3</sup> A proposal was made to construct new wash water holding tanks to minimize the need for excess inhibitor additions and to reduce surveillance frequency. The candidate material of construction for the new tanks is AISI Type 304L stainless steel (304L).

In addition to passivating anions, such as nitrite and hydroxide, the wash waters contain chlorides and fluorides which may induce localized attack of 304L (see Table 1). Since the wastes contain different passivating and aggressive anions and concentration ranges, reliable corrosion predictions cannot be based on previous results for 304L. One form of localized corrosion that is of particular concern is stress corrosion cracking (SCC). Constant Extension Rate Tests (CERT) were used to determine the susceptibility of 304L to SCC in these wastes.

The CERT technique provides a rapid assessment of a material's stress corrosion cracking susceptibility. For this technique, a tensile specimen is placed in the suspect environment and then strained to failure at rates between 10<sup>-4</sup>-10<sup>-7</sup> s<sup>-1</sup>. Metallographic examination of the fracture surface is then necessary to identify

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the presence of cracking.<sup>4</sup> Cracking susceptibility is also indicated by a decrease in mechanical properties compared with those observed in an inert environment. These properties include ultimate tensile strength, elongation at failure, and reduction of area. The presence of secondary cracks along the gage length of the specimen may also indicate SCC susceptibility.

The critical parameter for these tests is the strain rate. For each material-environment system a critical strain rate range exists within which cracking will occur.<sup>5</sup> The literature suggests that stainless steels in chloride environments will exhibit cracking at strain rates of approximately  $10^{-6}$  s<sup>-1</sup>.<sup>6</sup> This strain rate was chosen for the tests with 304L in the waste environments. As a comparison, the tests were also conducted with A537 carbon steel (A537) at the same strain rate. A537 is similar to the material of construction for the majority of the existing nuclear waste storage tanks at SRS.

## EXPERIMENTAL

The CERT tests were performed on a Cortest Corrosion Fatigue System. The system included a load cell, a Linear Variable Displacement Transducer (LVDT) measured, a signal conditioner for the load cell and the LVDT, and a temperature controller. Cortest software was utilized to acquire and store the data on a personal computer. A high temperature vessel constructed of Hastelloy C-276 with pyrex chamber walls contained solutions that were above ambient temperature.

Simulants of the actual waste streams were tested. The new tanks will receive wash waters from ITP and ESP and a mixture of three cleaning solution wastes from the RBOF. A waste from each source was tested. The concentration levels were selected to represent either the maximum halide ion content or the minimum inhibitor content for a particular waste. Table 1 lists the components and the concentrations of the tested wastes. Temperature is also an important variable for SCC. The tests were conducted at the maximum solution temperature expected in the tanks, 60° C, since SCC susceptibility increases with temperature.

The composition of the 304L and the A537 tensile specimens are given in Table 2. The 304L was tested in both the solution annealed and furnace sensitized conditions. The heat treatment was performed to simulate the microstructure in weld heat affected zones. Maximum sensitization, as determined by electrochemical potentiodynamic reactivation was achieved after heat treating a specimen at 650° C for 6 hours. Round bar tensile specimens with a one inch gage length and 0.25 inch diameter were used for the tests.

## RESULTS

The CERT tests were completed in three of the simulated solutions. The results of the tests are shown in Table 3. The strength and ductility of solution annealed 304L were observed to be independent of the test solution. In each case the specimen had a "cup and cone" appearance characteristic of ductile fracture. The fracture surface of the specimens tested in the ESP and the Resin Regeneration (RR) waste indicated ductile failure (see Figure 2). The specimen tested in the cask decontamination (CD) waste, however, showed evidence of cracks along the outer edge of the fracture surface. The relatively high fluoride concentration may be responsible for this attack. The inconsistency between the mechanical properties and the fracture surface for this test solution will be addressed in further tests. The strength, ductility, and the fracture mode for the heat treated specimens were independent of the test solution. The agreement between these parameters indicates that, at the test strain rate, furnace sensitized 304L is not susceptible to SCC.

The strength, ductility, and fracture mode for A537 specimens tested in the ESP and the RR solutions were similar. The specimens exhibited the characteristic "cup and cone" appearance. An example of the dimpled, ductile fracture surface is shown in Figure 3. For the CD solution the specimen demonstrated a significant decrease in strength and ductility. Figure 4 shows that the specimen gage length did not have a "cup and cone" appearance and that secondary cracks developed. These results however conflicted with observations of the surface which indicated ductile fracture occurred. Further tests will be done to resolve this conflict.

## CONCLUSIONS

CERT tests were performed on 304L and A537 in simulated waste environments. The tests performed thus far indicate that 304L is not susceptible to SCC in the wash waters from the ESP and RR processes. Further tests need to be performed on 304L in CD waste to resolve discrepancies in the observations. The tests conducted on A537 in ESP and RR also showed no susceptibility to SCC. Results from the tests performed in the CD waste did however indicate that A537 was susceptible to attack in that environment.

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Table 1.  
Molar Anion Compositions of Low Level Hazardous Waste

Solution Number	ESP 1	ITP 4	RR 8	TTC 11	CD 13
pH	13.7	13.5	12.7	12.4	12.5
OH <sup>-</sup>	2.1	1.3	0.15	-	-
CO <sub>3</sub> <sup>=</sup>	0.1	0.16	0.098	-	-
NO <sub>2</sub> <sup>-</sup>	1.1	0.6	0.07	-	-
NO <sub>3</sub> <sup>-</sup>	1.4	2.0	0.7	-	4.6
Cl <sup>-</sup>	0.022	0.022	0.0013	-	-
F <sup>-</sup>	0.011	0.015	-	-	0.039
SO <sub>4</sub> <sup>=</sup>	0.095	0.14	0.0079	-	-
Al(OH) <sub>4</sub> <sup>-</sup>	0.3	0.31	0.007	-	0.26
C <sub>2</sub> O <sub>4</sub> <sup>=</sup>	0.0051	0.014	-	-	-
CrO <sub>4</sub> <sup>=</sup>	0.0021	0.0033	0.00084	0.013	-
MoO <sub>4</sub> <sup>=</sup>	0.00027	0.00043	-	-	-
SiO <sub>3</sub> <sup>=</sup>	0.0021	0.0038	0.00058	-	-
PO <sub>4</sub> <sup>-3</sup>	0.0058	0.0085	0.014	0.22	-

Notes:

**Solution Designation**

ESP- Extended Sludge Processing slurry

ITP- In-Tank Precipitation slurry

RBOF- Receiving Basin for Off-site Fuels wastes:

RR- Resin Regeneration waste

TTC- Tritium Target Cleaning waste

CD- Cask Decontamination waste

Table 2.  
Analysis of Materials Tested (wt. %)

	C	Mn	Si	P	S	Cr	Ni	Mo	Fe	Cu
304L	.025	1.34	.38	.032	.026	18.3	8.96	0.35	bal.	0.43
A537	0.22	1.25	.22	.022	.016	0.17	0.17	.03	bal.	0.28

Table 3.  
CERT Results for 304L and A537 in Simulated Waste Tank Environments.

Material	Solution	Tensile Strength (lbs/in <sup>2</sup> )	Strain at failure	Reduction of Area (%)	Fracture Mode
304L-SA	ESP	89,200	0.5703	77.5	Ductile
	RR	87,000	0.5612	79.4	Ductile
	CD	88,800	0.566		Cracks*
304L-FS	ESP	87,000	0.5727	74.3	Ductile
	RR	85,200	0.5612	75.6	Ductile
	CD	83,000	0.553	79.4	Ductile
A537	ESP	73,900	0.2764	56.4	Ductile
	RR	82,200	0.2407	55.4	Ductile
	CD	79,200	0.235	37.9	Cracks**

Notes:

SA - solution annealed

FS - furnace sensitized

\* - Cracks observed on outer edge of fracture surface

\*\* - Secondary cracks and reduction of ductility indicates attack occurred

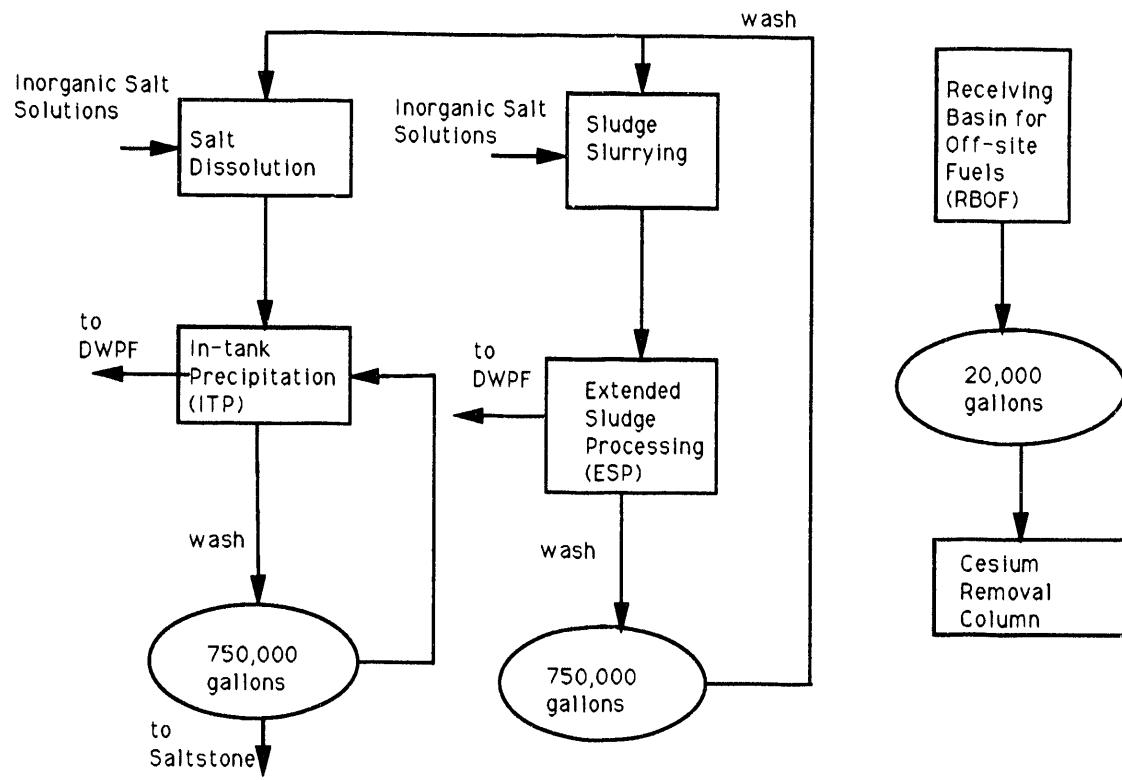


Figure 1. Waste Processing Flow Loop

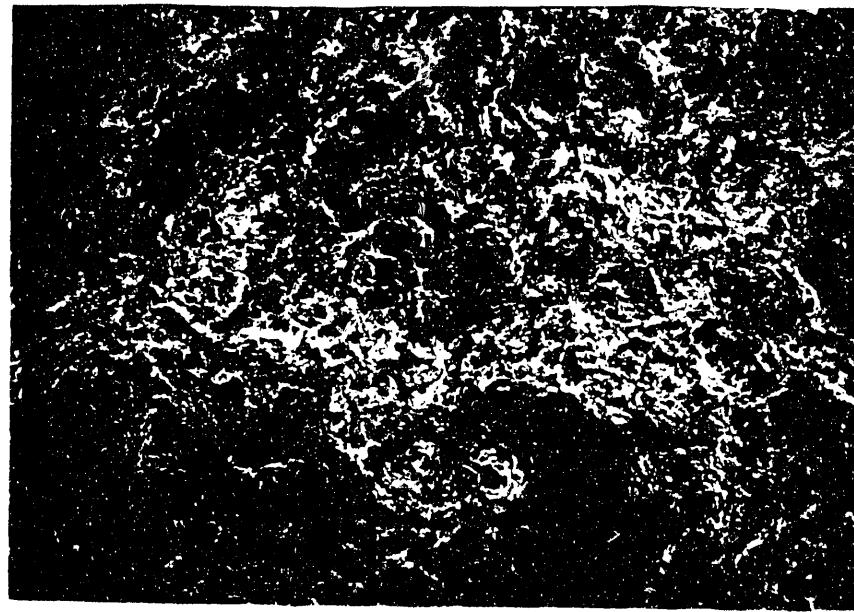


Figure 2. Fracture surface of 304L tensile specimen strained in RR waste solution at 60° C (100 X).

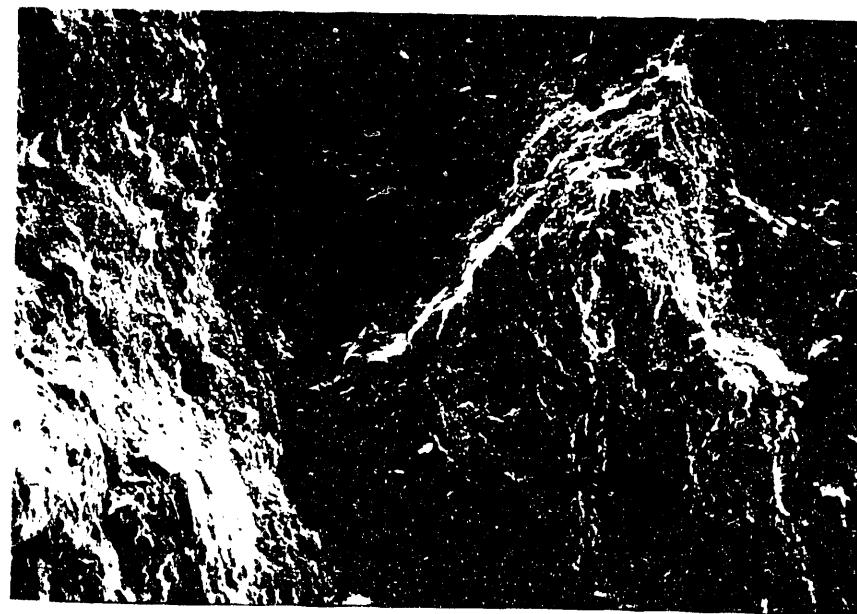


Figure 3. Fracture surface of A537 tensile specimen strained in ESP waste solution at 60° C (100 X).



Figure 4. Gage length of A537 tensile specimen strained in CD waste solution at 60° C (6.4 X)

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