WSRC-TR-97-0039
Revision 0

CORROSION RESISTANCE OF INCONEL 690 TO SODIUM CARBONATE, CALCIUM CARBONATE, AND SODIUM META SILICATE AT 900 AND 1100 °C (U)

KENNETH J. IMRICH

SAVANNAH RIVER TECHNOLOGY CENTER

Publication Date: January 29, 1997

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT DE-AC09-89SR18035
PRESENTLY UNDER CONTRACT DE-AC09-89SR18035

DISCLAIMER

DOCUMENT: WSRC-TR-97-0039 Revision 0

TITLE: CORROSION RESISTANCE OF INCONEL 690 TO SODIUM CARBONATE, CALCIUM CARBONATE, AND SODIUM META SILICATE AT 900 AND 1100 °C(U)

APPROVALS

K. J. Imrich, Author
Materials Applications & Corrosion Technology Group
Materials Technology Section

Date: 1/29/97

R.J. O'Driscoll, Customer Reviewer
Defense Waste Processing Engineering

Date: 2/6/97

N. C. Iyer, Manager
Materials Applications & Corrosion Technology Group
Materials Technology Section

Date: 1/30/97

T. L. Capeletti, Manager
Materials Technology Section

Date: 2/5/97

Dennis F. Bickford, Technical Reviewer
SRTC Vitrification Technology

Date: 2/6/97

WSRC-TR-97-0039
Revision 0

SMT
STRATEGIC MATERIALS TECHNOLOGY

Keywords:
DWPF
Melter
Corrosion
Tests
Cleaning
BLD-221S
Retention: Lifetime

CORROSION RESISTANCE OF INCONEL 690 TO SODIUM CARBONATE, CALCIUM CARBONATE, AND SODIUM META SILICATE AT 900 AND 1100 °C (U)

Kenneth J. Imrich

Issued:

January 29, 1997

T L Capo
Authorized Derivative Classifier

2/5/97
Date

SRTC SAVANNAH RIVER TECHNOLOGY CENTER, AIKEN, SC 29808
Westinghouse Savannah River Company
Prepared For The U.S. Department Of Energy Under Contract DE-AC09-89SR18035

Corrosion Resistance Of Inconel 690 To Sodium Carbonate, Calcium Carbonate, And Sodium Meta Silicate at 900 and 1100 °C (U)

Summary

Corrosive attack of Inconel 690 coupons was not observed following 3 day exposure tests to calcium carbonate, sodium carbonate, and sodium meta silicate at 900 °C. However, melt line attack was evident on coupons exposed to sodium meta silicate and sodium carbonate tested for 3 days at 1100 °C. In addition, intergranular attack (IGA), approximately 0.67 mils/day, was observed on the Inconel 690 coupon exposed to calcium carbonate at 1100 °C.

Calcium carbonate did not completely remove the glass coating at 950 °C. In fact, it was comparable to the results obtained by exposing a glass coated coupon at 950 °C in air. Therefore, calcium carbonate is not recommended for cleaning the DWPF melter pour spout. Both sodium carbonate and sodium meta silicate appear to remove most of the glass. However, these cleaning agents will remain on the metal surface following exposure at 950 °C resulting in a very rough surface and a potential for corrosive attack when heated to 1100 °C.

Background

The Materials Technology Section (MTS) was requested by Vitrification Technology to evaluate the corrosion resistance of Inconel 690 exposed for 3 days to sodium carbonate (Na_2CO_3 anhydrous), calcium carbonate ($CaCO_3$) and sodium meta silicate ($NaSiO_3 \cdot 9H_20$) at 900 and 1100 °C. In addition, preoxidized Inconel 690 coupons were coated with a DWPF glass and then exposed for 1 day to the chemical cleaning agents at 950 °C. Glass coating was accomplished by sprinkling ground glass from a DWPF cold run canister on one side of the coupons and heating them in a furnace at 900 °C for 3 days. The coated coupons were drilled to allow insertion of a 0.032" Inconel 690 weld wire which was used to suspend the coupons in the melt while keeping them from contacting the bottom of the crucible. This latter test was used to evaluate the cleaning effectiveness of the various compounds. All tests were performed in alumina crucibles except for the 1100 °C sodium carbonate test which was conducted in a platinum crucible. Coupons, 0.1275 inch thick by 3.00 " long by 1.00" wide, for the corrosion and cleaning tests were fabricated from Inconel 690. The elemental composition of the base material, obtained from TNX, is presented in Table 1. Similar tests were performed using borax, boric acid, and boron nitride[1]. All work was performed under Technical Task Request TTR-96-0093. Results of the scoping tests are summarized in this report.

Corrosion Testing Results

900 °C Corrosion Test

Coupons from the sodium meta silicate and the sodium carbonate 900 °C corrosion were covered with crusty scale below the air interface. The sodium meta silicate coupon also had a very thick scale on the portion exposed in the vapor space. The coupon placed in the crucible containing calcium carbonate showed no visible change (i.e. the oxide layer was still visible). The calcium carbonate did not melt, most likely calcined, and remained powdery following this test. Metallographic examination of the three coupons did not reveal any evidence of significant corrosive attack. Micrographs can be found in Lab Notebook WSRC-NB-93-30.

1100 °C Corrosion Test

The calcium carbonate again did not appear to melt and the coupon showed no visible evidence of external corrosive attack. Microstructural evaluations above and below the air/sodium carbonate interface exhibited a shallow IGA (approximately 2 mils in 3 days). The coupons exposed in sodium meta silicate and the sodium carbonate, however; exhibited significant melt line attack with a remaining coupon thickness of 0.0614" and 0.1106", respectively (original nominal wall thickness 0.1275"). A very thick deposit formed above and below the melt line on the coupon exposed in the sodium meta silicate. This deposit consisted of nickel oxide and sodium chromium oxide hydrate. A thinner scale, consisting of nickel chromium oxide and sodium carbonate, was observed on the coupon placed in sodium carbonate. Intergranular attack (approximately 3 mils in 3 days) was observed in the vapor and melt regions of the coupon.

950 °C Chemical Cleaning Test

The preoxidized, glass coated coupons were suspended in the various cleaning agents and evaluated for cleaning effectiveness after a one day exposure test at 950 °C. As was the case during the 900 °C corrosion tests, the calcium carbonate calcined and remained powdery following the test. The appearance of the coupon was not significantly different than the glass coated control sample that was placed in the furnace in an empty crucible. A thin glass coating, much thinner than original glass coating, remained on both the control and calcium carbonate test coupons. There was no externally visible corrosive attack of the coupon. A metallurgical evaluation of this coupon was not performed.

The sodium carbonate exposed coupon was covered above and below the melt line with a black scale and randomly spaced yellowish green deposits. This scale was very difficult to remove. Samples of the scale including the yellow deposits were analyzed and found to consist entirely of sodium carbonate. Most of the glass was removed from the specimen and there was no visible degradation (i.e. melt line or pitting attack). A metallurgical evaluation of this coupon was not performed.

Similar to the 900 °C corrosion test, the sodium meta silicate did not become fully molten during the 950 °C cleaning test. The coupon was almost completely covered with a rough, thick, tenaciously adhering black scale. Some yellowish green deposits were also observed. This layer was much thinner than that observed on the coupon from the 1100 °C corrosion test. Some

evidence of melt line attack was observed. Glass was still adhering to both sides of the lower 0.25" of this coupon. A metallurgical evaluation of this coupon was not performed.

Discussion

Corrosion resistance of the Inconel 690 exposed to the sodium carbonate and the sodium meta silicate can vary dramatically between 900 and 1100 °C. The corrosion resistance of the Inconel decreases at higher temperatures. This is evident by the melt line attack observed on both coupons at 1100 °C. Sodium meta silicate produced the most severe melt line attack (approximately 11 mils/day penetration). Testing also revealed that some of the chemical cleaning agents, especially the sodium carbonate, remain on the surface of the Inconel 690 following cleaning at 950 °C and therefore, corrosion may be expected upon reheating to 1100 °C. Although melt line attack was the primary degradation mechanism of the coupons exposed to the sodium carbonate and sodium meta silicate at 1100 °C, intergranular attack was also observed. Coupons exposed in the calcium carbonate experienced only minor intergranular attack. However, there was no difference in the amount of glass removed from the coated coupon exposed to calcium carbonate when compared to the control sample (glass coated coupon exposed in air only at 950 °C). Therefore, there is no advantage to using calcium carbonate as a chemical cleaning agent. The ineffectiveness to remove glass and lack of significant corrosive attack was attributed to the apparent calcination of the calcium carbonate.

Conclusions

Based on the results of this study the conclusions are as follows:

1) Corrosion of Inconel 690 was not observed following a 3 day exposure to calcium carbonate, sodium carbonate or sodium meta silicate at 900 °C.

2) Melt line attack was observed on the Inconel 690 coupons exposed to sodium carbonate and sodium meta silicate for 3 days at 1100 °C.

   - Sodium meta silicate - 11 mils/day penetration.
   - Sodium carbonate - 3 mils/day penetration and 1 mil/day IGA.

4) IGA attack (approximately 0.67 mils/day) was observed on the coupon exposed to calcium carbonate at 1100 °C.

5) Calcium carbonate does not effectively remove glass from Inconel 690 at 950 °C.

6) Deposits of sodium carbonate and sodium meta silicate remained on the glass coated coupons following a 3 day exposure at 950 °C. These deposits may result in corrosion of the Inconel when heated to 1100 °C.

References

1. K.J. Imrich, Corrosion resistance of Inconel 690 to borax, boric acid, and boron nitride at 1100 °C (U), WSRC-TR-96-0394, December 12, 1996.

Table 1. Elemental composition of the sheet material used to fabricate the corrosion coupons*.

Ni	Cr	Fe	Si	Mo	Ti	Mn	Al	Cu	Nb
60.398	29.027	8.859	0.318	0.386	0.350	0.163	0.077	0.174	0.196

* Product analysis performed on a Rigaku 3271 Wavelength Dispersive X-Ray Fluorescence Spectrometer using a Inconel 690 NIST traceable standard BM690 manufactured by the Brammer Standards Company.

WSRC-TR-97-0039
Revision 0

DISTRIBUTION:
R.J. O'Driscoll, 704-30S
J.E. Owen, 704-30S
W.D. Kerley, 704-25S
D.B. Little, 210-S
J.F. Ortaldo, 704-S
D.C. Iverson, 704-30S
J.F. Sproull, 704-30S
J.T. Gee, 704-25S
R.E. Edwards, 704-25S
C.T. Randall, 704-T
S. Wood, 773-A
G.T Wright, 773-A
L.M. Papouchado, 773-A
T.J. Lex, 719-4A
J.P. Morin, 719-4A
E.W. Holtzscheiter, 773-A
L.F. Landon, 704-1T
M.J. Plodinec, 773-A
D.P. Lambert, 704-1T
C.R. Wolfe, 773-A
T. L. Capeletti, 773-41A
N. C. Iyer, 773-A
D.T. Rankin, 773-A
R.L. Bickford, 730-A
M.A. Kyle, 723-A
T.A. Nance, 723-A
P.S. Hebert, 723-A
D.F. Bickford, 773-43A
C.F. Jenkins, 730-A
G.T. Chandler, 773-A
MTS Files, 773-A
TIM, 703-43A (4)