

RECORDS ADMINISTRATION



R0596492

WSRC-TR-96-0394  
Revision 0

*Strategic Materials*

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

**CORROSION RESISTANCE OF INCONEL 690 TO BORAX, BORIC  
ACID, AND BORON NITRIDE AT 1100 °C (U)**

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Issued:

December 12, 1996

*D. Thomas Rankin*

Authorized Derivative Classifier

*December 16, 1996*

Date

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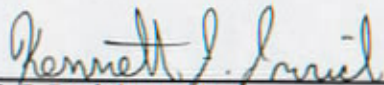
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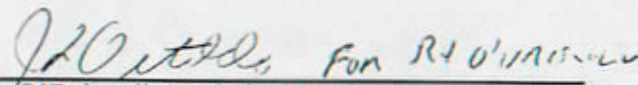
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TITLE: CORROSION RESISTANCE OF INCONEL 690 TO BORAX,  
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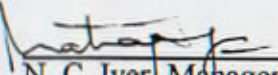
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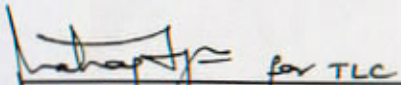
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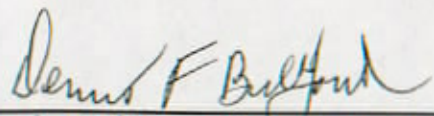
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# **Corrosion Resistance of Inconel 690 to Borax, Boric Acid, and Boron Nitride at 1100° C (U)**

by

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DOE Contract No. **DE-AC09-89SR18035**

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ACID, AND BORON NITRIDE AT 1100 °C (U)**

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Publication Date: December 12, 1996

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PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT DE-AC09-89SR18035  
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**CORROSION RESISTANCE OF INCONEL 690 TO BORAX, BORIC ACID,  
AND BORON NITRIDE AT 1100 °C (U)**

**Summary**

Significant general and localized corrosion was observed on Inconel 690 coupons following exposure to borax, boric acid and boron nitride at 1100 °C. Severe localized attack at and below the melt line was observed on coupons exposed to borax. An intergranular attack (IGA) of the Inconel 690 was also observed. Severe internal void formation and IGA (30 mils penetration after 3 days) was observed in the coupon exposed to boric acid. Both borax and boric acid remove the protective chromium oxide; however, this layer can be reestablished by heating the Inconel 690 to 975 °C in air for several hours. Inconel 690 in direct contact with boron nitride resulted in the formation of a thick chromium borate layer, a general corrosion rate of 50 to 90 mils per year, and internal void formation of 1 mil per day.

Based on the results from the corrosion tests the recommendations are as follows:

- Chemical cleaning using borax, boric acid, or boron nitride is not recommended in regions of the DWPF melter pour spout that are at a temperature of 1100 °C. Further testing of the candidate chemical cleaning agents below 900 °C is required before they are approved for use in the lower temperature regions of the DWPF melter pour spout.
- Boron nitride should not be used for extended periods of time (i.e. as insert material) because degradation of the Inconel 690, especially the pour spout knife edges, may result.
- Use of any technique that results in the removal of the protective chromium oxide film, e.g. mechanical cleaning, should be minimized and when used, carefully performed.
- Effects of the chromium borate layer on the glass pouring characteristics and corrosion of the Inconel should also be evaluated.

**Background**

The Materials Technology Section (MTS) was requested by Vitrification Technology to evaluate the corrosion resistance of Inconel 690 exposed to anhydrous boric acid, anhydrous borax, and boron nitride at 1100 °C, the temperature representative of the knife edge region of the DWPF melter pour spout. This work was performed under Technical Task Request TTR-96-0093. Vitrified waste production rates from the DWPF melter have decreased and have been attributed to the build-up of glass and spinels in the pour spout. Mechanical cleaning of the pour spout is currently being used but has not been entirely successful in removing all the material, and therefore, chemical cleaning of the pour spout is being investigated. To evaluate the corrosion



resistance of Inconel 690 to the various candidate chemical cleaning agents, a small scoping test was initiated.

In addition to concerns over chemical cleaning, questions were raised about the effects of the boron nitride insert on the DWPF melter Inconel 690 pour spout. This insert was placed near the pour spout upper knife edge for approximately six days earlier this year to evaluate glass flow stream characteristics. During this test, glass appeared to be fluxed from around the insert. Because of this observation, concerns were raised about possible degradation of the Inconel 690 pour spout. To determine the corrosion rate, a set of coupons (Inconel 690 weld wire) was exposed to boron nitride for six days. Results of this test and the chemical cleaning scoping tests are summarized in this report.

### **Corrosion Testing**

#### **Chemical Cleaning Qualitative Corrosion Resistance Tests**

Coupons for the chemical cleaning coupon tests were fabricated from Inconel 690 and welded using matching Inconel 690 filler material (P.O. No. AXC 772W, Material Cert. No 73778). The elemental composition of the base material, obtained from TNX, is presented in Table 1. A weld was made traversing the width in the middle of the coupons (0.1275 inch thick by 2.25" long by 1.00" wide). Two larger coupons, 2.00" by 2.25" by 0.1275", were fabricated with a vertical weld. All the coupons were oxidized at 975 °C for several days to establish a uniform chromium oxide layer. Boron nitride samples were made with similar dimensions to that of the smaller 690 coupons. A coupon was also made from a portion of the unheated lowermost section of the DWPF melter pour spout. This component was removed from the DWPF melter in 1995 after a through-wall penetration was found.

The first chemical cleaning corrosion test was setup using one boron nitride coupon and one of the smaller Inconel coupons. These coupons were placed in a partially covered alumina crucible several inches apart and held at 1100 °C for 3 days. Similar coupons were partially submerged in anhydrous boric acid and anhydrous borax (a platinum crucible was finally used with the borax) for 3 days at 1100 °C. Following the boric acid and borax immersion tests, coupons were suspended in an alumina crucible and reheated to 975 °C for several hours to evaluate how effectively the cleaning agents could be removed from the surface of the coupon. This test was incorporated to simulate the knife edges following a cleaning cycle. All coupons were visually examined following the tests and deposits from selected samples were analyzed using x-ray diffraction (XRD) and energy dispersive spectroscopy (EDS) on a scanning electron microscope (SEM). Metallurgical evaluations were also made using optical microscopy and a traveling microscope.

#### **Inconel 690 / Boron Nitride Corrosion Rate Test**

Inconel 690 weld wire (0.1237" diameter) was used to make four coupons for the boron nitride/Inconel 690 corrosion rate test. These coupons were approximately 1.5" long. Two of the four coupons were oxidized for 3 days at 975 °C prior to exposure to the boron nitride. Four holes approximately 0.125" in diameter by 2" deep were drilled into a boron nitride block. The coupons were placed into the block and the holes were filled with boron nitride powder



completely encapsulating the coupons. These coupons were placed in a furnace at 1100 °C for six days. Coupons were weighed on the calibrated scale, M&TE No. 3-1960, using weights, M&TE No. MT-253, for verification.

## Results

### **Chemical Cleaning Qualitative Corrosion Resistance Tests**

Visual examination of the coupon exposed to boron nitride exhibited a heavy black scale on all surfaces. This scale was identified by XRD and consisted primarily of chromium borate. Some chromium oxide was also identified. Results of the test are summarized in Table 2. Additional data can be found in Lab Notebook WSRC-NB-93-30. Portions of the chromium borate layer spalled off during cooling, exposing the tenaciously adhering grey chromium oxide scale. This oxide was more metallic in appearance than the original oxidized coupon. Scanning electron images of the two layers are shown in Figures 1, 2, and 3. The outer layer consisted of various geometric shapes, consisting mainly of Cr, Ni, Al, Ca, O, in a matrix composed of Al, Ca, and O. A small amount of Si was identified in both of these samples. Elemental analyses were performed on a SEM (the light element detector cannot detect boron). The grey layer beneath consisted primarily of Cr and O, although some aluminum was evident. Lighter colored particles randomly embedded in the matrix contained Ni, Cr, and Fe similar to that found in Inconel 690. Metallography revealed no evidence of localized attack; however, internal voids were observed to an average depth of 2 mils (Figure 4).

The coupon exposed in the borax exhibited significant melt line attack of both the weld and base metal. The exact exposure time for this test is not known since the alumina crucible failed by corrosive attack at the melt line during the first evening. The maximum time of exposure was 14 hours, however, the failure most likely occurred during the first eight hours. In any case, the observed corrosive attack of the Inconel 690 was highly localized and aggressive. A metallurgical specimen was prepared and thickness measurements were taken using a traveling microscope. Thickness measurements were: 0.1265" above the melt, 0.1126" at the melt line, and 0.1201" below the melt line (initial coupon thickness was 0.1275"). A metallographic specimen prepared revealed an intergranular attack (IGA) to an average depth of 4 mils. This attack occurred in less than 6 days. Scales were identified and consisted of mainly nickel iron oxide borate and a smaller amount of chromium oxide. A second coupon was exposed in the borax; however, this test was conducted in a platinum crucible (3 days at 1100 °C). The coupon again exhibited severe localized attack both at and below the melt line. The platinum crucible showed no evidence of corrosive attack. The coating that remained on the coupon was identified by XRD and was comprised of a nickel iron oxide borate with lesser amounts of chromium oxide. This coupon was placed back into the furnace for two hours at 1100 °C in an attempt to remove this coating. Most of the coating was removed but some still remained around the bottom edge.

The coupon placed in the boric acid contained a very thick greenish white deposit. This deposit contained chromium borate and was easily removed from the surface of the coupon at room temperature, leaving a thin grey boron oxide layer. The original chromium oxide was completely removed as a result of this exposure. Localized corrosion, i.e. pitting or melt line attack, was not evident. Coupon thickness above and below the melt line was 0.1269", corresponding to a corrosion rate of 0.1 mils per day. Metallographic examination of this coupon revealed a severe



internal attack. Both IGA and internal void formation were present with an average depth of attack of 30 mils (Figure 5), for an average rate of attack of 10 mils per day.

### **Inconel 690 / Boron Nitride Corrosion Rate Test**

The four coupons removed from the boron nitride block contained a thick black outer scale, most likely chromium borate, and an underlying grey tenaciously adhering scale. The inner scale was identified as chromium oxide. Corrosion rates, based on weight loss, for the four coupons ranged from 50 to 90 mils per year. Metallographic and SEM analyses revealed an internal attack of the Inconel 690 to an average depth of 6 mils (Figure 6). Chromium oxide was observed in several of the voids. Results from the 690 / boron nitride corrosion rate test are presented in Table 3.

### **Discussion**

The testing of coupons at 1100 °C exposed to boron nitride, anhydrous boric acid, and anhydrous borax exhibited evidence of corrosive attack of varying degrees. The most severe attack was on the coupon that was exposed to the borax. This coupon exhibited significant localized corrosion at and below the melt line (both base metal and weld fusion zones were attacked) and an intergranular attack of the base metal. The nickel iron oxide borate coating that covered the coupon following the test could not be thoroughly removed by heating to 1100 °C for two hours. This glassy coating remained on the lower edge of the coupon and may possibly result in further corrosion if the coupon were reheated. Due to the unpredictable, localized nature of the corrosive attack resulting from exposure to borax, the use of this chemical to clean the 1100 °C regions of the DWPF melter pour spout is not recommended.

Boric acid exposure resulted in the complete removal of the original chromium oxide layer. The thin boron oxide layer that remained on the coupon after cleaning at room temperature did not inhibit passivation of the coupon when reheated. Boric acid even resulted in partial removal of the heavy scale that covered the coupon sectioned from the unheated portion of the DWPF melter pour spout. The removal of the original chromium oxide layer and repassivation of the metal indicates that oxidation of the Inconel will still occur after boric acid exposure. Severe IGA and internal void formation (resulting from chromium depletion), to depths greater than 30 mils in only three days, should exclude boric acid from use in the 1100 °C region of the pour spout.

Boron nitride exposure resulted in high corrosion rates (50 to 90 mils per year) as measured by weight loss. The scatter in the reported corrosion rates resulted from incomplete removal of the tenaciously adhering chromium oxide layer. Aluminum observed in the chromium borate layer resulted from dissolution of the alumina crucible. Inconel coupons in intimate contact as well as coupons separated from the in boron nitride showed signs of internal void formation. The attack was most severe in the coupons that were in contact with the boron nitride, with an average depth of penetration of 6 mils (1 mil per day). Inconel 690 is moderately resistant to oxidation at 1100 °C, therefore, some of the voids may have resulted from oxidation. Oxidation is the primary degradation mechanism in the upper portion of the pour spout. Void formation results as chromium diffuses from the metal substrate to the surface of the metal to form chromium borate and chromium oxide. The pour spout knife edges may be especially vulnerable to internal attack resulting from corrosion and/or oxidation since they are exposed to the environment on two sides.



The stability and morphology (i.e. porosity) of the chromium borate layer will effect the diffusion of chromium thereby effecting the corrosion rate. If the chromium borate layer does not degrade from mechanisms such as thermal expansion mismatches, abrasive wear, thermal shock, internal stresses, or corrosion the driving force for continued diffusion of chromium from the base metal would most likely decrease. This study did not evaluate the stability or morphology of this layer, and therefore, further testing would be required to accurately determine the effects of chromium borate on the corrosion rate of Inconel 690. Furthermore, the porous structure created by the internal voids in the metal may trap these salts and increase the possibility of accelerated corrosion. The formation of the chromium borate or the various oxide scales which form in the pour spout may also influence glass pouring characteristics and should be investigated if chemical cleaning is pursued.

The complex environmental conditions that exist in the pour spout are difficult to model and were not evaluated as part of this study. Both chloride and sulfate salts have been identified on samples taken from the unheated portion of the DWPF melter pour spout above the bellows and scrapings removed from the pour spout during hot operations. The salts condense at temperatures less than 900 °C and concentrate, increasing the probability of corrosive attack. Temperatures in the lower portion of the pour spout are at or below this temperature. The failure of the unheated portion of the pour spout above the bellows during cold runs (penetration in approximately 8 months of service) and the borescope outer housings (corrosion rate in excess of 360 mils/year) were attributed to an accumulation of salts which attacked the protective chromium oxide layer and the underlying Inconel 690. Removal of the oxide layer from the unheated portion of the pour spout was attributed to thermal cycling (thermal shock due to steam cleaning and intermittent pouring) and repeated intense mechanical cleaning. The attack of the Inconel 690 by the salts is dependent on concentration and temperature. The conditions in the pour spout especially in the cooler sections may be conducive for condensation of the salts and localized accelerated corrosion.

The glass and hard dense spinel crystals in the upper portion (> 900 °C) of the pour spout may act as a protective layer and minimize oxidation of the Inconel. This assumes that the spinel/glass layer is not porous and that all surfaces are completely covered with these deposits. Chemical and mechanical cleaning are intended to remove these deposits from the surface of the metal. However, the cleaning agents (and possibly the mechanical cleaning tools) will remove the protective oxide scales. Controlled application of the chemical cleaning agents would be difficult since crystal/glass buildup may not be uniform in the upper region of the melter. Areas of exposed metal may be subject to the corrosive cleaning agents while other areas are still covered with glass and spinel deposits. Exposed metal surfaces will therefore be degraded by the chemical cleaning agents evaluated in this study and normal oxidation at 1100 °C. As stated before, the knife edges are particularly vulnerable to degradation. Therefore, institution of a chemical cleaning program using the borax, boric acid or boron nitride in this region would result in corrosive attack of the Inconel 690 knife edges. Attack of the Inconel by the boric acid and borax is so rapid that even limited use is not recommended. Although the corrosion resulting from the boron nitride is more predictable, the possible degradation of the knife edges also excludes its use on a limited basis.

Devitrified glass, a mixture of glass and crystals, is present in the lower region of the pour spout (below 900 °C). Oxide scales in this region protect the Inconel from attack by the chloride and sulfate salts that condense below this temperature. The glass deposits may also provide some protection from the aggressive salts. However, if the deposits and the protective oxide scales are removed, the salts will attack the base metal. Tests should be conducted to determine if the



chemical cleaning agents will remove the protective deposits and oxide scales below 900 °C. Mechanical cleaning has been reasonably successful in removing the glass deposits from the lower portion of the pour spout. However, mechanical cleaning techniques may also remove the protective scales (as evidenced by the degradation of the unheated portion above the bellows during cold runs). Therefore, mechanical cleaning of the pour spout should only be used when absolutely necessary and performed in a way that minimizes oxide scale removal.

In summary general and/or localized corrosion of Inconel 690 will result from chemical cleaning at 1100 °C with any of the cleaning agents evaluated in this scoping test. The environment inside the DWPF melter pour spout is complex (i.e. thermal gradients, thermal cycling, molten glass, and volatile aggressive species from the glass). The effects of these variables alone would be expected to adversely affect degradation of the pour spout. A further increase in corrosion rate may be expected if the protective oxide layer is removed by either chemical or mechanical cleaning. Based on the results of this study chemical cleaning of the upper region of the pour spout (1100 °C) is not recommended. Further testing at lower temperatures is recommended before chemical cleaning of the lower temperature regions of the melter pour spout is attempted.

### **Conclusions and Recommendations**

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

- 1) Significant localized and/or general corrosion was observed in the Inconel 690 following exposure to borax, boric acid, and boron nitride at 1100 °C.
  - Exposure to borax resulted in severe localized corrosion at and below the melt.
  - Exposure to boric acid resulted in deep internal void formation from chromium depletion and intergranular attack. Boric acid also resulted in the removal of the chromium oxide scale.
  - Exposure to boron nitride whether in intimate contact or separated by several inches, resulted in the formation of a thick chromium borate layer and internal voids (2 to 6 mils in depth).
  - The corrosion rate of Inconel 690 when exposed to boron nitride at 1100 °C ranged from 50 to 90 mils per year.
- 2) Chromium oxide was removed from the coupons exposed to boric acid and borax. This layer would reform when the coupons were reheated to 975 °C.
- 3) Chemical cleaning using borax, boric acid, or boron nitride is not recommended in regions of the DWPF melter pour spout that are at a temperature of 1100 °C.
- 4) Boron nitride should not be used in the pour spout at 1100 °C as an insert material because degradation of the exposed pour spout knife edges may result.
- 5) Further testing at temperatures below 900 °C is required before these chemical cleaning agents are approved for cleaning the lower temperature regions of the pour spout.



- 6) Use of any technique that results in the removal of the protective chromium oxide film, e.g. mechanical cleaning, should be minimized and when used carefully performed.
- 7) Effects of the chromium borate on the corrosion rate of the Inconel 690 and the glass pouring characteristics should be evaluated.

**Table 1. Elemental composition of the material used to fabricate the various 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

**Table 2. Results corrosion tests results for Inconel 690 with borax, boric acid, and boron nitride.**

Test No.	Oxidized (975 C for 3 days)	Environment	Temperature (C)	Test Duration (days)	Deposits Analyzed by XRD or EDS	Comments
1a	Yes	Borax	1100	3 *	Deposits above and below the melt line consisted of Nickel iron oxide borate and chromium oxide.	Coupon exposed to borax less than 12 hours most likely 4 or 5 hours because the crucible failed. Severe melt line attack and heavy scale in both vapor and melt regions.
1b	Yes	air	975	3	NA	Section of coupon with melt line attack from 1a repassivated. Metallography revealed an intergranular attack to an average depth of 4 mils.
2a	Yes	Borax	1100	3	NA	Same test as 1a but coupon was placed in a Pt crucible. Localized attack on most of the coupon. Most severe attack was at the melt line. Coupon was dark green and contained a thick glassy coating (boric acid).
2b	Yes	air	1100	2 hours	Deposits consisted of boric acid. Scale primarily chromium borate.	Test to evaluate if deposits on 2a coupon could be completely removed. Most of the greenish glassy deposits were removed after exposure to 1100 C. However, a thin layer was still observed around the side edges and on the bottom of the coupon.
3a	Yes	Boron Nitride	1100	3	Scale consisted mainly of chromium borate with some chromium oxide.	The scale was thick and consisted of chromium borate and chromium oxide. This indicates that depletion of the chromium from the metal has occurred. Corrosion was uniform i.e. no localized attack.
3b	No	Boron Nitride	1100	4	Scale consisted mainly of chromium borate with some chromium oxide.	Scale that formed on this coupon (not preoxidized) looked similar to the scale on the coupon that was oxidized before exposure to the boron nitride.
4a	Yes	Boric Acid	1100	3	Scale contained mainly chromium borate; however, some hydrogen borate(boric acid) was identified. The thin grey layer which was under the chromium borate was boron oxide.	Coupon was covered with a very thick white and green deposit. This deposit could be fractured and removed easily at room temperature. Metal surface below deposit was light gray in color and smooth. IGA and void formation to depth of 30 mils.



**Table 2. Results corrosion tests results for Inconel 690 with borax, boric acid, and boron nitride (continued).**

Test No.	Oxidized (975 C for 3 days)	Environment	Temperature (C)	Test Duration (days)	Deposits Analyzed by XRD or EDS	Comments
4b	Yes	air	975	2 hours	Deposits consisted of chromium borate. Scale contained mainly chromium borate and some chromium oxide.	The deposits were not removed by exposure at 975 C. However, the deposits could be fractured and removed at room temperature. The coupon did form a scale after this exposure.
5	Yes	Boric Acid	1100	3	NA	Deposits similar to that in test 4a; however, these deposits could not be easily removed. The coupon was placed in water for approximately 12 hours and the deposits were removed. The scale beneath the deposits was removed to base metal in several spots.

\* - Alumina crucible failed.

\*\* - Portion of DWPF Pour Spout (unheated section above bellows) contained heavy irregular scale.

Exposed to DWPF pour spout environment for approximately 1 year (chloride and sulfate salts were present).

**Table 3. Results of Inconel 690 / boron nitride chemical corrosion rate tests.**

Test No.	Oxidized (975 C for 3 days)	Environment	Temperature (C)	Test Duration (days)	Deposits Analyzed by XRD or EDS	Comments
1	Yes	Boron Nitride	1100	6	Metallography - Depth of attack 6 mils.	Measured corrosion rate of 93 mils per year.
2	Yes	Boron Nitride	1100	6	NA	Measured corrosion rate of 51 mils per year.
3	No	Boron Nitride	1100	6	(EDS) Outer layer contained Cr, Ni, Al, Ca, and O. Inner layer contained Cr and O in the matrix and Ni, Cr, and Fe in particles.	Measured corrosion rate of 54 mils per year.
4	No	Boron Nitride	1100	6	Inner deposit contained chromium oxide. Outer deposit was not tested but was probably chromium borate like identified in tests 3a and 3b.	Most of the outer black deposit spalled off easily at room temperature. Remaining outer deposit and inner scale were difficult to remove completely. Measured corrosion rate 59 mils per year.



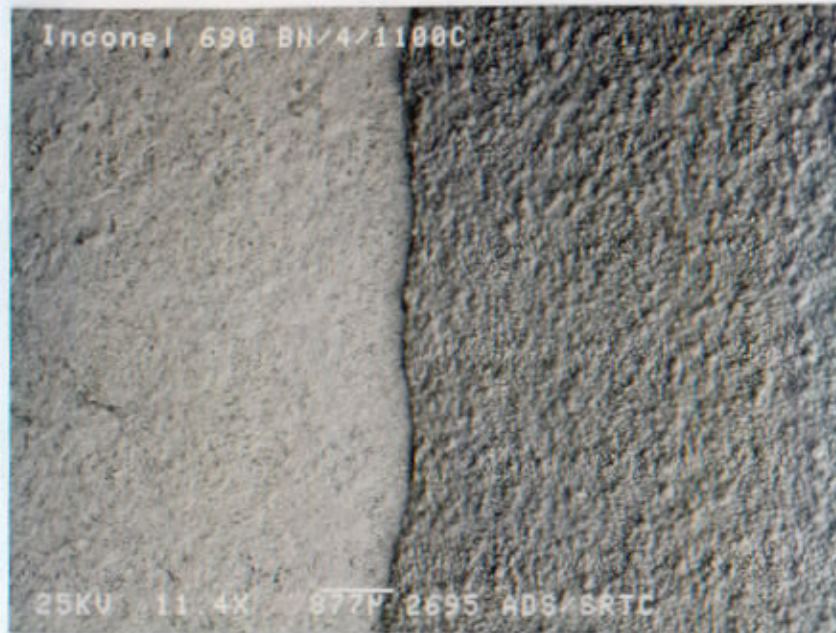


Figure 1. SEM image showing thick chromium borate outer layer (darker region) and the underlying chromium oxide layer (lighter region) that formed on Inconel 690 coupon exposed to boron nitride for 3 days at 1100 °C (690 coupon was not in direct contact with boron nitride coupon).



Figure 2. SEM image of chromium borate layer (same coupon as in Figure 1, boron nitride exposure). The surface was rough and various geometric shapes are shown protruding from matrix.



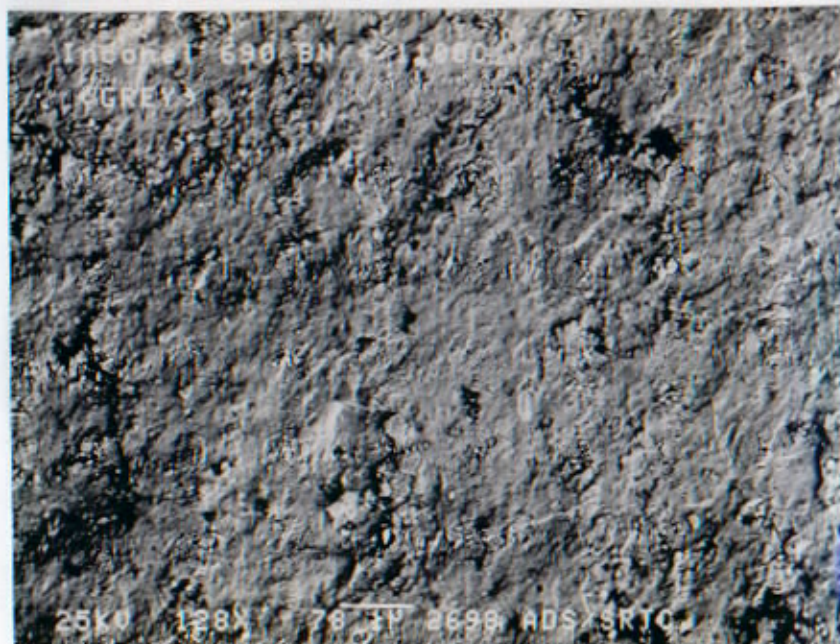


Figure 3. SEM image of the chromium oxide layer (same coupon as in Figure 1, Boron Nitride exposure).



Figure 4. Microstructure of an as-polished Inconel 690 coupon showing internal void formation and some evidence of IGA (same coupon as in Figure 1, magnification 250 X).

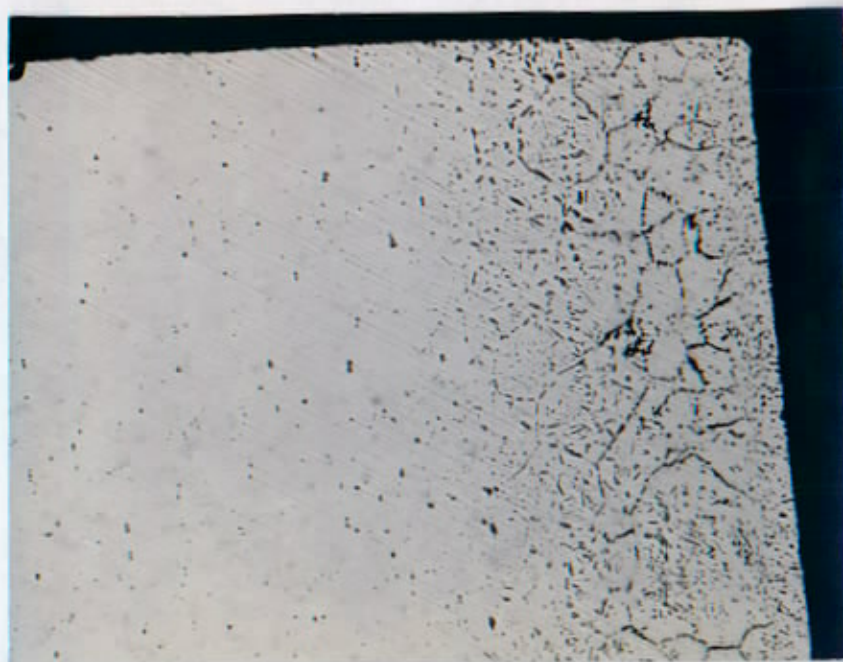


Figure 5. Microstructure of an as-polished Inconel 690 coupon exposed to boric acid for 3 days at 1100 °C (magnification 50 X). Significant internal void formation and IGA is evident to a depth of 30 mils.



Figure 6. SEM image showing internal void formation in Inconel 690 coupon exposed in direct contact to boron nitride for 6 days. Average depth of penetration was 6 mils.



WSRC-TR-96-0394

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