THE DIFFUSION-BONDING OF CORROSION-RESISTANT SHEET CLADDINGS TO IN-738

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ABSTRACT

Diffusion-bonded sheet claddings were investigated as an alternate means of protecting IN-738, a high-strength Ni-base gas turbine alloy, from oxidation and hot-corrosion attack. Sheet alloys Inconel 671 (Ni-50% Cr) and GE 2541 (Fe-25% Cr-4% Al-1% Y) were bonded to IN-738 by hot-isostatic-pressing, using braze-HIP and glass-HIP processing techniques. Claddings of 0.25 mm thickness were applied using autoclave parameters in the range 1093 to 1190C, 10.5 kg/mm², 1 to 2 hours, in order to independently study the corrosion behavior of the cladding and clad/substrate interdiffusion at the bondline.

Clad corrosion discs were evaluated in simulated gas turbine environments using small combustion burner rigs. Exposures of up to 5,000 hours were obtained in sea salt-doped diesel oil containing 1% S at 871 and 982C, and up to 10,000 hours in undoped propane at 982 and 1038C. It was concluded that both Inconel 671 and GE 2541 provide significant hot-corrosion resistance at 871C, and that GE 2541 is clearly superior to Inconel 671, especially at the higher temperatures. Although interdiffusion rates are relatively low at 871C, the diffusion of Cr from Inconel 671 and Fe from GE 2541 into IN-738 promote the precipitation of acicular sigma phase in the substrate near the bondline. Interdiffusion increases markedly at 982 and 1038C, especially that of Fe and Ni in the case of GE 2541.

INTRODUCTION

Diffusion-bonded claddings were investigated as a potential means of protecting critical gas turbine components from severe exidation and hot-corrosion environments encountered in marine applications involving residual oil combustion. The purpose of this work, sponsored in part by the Maritime Administration-Department of Commerce⁽¹⁾, was to (i) characterize the inherent corrosion resistance of a clad superalloy, and (ii) determine the metallurgical behavior of the clad/substrate bondline, including bondline interdiffusion rates, thermal cycling stability of the bondline, and the formation of intermetallic phases at or near the bondline. Ni-base superalloy IN-738, a typical high-strength first-stage bucket alloy, was chosen for the substrate. A 0.25 mm cladding thickness was selected in order to initially isolate surface corrosion effects from bondline behavior.

Previous investigations of clad superalloys by Gedwill $^{(2)}$ and Grisaffe and Gedwill $^{(3)}$ demonstrated that oxidation-resistant cladding alloys such as Fe-25Cr-4Al-1Y and Ni-20Cr-4Al-1Si afforded excellent protection to WI-52 and IN-100 at 1038 and 1093C in normal oxidizing environments for exposures up to 400 hours. The primary objective of this present study, however, was to provide longer-term hot-corrosion resistance between 87lC and 982C, the temperature regime wherein hot corrosion is particularly severe due to condensed corrodents such as Na₂SO₄. A secondary objective was to identify potential hot-isostatic-pressing (HTP) diffusion-bonding techniques which would be applicable to cladding actual airfoil configurations.

EXPERIMENTAL PROCEDURE

Alloy Selection and Specimen Preparation

Extensive burner rig test experience with various Fe, Ni and Co-base sheet alloys narrowed the choice of cladding alloys to Inconel 671 and GE 2541. Inconel 671, a Ni-base alloy containing essentially 50 wt. % Cr, was obtained as 0.3175 cm thick hot-rolled plate from Huntington Division of International Nickel Company. This material was further processed to produce 0.25 mm sheet. A final anneal was conducted at 1260°C for 30 minutes in a very dry $\rm H_2$ atmosphere (dew point -73C).

GE 2541 is an Fe-base alloy containing 25% Cr, 4% Al and 1% Y (wt. %), available only in development quantities. Ingots of the alloy were vacuum induction melted, and hot- and cold-rolled to 0.25 mm. Prior to the fabrication of specimens, the sheet was vacuum annealed (5 x $1\bar{0}^5$ Torr) at 1175C for 30 minutes.

Investment cast bars of IN-738 were cut and ground to 0.16 cm thick x 2.54 cm diameter corrosion discs with a 32 rms surface finish, and all edges were chamfered 45° to prevent tearing of the cladding during HIP deformation and bonding.

Initially, a "braze-HIP" technique was utilized to clad corrosion discs. (4) Individual clad/disc/clad layered specimens were vacuum brazed at 1190C for 30 minutes with Nicrobraze 30 filler alloy placed around the entire disc periphery to establish a leak tight seal between the cladding and substrate, as required for HIP bonding. Two sets of discs were fabricated, one side of each with an Inconel 671 and GE 2541 cladding, and these were HIP-diffusion bonded at 1093C, 10.5 kg/mm², 2 hrs. and 1190C, 10.5 kg/mm², 2 hrs.

Premature failure of braze-HIP clad discs due to preferential attack of the exposed disc edges led to the development of fully clad discs prepared by a "glass-HIP" technique. Prior to assembly, the corrosion discs were glass-bead blasted, and the cladding pieces were polished with 480 grit Al₂O₃ paper; all components were vapor degreased in trichlorethylene.

Small packages of three to six clad corrosion discs were fabricated by spot-welding the discs on a clad sheet, and securely spot-welding the clad-to-clad seams to prevent glass penetration during the HIP cycle.

The cladding packages were loaded into mild steel containers filled with soda-lime glass cull, and mild steel covers with an evacuation tube were GTA-welded in place. The containers were leak-checked on a He mass spectrometer and outgassed at 316C for 16 hours at a vacuum of $1\bar{0}^3$ mm, Hg followed by forge-welding of the evacuation tube.

Diffusion bonding was accomplished in an argon-pressurized autoclave by hot-loading the containers at 982C, closing the vessel, and simultaneously increasing temperature and pressure to the preset levels of 1149C, 10.5 kg/mm². Following a l hour hold, the autoclave was depressurized and furnace power shut-off, permitting removal of the containers at approximately 870C. The containers were directly water-quenched to fracture the glass, allowing easy removal of the cladding packages and subsequent final cleanup of the specimens by glass-bead blasting. The individual clad discs were burner rig tested with small clad-to-clad fins on both the disc periphery and the support-pin center hole to preclude premature failure due to exposed clad/ substrate edges.

Testing and Evaluation

The clad IN-738 corrosion discs were evaluated in simulated gas turbine environments using small combustion burner rigs. (5) Tests were conducted in a normal oxidizing environment at 982 and 1038C in combusted undoped propane, and in a hot-corrosion environment at 871 and 982C, produced by burning diesel oil containing 1% S and doped with artificial sea salt mixed with the fuel to yield approximately 8 ppm Na in the combustion products. The burner rigs operate at atmospheric pressure, 20 mps gas velocity, and a maximum air: fuel ratio of 50:1 at 871C. The specimens, which are stationary during test, are cycled to room temperature by air blasting approximately every 50 hours.

Following test, the specimens were examined metallographically at 100 times magnification to determine (i) maximum depth of attack, (ii) average surface loss of the cladding, and (iii) depth of internal oxide, nitride or sulfide formation in the substrate below the bondline. Data was obtained on the basis of the original disc and cladding dimensions, using the clad/substrate bondline as the reference point. Selected specimens were examined on a Cameca electron microprobe analyzer to determine the extent of clad/substrate interdiffusion, cladding composition gradients, and the composition of precipitate phases.

RESULTS

Inconel 671 Cladding

As-Bonded Condition – Inconel 671 is a two-phase alloy containing a dispersion of α -Cr particles, with the approximate composition 85 wt. % Cr, 15 wt. % Ni, in a γ -Ni matrix containing about 35 wt. % Cr. In the as-bonded condition, the braze-HIP specimens processed at 1093C, 10.5 kg/mm², 2 hr. exhibited an α -Cr denuded region approximately 0.02 mm deep at the surface due to oxidation during the autoclave cycle (Figure 1a). The bondline between the cladding and substrate is clearly delineated by a discontinuous string of small oxide particles, which according to probe analysis are oxides of Al and Zr. Beneath the bondline, a 0.01mm wide γ '-denuded zone developed in the IN-738 due to Al diffusion into the cladding. The specimens diffusion-bonded at 1190C, 10.5 kg/mm², 2 hr. experienced significant coarsening of the α -Cr precipitate in Inconel 671, an increase in the surface α -Cr denuded zone to 0.035 mm, and an increase in the IN-738 γ ' denuded zone to 0.02 mm (Figure 1b).

Although the glass-HIP specimens were bonded at 1149C, a reduction in hold time to 1 hour and an overall reduction in cycle time produced little coarsening of the $\alpha\text{-Cr}$, and an IN-738 $\gamma^{\text{I}}\text{-denuded}$ zone of less than 0.005 mm. Furthermore, there was no indication $\alpha\text{-Cr}$ surface depletion or reaction with the soda-lime glass (Figure 1c).

Burner Rig Tests - Metallographic measurements of Inconel 671 clad IN-738 specimens following oxidation and hot-corrosion burner rig testing are listed in Table I. Hot corrosion testing in doped diesel oil at 871C confirmed Inconel 671's excellent resistance in this environment, with about one-third the cladding thickness penetrated in 3000 hours (Figure 2a). There was little difference in performance for the braze-HIP versus glass-HIP specimens, except for the braze-HIP specimens bonded at 1190C. The wider initial α -Cr denuded zone at the cladding surface, indicative of Cr depletion, resulted in a higher rate of attack.

The protective oxide mechanism for Inconel 671 involves the formation of a continuous, adherent Cr_2O_3 film, generated by the diffusion of Cr out of the cladding. The hot corrosion of Inconel 671 proceeds by the consumption of Cr in the alloy via the formation of the surface Cr2O3 layer concurrent with the in-situ formation of "CrS" particles in the $\alpha\text{-Cr}$ denuded zone. Corrosion proceeds generally in "plane-front" fashion, with no indication of preferential grain boundary attack.

Microprobe analysis of specimen 1B-1, exposed for 2026 hours at 871C determined the width of the surface α -Cr denuded zone to be about 0.1 mm. The Cr concentration dropped from nominally 35% in the alloy to 25% at the surface, and to 16% at the bondline, which is the level present in IN-738. The bondline diffusion zone remained quite narrow (.025 mm) and the diffusion distance of A1 and Co into Inconel 671 from IN-738 was less than 0.02 mm.

Hot corrosion testing at 982C proved far more severe, producing total penetration and failure of the cladding in 1600 to 2000, hours, regardless of processing technique or bonding cycle (Figure 2b). Degradation of the cladding, as in the 871C exposures, was principally related to the consumption of Cr in the surface oxide and by internal oxidation and sulfidation. At failure, the IN-738 substrate exhibited large quantities of Cr- and Ti-rich nitrides and Al $_2$ 0 $_3$. However, there was no indication of Ni $_3$ S4:Ni eutectic formation, which is generally associated with the catastrophic hot-corrosion of Ni-base alloys. Interdiffusion at 982C produced a γ '-denuded zone almost 0.2 mm deep after 1500 hours.

Normal oxidizing exposures in undoped propane at 982 and 1038C produced similar results and structures, due to the inability of Inconel 671 to form an adherent and protective oxide at these temperatures. Internal oxidation of the cladding in approximately 4000 hours at 982C (Figure 2c) and 2000 hours at 1038C (Figure 2d) led to severe internal oxidation and nitrification of the substrate to a depth of about 0.25 mm in these exposures. In general, the depth of these zones corresponds to the γ^{\prime} -denuded region. Due to the severity of attack, it was not possible to obtain meaningful data for the cladding and substrate.

GE 2541 Cladding

As-Bonded Condition - GE 2541 is a ferritic Fe-base alloy containing a dispersion of Y-Fe intermetallic particles. $^{(6)}$ In the as-bonded condition, Fe diffuses into the substrate and increases the solubility for A1, resulting in a denuded zone devoid of the fine γ' particles. However, microprobe analysis showed the larger particles in this zone to be essentially γ' (Ni₃A1) composition, hence, the larger particles appear to grow at the expense of the fine particles during the bonding cycle. This zone measured 0.02 mm for the 1093C bonded braze-HIP specimens (Figure 1d), and 0.015 mm

for the 1149C-bonded glass-HIP specimens (Figure 1f). The braze-HIP specimens bonded at 1190C showed signs of braze alloy along the bondline, producing a porous and mottled appearance in the diffusion zone indicative of incipient melting (Figure 1e). The higher bonding temperature also produced grain growth in the cladding normal to the bondline, and a narrow band (0.013 mm) of austenitic FeCrAIY due to the diffusion of Ni from the substrate.

Burner Rig Tests - The results of the metallographic measurements are listed in Table I. At each test condition and temperature, GE 2541 exhibited corrosion resistance superior to Inconel 671. Approximately one-third of the cladding thickness was penetrated after 5000 hours in 871C hot corrosion (Figure 3a). In the more severe test at 982C, GE 2541 showed signs of failure after 2500 to 3000 hours (Figure 3b). GE 2541 is clearly more oxidation resistant than Inconel 671, following exposures of 10,000 hours at 982C (Figure 3c) and 2000 hours at 1038C (Figure 3d) in undoped propane.

As in the case of Inconel 671, the hot corrosion of GE 2541 proceeds in a plane-front mode without preferential grain boundary attack. GE 2541, however, develops a continuous, adherent surface oxide of Al_20_3 , which is apparently more impervious to the passage of sulfur and oxygen. The rate of formation of internal sulfides and oxides was significantly lower in all of the exposures than in the case of Inconel 671.

Interdiffusion between GE 2541 and IN-738, however, occurred at a higher rate. Microprobe analysis of specimen 1B-1 exposed for 2026 hours at 871C measured Fe diffusion into IN-738 to a depth of 0.065 mm, which is essentially coincident with the γ' -denuded zone. Ni and Co diffused from IN-738 completely through the cladding to uniform levels of 5% and 1%, respectively. In addition, a 0.015 to 0.02 mm wide zone formed adjacent to the bondline with the approximate composition Fe-20Cr-10Ni (in wt. %), which is sufficient to transform the matrix to the γ -austenite phase. For specimens bonded at 1190C, which exhibited the austenitic zone initially, 871C exposures ad two effects: (i) the austenitic FeCrAlY zone grew to approximately 0.04 mm width after 2800 hours, and (ii) exposures as short as 1500 hours induced the formation of accicular sigma phase to a depth of 0.15 mm in IN-738.

Interdiffusion increased significantly in the higher temperature exposures, with Fe diffusion to a depth of 0.02 mm in IN-738 after 9452 hours at 982C, and to 0.15 mm in 2113 hours at 1038C. GE 2541 appears to have been completely transformed to austenite via outward Ni diffusion in these exposures.

DISCUSSION

This study has demonstrated that diffusion-bonded claddings are a viable means of protecting IN-738, a typical gas turbine bucket alloy, from severe oxidation and hot-corrosion attack. Both cladding alloys, Inconel 671 and GE 2541, exhibit corrosion resistance far superior to conventional aluminide coatings in an environment containing 8 ppm Na at 871C. GE 2541 is clearly more oxidation and corrosion resistant than Inconel 671, particularly at the higher test temperatures of 982 and 1038C.

The difference in the corrosion behavior of the two cladding alloys is clearly related to the type of oxide formed. Inconel 671 relies on the formation of a surface layer of Cr_2O_3 for its protection. At 871C, the oxide is stable and adherent, and the consumption of Cr in the alloy is relatively slow. At higher temperatures, however, Cr_2O_3 is less stable and adherent. Loss of Cr_2O_3 via the volatilization of Cr_3 may contribute to cladding degradation. (7) The inward diffusion of sulfur and oxygen, resulting in in situ Cr_2O_3 and Cr_3 formation, further reduces the residual Cr content of the cladding.

Despite the high Cr content in Inconel 671, interdiffusion with IN-738 is relatively slow during the bonding cycle and subsequent exposure at 871C. Counterdiffusion of substrate elements into the cladding is also slow, hence, the bondline region is relatively stable. However, the diffusion of Cr into IN-738 can be expected to eventually promote the precipitation of sigma phase in the temperature range of maximum sensitivity, 816 to 927C. The time at temperature during the initial bonding cycle may be important in establishing concentration gradients which delay the onset of this reaction by minimizing the initial diffusion zone. Surface reactions during the bonding cycle should also be avoided to minimize the depletion of Cr.

GE 2541, on the other hand, forms a stable, adherent Al₂03 film, which remains protective over the entire range of conditions investigated. In-situ oxidation and sulfidation of the cladding is much slower than in the case of Inconel 671, hence, Al is consumed at a low rate. Conversely, interdiffusion of Fe and Ni between cladding and substrate is rapid, even in 871C exposures. The presence of Fe in IN-738 can also lead to sigma phase formation, hence, it is important that the bonding cycle be minimized to delay this effect. Despite this interaction, GE 2541 is capable of maintaining its protective capability for up to 10,000 hours at 982C in normal oxidizing atmospheres. It is not clear whether interdiffusion has any affect on cladding oxidation or hot-corrosion resistance in these exposures.

Both the braze-HIP and glass-HIP processes were found suitable for fabricating clad test specimens, however, the use of molten glass as the pressure transmitting media appears more reliable than obtaining a leak-tight braze. This process has subsequently been scaled-up and proven reliable for diffusion-bonding these cladding alloys to first-stage gas turbine bucket airfoils, which are currently on test in a turbine burning treated residual oil.

SUMMARY AND CONCLUSIONS

This study demonstrated the feasibility of diffusion-bonding oxidation and hot-corrosion resistant sheet cladding alloys Inconel 671 and GE 2541 to IN-738, a γ^{\prime} -strengthened Ni-base superalloy. Braze-HIP and glass-HIP processing techniques were successfully developed and utilized in the fabrication of clad corrosion discs, which were burner rig tested in doped diesel oil at 871 and 982C and undoped propane at 982 and 1038C. HIP bonding parameters in the range 1093 to 1190C, 10.5 kg/mm² pressure, for 1 to 2 hours produce excellent metallurgical bonds, but the extent of interdiffusion with the substrate can influence phase reactions such as sigma precipitation during subsequent exposure.

Both Ni-base alloy Inconel 671 and Fe-base alloy GE 2541 impart excellent hot-corrosion resistance to IN-738 at 871C. However, Inconel 671 fails to form an adherent and protective oxide at 982C. Despite the alloy's high Cr-content, Inconel 671 is relatively compatible with the substrate, and interdiffusion is slow at 871C.

GE 2541, an ${\rm Al}_20_3$ -forming alloy, is superior to Inconel 671 under all test conditions, and provides useful protection to IN-738 up to 1038C. However, interdiffusion with the substrate, especially Fe and Ni, is significant even at 871C.

ACKNOWLEDGEMENTS

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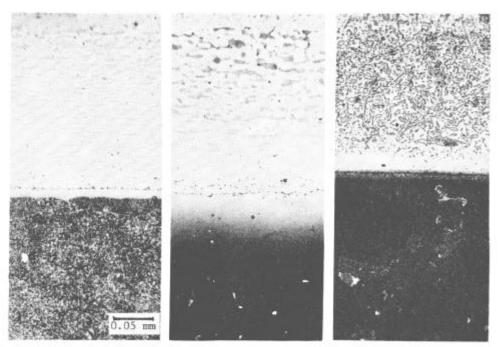
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Table I Clad IN-738 Burner Rig Results

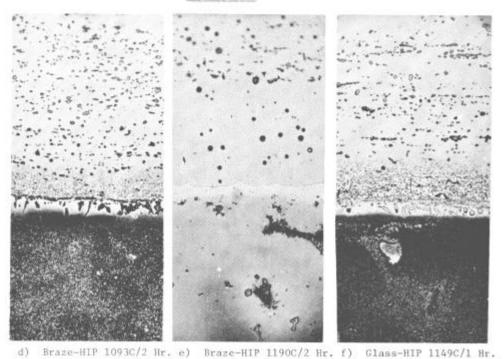
			Temp.	Time,	Max. Penetrațion	Loss	Internal Oxid.
Cladding	Specimen*	<u>Fuel</u>	°C_	Hr.	mmx10 ²	mmx10 ²	mmx10 ²
Incone1	1G-2	D.O.+S.S.#	871	1000	5.0	3.5	-
671	2B-1			1573	7.8	4.3	_
	1G-4			2025	6.9	3.8	-
	1B-1			2026	3.3	3.3	_
	2B-3			2793	14.7	3.3	_
	1G-6			2999	7.6	3.5	-
	1B-4	D.O.+S.S.	982	604	10.7	3.8	_
	1G-17			1002	9.4	4.6	-
	1G-18			1670	25.4	25.4	16.0
	2B-2			2011	25.4	12.4	22.6
	1B-5	Propane	982	4006	13.7	4.8	24.4
	2B-6			9452	D	estroyed	
	2B-5	Propane	1038	2113	25.4	18.0	25.7
GE 2541	3G-2	D.O.+S.S.	871	1020	6.6	0.7	8.009
	2B-1			1573	2.8	2.8	_
	3G-4			2019	6.3	1.8	-
	1B-1			2026	0.7	0.7	_
	2B - 3			2793	1.8	1.8	_
	3G-8			3993	8.9	1.0	_
	3G-10			5000	8.9	1.3	_
	1B-4	D.O.+S.S.	982	604	0.7	0.7	-
	2B-2			2011	7.4	0.7	-
	2B-4			2352	16.5	2.0	19.1
	1B-5	Propane	982	4006	0.7	0.7	_
	2B-6			9452	5.1	1.3	_
	2B-5	Propane	1038	2113	14.2	0.7	13.5

^{*1}B - Braze HIP 1093C, 10.5 kg/mm², 2 hr. 2B - Braze HIP 1190C, 10.5 kg/mm², 2 hr. 1,3G - Glass HIP 1149C, 10.5 kg/mm², 1 hr.

#Dieser Oil + Sea Salt.

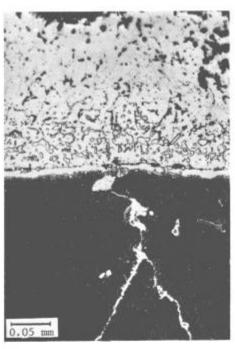


a) Braze-HIP 1093C/2 Hr. b) Braze-HIP 1190C/2 Hr. c) Glass-HIP 1149C/1 Hr.
Inconel 671

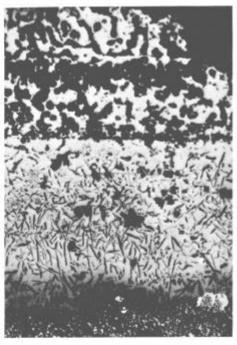


GE-2541

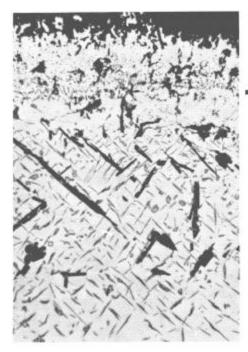
Figure 1 - As-Bonded Microstructures of Clad IN-738.



a) Specimen 1G-6 D.O.+S.S. 871C/2999 Hr.



b) Specimen 1C-18 D.O.+S.S. 982C/1670 Hr.



c) Specimen 18-5 Propane 982C/4006 Hr.



d) Specimen 2B-5 Propane 1038C/2113 Hr.

Figure 2 - Burner Rig Exposures of Inconel 671 Clad IN-738.

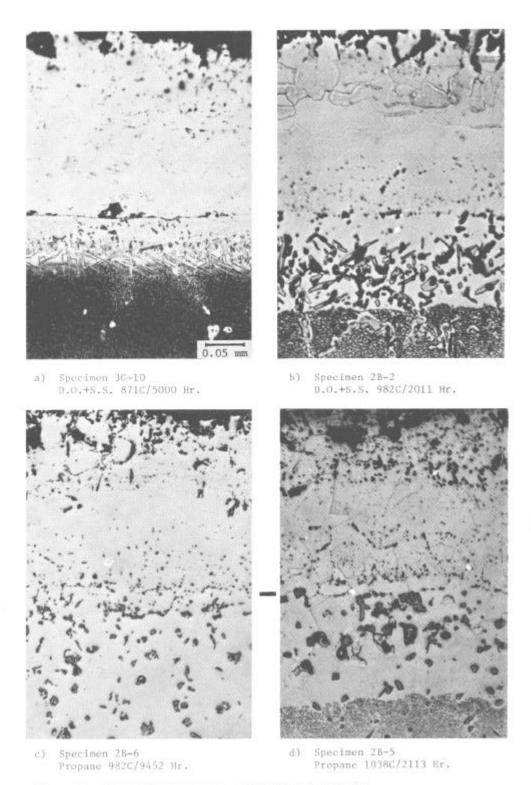


Figure 3 - Burner Rig Exposures of GE 2541 Clad IN-738.