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**WELDABILITY AND JOINING TECHNIQUES
FOR
ADVANCED FOSSIL ENERGY SYSTEM ALLOYS**

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Report Prepared by

C. D. Lundin, C. Y. P. Qiao, W. Liu, D. Yang, G. Zhou and M. Morrison

Materials Joining Research
Materials Science and Engineering Departments
The University of Tennessee, Knoxville, TN 37996-2200
under
ORNL/Sub/88-07685, WBS Element UT-2

for

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
Managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.
for the
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WELDABILITY AND JOINING TECHNIQUES FOR ADVANCED FOSSIL ENERGY SYSTEM ALLOYS

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Research sponsored by the U. S. Department of Energy,
Fossil Energy
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FORWARD

This report covering the effort over the period from April 1995 to March 1998 is presented in parts according to the Tasks defined concerning the Weldability, Performance of Advanced Austenitic Alloys and Iron Aluminides. Each Task is divided into sub-sections where appropriate and the conclusions reached are presented within each sub-section.

The Tasks undertaken and reported on herein are:

TASK 1. Weldability and Metallographic Evaluation of 310HCbN Type Stainless Steel.

This section of the report covers weldability testing of 310HCbN using two weldability test methods, the metallurgical evaluation of the HAZ as a function of peak temperature and additional hot cracking evaluations utilizing a specially developed Tube Weld Cracking Test. These studies amplify the work done in the previous work period from August 1991 to June 1994 and brings to a culmination the information available on 310HCbN as compared to traditional austenitic stainless steel alloys.

TASK 2. Weldability and Metallographic Evaluation of a Newly Developed 310 Tantalum Bearing Stainless Steel: 310TaNb

This section of the report covers weldability evaluations and the basic metallurgy of the 310TaNb stainless as to the strengthening mechanisms , aging and the potential for property degradation in the weld HAZ.

TASK 3. Iron Aluminide Cladding on Stainless Steel Tubing

This section amplifies weld cladding experiments with Iron Aluminide on stainless steel tubing materials for high temperature erosion and corrosion protection.

EXECUTIVE SUMMARY

The research reported on herein was undertaken in support of the DOE Fossil Energy Materials development of new alloys for advanced power generation systems. The efforts represent the concerns for the basic understanding of the weldability and fabricability of the advanced high temperature alloys so necessary to affect increases in the efficiency of the next generation Fossil Energy Power Plants. The effort was divided into three tasks with the first effort dealing with the welding and fabrication behavior of 310HCbN (HR3C), The second task details the studies aimed at understanding the weldability of a newly developed 310TaN high temperature stainless (a modification of 310 stainless) and Task 3 addressed the cladding of austenitic tubing with Iron-Aluminide using the GTAW process.

Task 1 consisted of microstructural studies on 310HCbN and the development of a Tube Weldability test which has applications to production welding techniques as well as laboratory weldability assessments. In addition, the evaluation of ex-service 310HCbN which showed fireside erosion and cracking at the attachment weld locations was conducted. This section is divided into 4 parts addressing each of the above evaluations.

Task 2 addressed the behavior of the newly developed 310TaN modification of standard 310 stainless steel and showed that the weldability was excellent and that the sensitization potential was minimal for normal welding and fabrication conditions. The microstructural evolution during elevated temperature testing was characterized and the second phase particles evolved upon aging were identified.

Task 3 details the investigation undertaken to clad 310HCbN tubing with Iron Aluminide and developed welding conditions necessary to provide a crack

free cladding. The work showed that both a preheat and a post-heat was necessary for crack free deposits and the effect of a third element on the cracking potential was defined together with the effect of the aluminum level for optimum weldability.

TASK 1

Weldability and Metallographic Evaluation of 310HCbN Type Stainless Steel.

MICROSTRUCTURAL CHARACTERIZATION AND WELDABILITY EVALUATION OF THE WELD HEAT AFFECTED ZONE (HAZ) IN 310HCbN TUBING

ABSTRACT

Metallographic evaluation on Gleeble simulated HAZ samples of 310HCbN tubing material was performed in order to reveal potential degradation in mechanical properties and corrosion resistance. The carbide evolutionary process in the HAZ samples was studied. It is indicated that 310HCbN material showed a weld HAZ sensitization tendency that is associated with the formation of Cr_{23}C_6 .

INTRODUCTION

310HCbN tubing has been accepted for boiler applications under Code Case 2115. It has been employed by utilities in the United States for replacement of conventional stainless steels. Excellent high temperature mechanical properties, such as creep and high temperature tensile strength, have been reported [1]. However, need for the detailed information on aged base metal and weld HAZ microstructures and their relationship to material properties is apparent. In addition, weldability and welded fabrication associated property variations should be studied for optimum use of the material.

Weldability and microstructural evaluations of 310HCbN were conducted at Oak Ridge National Laboratory and the University of Tennessee [2-4]. The weldability assessment was conducted using the Varestraint hot cracking test and a newly developed tubing weld cracking (TWC) test. It is concluded that 310HCbN possesses an excellent hot cracking resistance. In addition, the TWC

test is proven to be a viable tubing material weld hot cracking evaluation method.

The weld HAZ microstructural evaluation was conducted using Gleeble simulated HAZ samples and electric resistance spot welded samples. The metallographic evaluation was performed using OLM, SEM and STEM. The microstructural changes and secondary phase evolution processes as a function of the weld HAZ peak temperature were investigated. The results from this study were used to explain the HAZ hot cracking mechanism as well as the weld HAZ stress corrosion cracking sensitivity (ASTM 262 Practice A).

EXPERIMENTAL PROCEDURES

The material used in this study is a commercial heat of 310HCbN tubing. The composition of the 310HCbN heat is presented in Table 1. The 310HCbN tubing was in the annealed condition.

Table 1. Composition of the 310HCbN tubing.

C	Mn	P	S	Si	Ni	Cr	Mo	V	Nb
0.061	1.19	0.010	<0.001	0.36	19.92	24.06	0.07	0.04	0.45
Ti	Co	Cu	Al	B	W	Fe	N	O	Ta
<0.01	0.27	0.09	0.023	0.002	<0.01	53.3	0.26	0.005	<0.01

* The chemical composition was analyzed at ABB Combustion Engineering.

The microstructural evaluation was carried out on the base metal and Gleeble simulated HAZ samples. For the Gleeble simulated HAZ samples, seven different peak temperatures (900°C, 1000°C, 1100°C, 1200°C, 1250°C, 1300°C and 1320°C) were selected. Optical light, scanning electron and transmission electron microscopy was employed for the metallographic

examinations. The ASTM 262 Practice A was conducted on the Gleeble simulated HAZ samples. Limited precipitate particle characterization was performed using EDS and X-ray diffraction techniques.

RESULTS AND DISCUSSION

The previous investigations [3] at the University of Tennessee revealed that 310HCbN tubing possesses an excellent weld hot cracking resistance (using both Varestraint and TWC tests). Thus, the emphasis in this tubing evaluation of 310HCbN was concentrated on the HAZ microstructural evolution and aging. One of the major microstructural concerns is the carbide evolution behavior and its relationship to the mechanical properties and metallurgical and corrosion behavior.

Optical Microstructural Evaluation

Figure 1 shows the optical microstructural morphology of base metal and the simulated HAZ samples (etched with aqua regia). Clearly, the carbides and nitrides are uniformly distributed in the base material matrix in the annealed condition. In the samples with the simulated peak temperatures of 900°C and 1000°C, a greater amount of precipitate was observed compared to the base metal, especially for the small size particles. The precipitate is slightly reduced in the sample with a simulated peak temperature of 1100°C compared to base metal. This may reflect the initiation of precipitate dissolution for certain precipitate particles (e.g. $M_{23}C_6$). In addition, fine precipitates were observed and it is believed that MC type carbides start to form under this HAZ thermal expose. When the simulated peak temperature is in excess of to 1200°C, the fine precipitates increase and it is expected that the newly formed precipitates are dominantly NbC and/or NbN carbides. A similar microstructural morphology was found in sample with the simulated peak temperatures of 1250°C and

1300°C. Additionally, evidence of a reaction between carbides and matrix was observable although it appears that the reaction is in the initial stages. In the simulated HAZ sample with a peak temperature of 1320°C (ZDT), significant evidence of a liquation reaction is apparent. It is noticed that the liquated areas are distributed both intergranularly and intragranularly.

HAZ Sensitization Evaluation

Figure 2 presents the microstructural morphologies of the Gleeble simulated HAZ samples after electrolytic etching with oxalic solution (ASTM A262 Practice A). It is evident that the base metal is not sensitized. It also shows that the precipitates are uniformly distributed in the base material. In the simulated HAZ samples with the peak temperatures of 900°C and 1000°C, a ditched type grain boundary morphology is present with grains completely surrounded. This is an indication of sensitization. This temperature range (900°C-1000°C) reflects the lower boundary of the HAZ. Therefore, *intergranular type localized corrosion may take place in the as welded HAZ if aqueous conditions prevail.* The cause of the sensitization is the formation of Cr₂₃C₆ type carbides along the grain boundaries and a Cr depleted zone adjacent to the grain boundaries.

It should be noticed that for the HAZ sample with the peak temperature of 1100°C, the grain boundaries were not ditched. Further, the amount of precipitates shown in the sample with the peak temperature 1100°C is significantly reduced compared to that in the simulated HAZ with peak temperatures of 900°C and 1000°C. This observation indicates that dissolution of Cr₂₃C₆ occurred for the 1100°C thermal history. Evidence of grain boundary ditching begins to be seen in the sample with a peak temperature of 1200°C and more evidence of grain boundaries is found in the sample with a peak temperature of 1250°C. However, this is step structure in the simulated HAZ sample with a peak temperature of 1250°C. A dual structure is evident in the

simulated HAZ sample with a peak temperature of 1300°C and this becomes aggravated when the peak temperature is increased to 1320°C (ZDT). The explanation of the higher temperature range sensitization is due to the dissolution of MC and/or MN type precipitates and followed by relatively fast cooling. In this case, reformation of MC and/or MN type precipitates upon cooling is limited and during cooling in the lower temperature portion of welding thermal cycle, Cr₂₃C₆ is formed. This discussion indicates that "knife line" type attack may occur in the weld HAZ of as-welded 310HCbN.

TEM Microstructural Evaluation

In order to confirm the above observations and postulation, carbides and/or nitrides particles were extracted from the base material and EDS checked. Figure 3 shows the SEM morphology of particles extracted from the 310HCbN base material. The dominant particles are within the size range of 1μm to 10μm. The EDS examination results revealed that most of the particles are Nb rich carbides or nitrides. A small amount of Ti and Cr may be contained in the NbC and NbN. A typical EDS spectrum for particle "a" in Figure 3 is exhibited in Figure 4.

Figure 5 shows the typical TEM microstructural morphologies of the base metal in which intragranular carbides are predominantly present. The dislocation density in the matrix of base material is low.

Typical TEM microstructural morphologies in the simulated HAZ samples with the peak temperatures of 900°C and 1000°C are presented in Figures 6 and 7, respectively. Evidence of an increased amount of precipitates and the appearance of intergranular precipitates are clearly indicated. The increase in carbides is due to the formation of Cr₂₃C₆ (preferentially along the grain boundaries). A typical EDS spectrum for these carbides is exhibited in Figure 8.

Typical TEM microstructural morphology of the simulated HAZ samples with a peak temperature of 1100°C is shown in Figure 9. Amount of precipitates

(both intergranular and intragranular) in this sample is apparently reduced. Fine particles that are rich in Cr and Nb were present. A typical EDS spectrum of these particles is exhibited in Figure 10.

When the peak temperature increases to 1200°C, Nb rich type carbides become predominant and the size of Nb rich carbides increased compared to those observed in Figure 9 (1100°C) (see Figure 11). Figure 12 shows a typical TEM microstructural morphology in a sample with a peak temperature of 1250°C. More fine, rectangular, bar shaped carbides were formed at this temperature. A typical TEM microstructural morphology of the simulated HAZ sample with a peak temperature of 1300°C is shown in Figure 13. Clearly, the intergranular precipitates are dominant. A typical EDS spectrum of the particles in Figure 12 is shown in Figure 14. It is noticed that Nb content in carbides is increased in the simulated HAZ sample with a peak temperature of 1300°C compared to that in the simulated HAZ sample with a peak temperature of 1100°C. A typical TEM microstructural morphology of the simulated HAZ sample with a peak temperature of 1320°C is shown in Figure 15. The quantity of precipitate is further increased in this sample both intergranularly and intragranularly compared to that in the simulated HAZ sample with a peak temperature of 1300°C.

The HAZ hardness as a function of HAZ peak temperature in 310HCbN material is presented in Figure 16. In general, the variation of hardness in the HAZ is relatively small. Compared to base metal (190 HV), the entire HAZ region showed a lower hardness. As indicated in Figure 16, the maximum hardness was reached with a peak temperature of 1100°C, in which the fine Nb rich particles started to form according to the above metallographic evaluation results. It should be noted that hardness in weld HAZ is not only affected by the precipitate distribution but also associated by grain size, thus, the combined information should be considered in order to interpret the HAZ hardness behavior. Nevertheless, the microhardness measurements agree with the metallographic examination results.

SUMMARY

1. Metallographic examination indicated that carbide evaluation in the weld HAZ is the major factor causing the mechanical property and corrosion resistance changes in 310HCbN material.
2. A sensitized HAZ microstructure was found in the simulated HAZ samples of 310HCbN with peak temperatures of 900°C and 1000°C. The formation of $M_{23}C_6$ type carbides along the grain boundaries is directly responsible for this sensitization.
3. A dual type structure was observed in the simulated HAZ sample with peak temperature of 1320°C in A262 Practice A. This may imply that a "knife line" attack tendency may be present for 310HCbN.
4. This investigation also showed that carbides (both Nb rich MC type or Cr rich $M_{23}C_6$) evolution processes can occur in the weld HAZ of 310HCbN. The formation of MC type precipitates in the weld HAZ may enhance the reheat cracking tendency because fine MC type carbides preferentially form intragranularly at the dislocation and/or stack faulting sites.

ACKNOWLEDGMENTS

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Figure 1. Optical microstructural morphology of base metal and the simulated HAZ samples. (a) Base Metal, (b) HAZ (PT: 900°C), (c) HAZ (PT: 1000°C), (d) HAZ (PT: 1100°C), (e) HAZ (PT: 1200°C), (f) HAZ (PT: 1250°C), (g) HAZ (PT: 1300°C) and (h) HAZ (PT: 1320), 400X.

Figure 2. Microstructures in the base metal and the simulated HAZs after A262 Practice A test. (a) Base Metal, (b) HAZ (PT: 900°C), (c) HAZ (PT: 1000°C), (d) HAZ (PT: 1100°C), (e) HAZ (PT: 1200°C), (f) HAZ (PT: 1250°C), (g) HAZ (PT: 1300°C) and (h) HAZ (PT: 1320), 100X.

Figure 3. SEM morphologies of carbides extracted from base metal.

Figure 4. EDS spectrum for particle "a" in Figure 3.

Figure 5. TEM microstructure of base metal.

Figure 6. TEM microstructure in the simulated HAZ with a peak temperature of 900°C.

Figure 7. TEM microstructure in the simulated HAZ with a peak temperature of 900°C.

Figure 8. Typical EDS spectrum for the carbides in Figures 6 and 7.

Figure 9. TEM microstructure in the simulated HAZ with a peak temperature of 1100°C.

Figure 10. Typical EDS spectrum for the particles in Figure 9.

Figure 11. TEM microstructure in the simulated HAZ with a peak temperature of 1200°C.

Figure 12. TEM microstructure in the simulated HAZ with a peak temperature of 1250°C.

Figure 13. TEM microstructure in the simulated HAZ with a peak temperature of 1300°C.

Figure 14. Typical EDS spectrum of the particles in Figure 13.

Figure 15. TEM microstructure in the simulated HAZ with a peak temperature of 1320°C.

Figure 16. HAZ microhardness in the simulated HAZs.

MICROSTRUCTURAL EVALUATION OF THE WELD HAZ IN 310HCbN

ABSTRACT

Metallographic evaluation indicated that the HAZ liquation type in 310HCbN belongs to the non-uniformly distributed grain boundary liquation type which is considered to be relatively innocuous to HAZ liquation cracking. Semi-quantitative chemical analysis of the liquid "pocket" indicated that the liquid "pocket" occurred due to a reaction between Nb and the matrix. The quantitative evaluation, in terms of critical amount of Nb content to form eutectic reaction along the grain boundary in the HAZ of a Nb bearing alloy, is addressed.

INTRODUCTION

The previous hot crack testing using both Varestraint testing and the tubing weld cracking (TWC) test, which recently was developed, and hot ductility testing results indicated that 310HCbN alloy possesses an excellent hot cracking resistance compared to modified 800H [1]. In order to provide a proper explanation of the hot crack testing results, a metallographic examination was performed with an emphasis on the HAZ liquation morphology, extent and distribution.

A liquation influence on the hot cracking behavior in Nb bearing austenitic alloys is controversial. Nevertheless, it was confirmed in a previous study that eutectic related HAZ liquation is generally not egregious to HAZ liquation cracking. In fact, eutectic reaction related HAZ liquation acts as a reservoir which enhances crack healing. Thus a material exhibiting an eutectic reaction type HAZ liquation is, in general, HAZ hot cracking resistant.

MATERIALS AND EXPERIMENTAL PROCEDURES

One heat of 310HCbN (HR3C-C) tubing alloy was employed for this part of the research and the chemical composition is given in Table 1.

Table 1. Composition of the 310HCbN tubing alloy.

UT Code	Cr	Ni	Mn	Si	C	Nb	Mo	To	V	S	P	N	Al	Fe
HR3C-C	24.06	19.92	1.19	.36	.061	.45	.07	.01	.04	.01	.01	.25	.023	Bal

The resistance spot welding process, Gleeble simulation and GTA welding were utilized to generate HAZ metallographic samples. OLM and SEM were employed to examine the microstructural morphology and EDS was used for semi-quantitative chemical analysis. The elemental line scan technique was applied to analyze localized elemental distributions.

RESULTS AND DISCUSSION

Microstructures in the Resistance Spot Weld HAZ

No cracking was observed in either the fusion zone or the HAZ of the resistance spot weld. The eutectic reaction related constitutional liquation is the dominant liquation type along the grain boundaries, the twin boundaries as well as the grain matrix. Figure 1 shows the typical microstructure of the HAZ in the resistance spot welded sample. It is clear that HAZ liquation formed both intergranularly and transgranularly. The liquid "pockets" shown in Figure 1 associated with the NbC dissolution and reaction with grain matrix.

Figure 2 shows the SEM micrograph of the microstructures in the HAZ of resistance spot weld in HR3C-C. It is clear, in general, that the liquid "pocket" located remote from the fusion boundary is smaller than that of liquid "pocket"

adjacent to the fusion boundary due to the fact that the greater the distance from fusion boundary, the lower the peak temperature.

A higher magnification micrograph of location A in Figure 2 is exhibited in Figure 3. Elemental line scans were performed across the liquid "pocket" and the location of each EDS measured spot is indicated in Figure 4. The elemental line scan results for lines ab and cd are indicated in Figures 5 and 6, respectively. Obviously, along line ab, the area where Nb is enriched, Fe and Ni are significantly lower but Cr is not. This is true for line cd as well. These data indicate that average Nb content in the liquid "pocket" along line cd is about 4 to 8 wt.%.

Figure 7 shows a higher magnification micrograph of location B in Figure 2. There is no carbide remaining at the center of liquid "pocket" as in the "pocket" in Figure 3. While a network type structure is dominant, there is a "flat" region surrounding center network structure. It is observable that some secondary phases are distributed along the network. The semi-quantitative EDS analysis at locations A to C, as indicated in Figure 7, are shown in Table 2. Location A represents a network ridge area, location B represents the center of network cell and location C represents the flat edge area of the liquid "pocket".

Table 2. EDS results of locations A to C in Figure 7.

Element	Location A		Location B		Location C	
	wt. %	Error Wt. %	wt. %	Error Wt. %	wt. %	Error Wt. %
Mg	<0.109	0.045	<0.104	0.052	<0.111	0.055
Ta	<0.948	0.474	0.975	0.488	0.971	0.486
Mn	1.717	0.246	1.728	0.240	1.223	0.242
P	<0.151	0.076	<0.121	0.060	<0.119	0.059
S	<0.545	0.272	<0.465	0.232	<0.451	0.226
Si	1.527	0.305	1.460	0.283	0.624	0.299
Ni	15.663	0.437	18.503	0.464	18.395	0.474
Cr	30.104	0.401	29.199	0.384	28.181	0.382
Mo	<1.199	0.599	<1.008	0.504	<0.975	0.487
V	<0.157	0.078	<0.165	0.082	<0.162	0.081
Nb	13.623	0.702	1.971	0.497	<0.534	0.267
Ti	<0.148	0.074	<0.152	0.076	<0.152	0.076
Co	<0.386	0.193	<0.416	0.208	<0.418	0.209
Cu	0.440	0.220	<0.451	0.225	<0.451	0.226
Al	0.124	0.062	<0.112	0.056	<0.118	0.059
Fe	36.786	0.483	46.268	0.516	50.523	0.546

It is clear that location A shows the greatest Nb content while location C has the least Nb among the locations checked. Another evident chemistry difference between these locations is the Ni content. Location A has the lowest Ni content. S and P were also found along the network cell walls and this is in agreement with a previous investigation [1]. According to the previous investigation, if residual S and P are held in the liquid "pocket", the intergranular cracking tendency of base material HAZ is reduced. Another beneficial influence of liquid "pocket" type of liquation on hot cracking resistance is that the volume of the eutectic reaction constituents are always greater than that of the original solid solution, as a result of this "expansion", the liquation cracking tendency decreases.

Microstructural Examination of Gleeble Simulated HAZ Samples

Figure 8 shows the typical microstructure in a Gleeble simulated HAZ sample with a peak temperature of 1325°C (ZDT). It is clearly noted that a liquation reaction has occurred at ZDT. It should be noticed that the simulated thermal cycle reflects a SMAW condition, therefore, both the heating and cooling rates are less than those of resistance spot welding. This indicates that a rapid heating rate does not necessarily increase the extent of constitutional liquation in the weld HAZ as has been claimed previously [4]. There should be no strain effect on the microstructure in the sample with a temperature equivalent to the ZDT since the strength of the material is near zero.

Microstructures in GTA Weld HAZ

A HAZ sample was extracted from a Varestraint hot cracking tested specimen. It was reported previously that HR3C-C has a high hot cracking resistance compared to other advanced austenitic alloys [5]. The general microstructural morphology in the HAZ in and near the tested strained region is shown in Figure 9. It should be mentioned that the liquid "pocket" type liquation exists in the HAZ slightly away from the bent region. However, near the bent region, the evidence for a liquid "pocket" has disappeared. A higher magnification micrograph of a grain boundary in this region, which was not separated but did liquated during welding, is shown in Figure 10. A Nb rich secondary phase can be observed. An elemental line scan across the grain boundary was performed and the results are indicated in Figure 11. The cause of Nb enrichment is apparently grain boundary segregation prior to welding and diffusion during the welding thermal exposure.

SUMMARY

Eutectic reaction related grain boundary liquation is characteristic of the HAZ of HR3C-C material. It is revealed that the low HAZ hot cracking tendency of the 310HCbN materials is due to the fact that eutectic reaction related HAZ liquation is the dominant HAZ liquation mechanism. These results agree with the previous investigation that, when the Nb content level is about 0.45 wt.%, the Fe-25Cr-20Ni alloy system shows an eutectic reaction related HAZ liquation type. The greatest shrinkage stress occurs in the area adjacent to fusion boundary, as a result, HAZ liquation cracking can be initiated in this location. The HAZ liquation cracking tendency is significantly reduced in the Nb bearing 310HCbN materials due to the fact that a significant amount of eutectic reaction related HAZ liquation forms adjacent to the fusion boundary and that acts as a reservoir which can fill the grain boundary cracks if they form.

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Figure 1. Optical light microscopy image of typical microstructure of HAZ in the resistance spot weld sample.

Figure 2. SEM microstructure morphology in the HAZ of resistance spot weld of HR3C-C.

Figure 3. An enlarged area of location A in Figure 2.

Figure 4. Locations for conducting elemental line scan.

Figure 5. Elemental line scan results for line ab.

Figure 6. Elemental line scan results for line cd.

(a)

(b)

Figure 7. An enlarged area of location B in Figure 2. (a) Secondary electron image, (b) backscattered electron image.

Figure 8. Typical microstructure in a Gleeble simulated HAZ sample with a peak temperature of 1325°C (ZDE).

Figure 9. A general microstructural morphology in the base metal HAZ of Vareststraint tested sample near the bent region.

(a)

(b)

Figure 10. A higher magnification of a grain boundary, which is not separated but liquated during welding. (a) SEM morphology, (b) elemental dot mapping.

Figure 11. (a) Location where elemental line scans were conducted and (b) Results of an elemental line scan across a grain boundary.

WELD HOT CRACKING TEST FOR TUBING

ABSTRACT

A tubing weld cracking (TWC) test was developed for application involving advanced austenitic alloys (such as modified 800H and 310HCbN). Compared to the Finger hot cracking test, the TWC test shows an enhanced ability to evaluate the crack sensitivity of tubing materials. The TWC test can evaluate the cracking tendency of base as well as filler materials, thus, is a useful tool for tubing suppliers and filler producers and fabricators.

INTRODUCTION

Over the last several decades, significant effort has been expended, mainly in the US and Japan, to develop advanced austenitic alloys for superheater and reheater systems for power plants. Several candidate materials are now available for this type of elevated temperature service. Notably, there are increased requirements for quality and durability in the more severe environment for these newly developed materials principally because of the increased service temperature. In addition, fabrication considerations, especially weldability, during tubing material development and qualification are paramount.

For the advanced austenitic alloys, enhanced corrosion and oxidation resistance and high temperature mechanical properties have been achieved compared to conventional stainless steels. These enhancements partially result from the increased Cr and Ni content plus the addition of stabilizing alloying elements. In turn, however, the welding related issues become more metallurgically complicated. Filler material development is another issue which must be faced for welded fabrication since the filler material composition usually differs from the composition of the base material (matching of mechanical

properties). Thus, the simultaneous development of base and filler materials is the most rational approach during material development.

One of the major welding related issues for high temperature tubing and filler materials is weld hot cracking sensitivity. Therefore, it is necessary to conduct weldability evaluations during the base and filler material development stage as well as to be able to verify weldability on a heat to heat basis in the field. Many hot crack testing methods are available, however, none are considered applicable for direct tubing hot cracking assessment. In order to provide a simple tubing geometry related testing method, a tubing weld cracking (TWC) test was developed at The University of Tennessee. This test is a semi-self restraint hot cracking test and the basic concept of the TWC test allows not only evaluation of tubing base material hot cracking tendency but also enables cracking definition of filler materials. This method does not require specialized equipment. An added advantage is that TWC test can be conducted in the laboratory as well as in the field. The idea behind the TWC test originated in the Finger hot cracking test, for which six flat segments are employed. In the TWC segments of the tube are utilized. Thus, the fundamental basis for the TWC is similar to Finger hot cracking test.

Many hot cracking testing methods are available for assessing material hot cracking sensitivity. It has been well defined that two major sets of factors affect hot crack formation; the mechanical factors and metallurgical factors. Often, the mechanical factors are controlled and the hot cracking sensitivity is evaluated as a function of metallurgical factors for both laboratory and field type hot cracking tests. Basically, the methods can be classified into three categories in terms of strain characteristics, they are: self-restraint (bead-on-plate, Y-groove), semi-self-restraint (Finger hot cracking test) and augmented restraint (Varestraint and Sigmajig).

The choice of testing method is often based upon applications. For instance, for alloy development, detailed information is required, and thus, Varestraint and/or Sigmajig tests are selected. For general assessment or

comparison, simple methods are normally employed due to the fact that the simple test methods are usually more economic. Each testing approach has its own attributes, thus, reflecting different fabrication and/or service conditions. Proper techniques are necessary in order to more precisely define materials weldability.

MATERIALS AND EXPERIMENTS

Two types of advanced austenitic alloy tubing (modified 800H and 310HCbN) were employed in the initial assessment of the test method. The composition of these materials are given in Table 1.

Table 1. Composition of the materials.

Element	310HCbN (HR3C-C)	Mod. 800H (BWT7)
C	0.061	0.10
S	0.001	0.006
P	0.010	0.022
Cr	24.06	20.13
Ni	19.92	30.09
Mo	0.07	1.83
Mn	1.19	2.00
Si	0.36	0.19
V	0.04	0.59
Ti	<0.01	0.28
Nb	0.45	0.22
N	0.26	0.007
Cu	0.09	0.01
B	0.002	0.006
Fe	Bal.	Bal.

Three parameters; total crack length (TCL), maximum crack length (MCL) and the ratio of crack length to weld width (RCW) were measured and documented for the TWC tests. The GTA welding process with a current of 125A, at 12V in argon using weld speeds of 5 and 10 in/min were employed to fabricate circumferential welds in the test coupons.

The sample preparation procedures for TWC test coupons are described as follows: (1) sectioning 3" length of tubing into four 90° segments; (2) machining of segment surface; (3) clamping to make good surface contact, and (4) spot welding the pieces at both ends.

A circumferential autogeneous GTA weld was made at two locations on the assembled coupons for base metal assessment. For a consumable method of deposition a groove is employed. The testing procedures of TWC testing is schematically shown in Figure 1.

Four locations (gaps between each pair of segments), were examined and a crack length was measured at both sides of the weld. The total crack length (TCL), maximum crack length (MCL) and the ratio of crack length to weld width (RCW) were recorded .

RESULTS AND DISCUSSION

The TWC testing results for BWT7 and HR3C-C are given in Table 2. Two heat input levels were employed. It is clear that the modified 800H tubing material (BWT7) shows a significant hot cracking tendency compared to 310HCbN tubing material (HR3C-C). This result agrees with previous Varestraint and Finger hot cracking testing results [1]. The cracking extent is notably related to the alloy systems (Cr-Ni-Fe) and the different extent of residual elements (such as S, P and B).

The cracking tendency in TWC is also a function of weld energy input. For both 310HCbN and modified 800H alloys, in general, high weld energy input (Bead 1) shows a greater crack length than for low heat input (Bead 2). Above result is related thermal strain and its distributions during welding. A bar graph showing MCL, TCL and RCW for all TWC testing results is indicated in Figure 2. Obviously, BWT7 showed a greater cracking tendency compared to 310HCbN.

Table 2. Summary of crack length measured from TWC samples.

	HR3C-C			BWT7
	Sample 1 (mm)	Sample 2 (mm)	Average (mm)	Sample 1
	Bead 1	Bead 1	Bead 1	Bead 1
Location 1	0.19	0.59	0.39	4.07
Location 2	0.49	1.38	0.94	0.53
Location 3	0.38	1.10	0.74	2.40
Location 4	1.01	0.37	0.69	0
Max.	1.01	1.38	0.94	4.07
C/W*	0.13	0.21	0.17	0.54
Total	2.07	3.44	2.76	7.00
	Bead 2	Bead 2	Bead 2	Bead 2
Location 1	0.57	0.30	0.43	2.74
Location 2	0.15	0.39	0.27	0.27
Location 3	1.52	0.17	0.85	0.63
Location 4	0	0.04	0.02	0.35
Max.	1.52	0.39	0.42	2.74
C/W*	0.12	0.06	0.18	0.42
Total	2.24	0.90	1.57	3.99

* Ratio of maximum crack length to weld width.

The three criteria, MCL, TCL and RCW, showed good correlation and the interrelationship between them is indicated in Figure 3.

Comparison between TWC test and Finger hot crack test is shown in Figure 4. Evidently, the TWC test has an enhanced ability to define tubing hot cracking sensitivity. It is evident that TWC test shall be capable of filler material hot cracking resistance evaluations and this study is in progress.

SUMMARY

A newly developed tubing weld crack testing method provides a relatively simple but efficient approach to evaluate hot cracking sensitivity of base and filler materials. The TWC testing results agree with the results obtained from Varestraint and Finger hot cracking tests. In addition, tubing weld crack test showed an enhanced discrimination between materials in terms of hot cracking tendency contrasted to Finger hot cracking test.

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Figure 1. Schematic of TWC testing procedures.

Figure 2. TWC test results.

Figure 3. TWC test - TCL and RCW versus.

Figure 4. Comparison between Finger and TWC test results on modified 800H and 310HCbN materials.

METALLOGRAPHIC EXAMINATION OF SERVICE EXPOSED SA 213 (GRADE HR3C) HOT REHEAT TUBING AT TVA GALLATIN UNIT 2

INTRODUCTION

Approximately, a 1' long piece of HR3C type hot reheat tubing (reheat outlet pendant) in which severe corrosion/erosion was observed was extracted from TVA Gallatin Unit 2 for failure analysis. The total operational time to date of removal was 21,000 hours with an average operating temperature of 1025°F (lower than the designed operating temperature of 1050°F) at an operating pressure of 668 psi (same as the designed operating pressure). The average annual plant operation time is 7,000 hours. According to TVA, the tubing wall thickness was remarkably reduced and this reduction was believed due to slag liquid phase corrosion.

A preliminary metallographic examination was carried out on the samples extracted from the tube section in order to reveal, the microstructure, the existence of cracking and the fundamental failure mechanism. Three samples from different locations were sectioned and used for metallographic examination.

Significant cracking was observed in the HAZ of tube at the attachment welds and this cracking is believed to be reheat cracking based on the preliminary evaluation results. However, additional experiments must be conducted to provide a firm conclusion. It is evident that the coal-ash type corrosion/erosion took place at certain locations (fire side of the OD surface) and this significantly reduced the tube wall thickness. Some small cracks were detected in the tube surface at the locations where severe corrosion/erosion attack took place.

PRELIMINARY EXAMINATION RESULTS

A photograph showing the as-received tubing section is presented in Figure 1. The location and morphology of the attachment welds are indicated in the photograph. No severe corrosion was found on this side of tube surface. A powdery material is distributed longitudinally on the tubing surface and is composed of particles rich in Al, Si, As, Ca and Pb, the example of EDS spectrum of the particles is given in Appendix I.). The cutting lines for preparing fractographic and metallographic samples (A, B and C) are also indicated in Figure 1.

Figures 2 and 3 show the fire side of the tubing where serious corrosion/erosion attack took place. The width of the attacked region at both locations is approximately 35 mm. These two corrosion/erosion attacked locations are located an equal distance from the attachment welds. The tube cross section and the general attack locations are schematically illustrated in Figure 4.

Sample A, sectioned from the received tubing end that is remote from the attachment welds, was ultrasonically cleaned with in CRL solution for 1 hour to remove the thick oxide in order to reveal the original metal surface. OLM observations indicated that the corrosion/erosion attack occurred at the fire side of the tube belongs to a coal-ash corrosion/erosion type. No evidence of stress corrosion cracking is manifest from surface examination under OLM. The minimum wall thickness is 3 mm in the attacked region and the wall thickness for the non-attacked region is approximately 4.5 mm.

Sample B was cross-sectionally cut beside Sample A. Sample B was then mounted, polished and etched with aqua regia. Figure 5 shows a low magnification photomicrograph of the most severe corrosion/erosion attacked region. At the center of the wall cross section region, the grain boundaries etched darker compared to regions adjacent to both the OD and the ID surfaces. However, in a narrow band continuous to the OD surface, the etched

microstructural morphology is similar to that of the center region. The above observation elucidates that precipitation dissolution occurred at the locations adjacent to but not contiguous with the OD tubing surface and near the ID tubing surface. Most probably, precipitate dissolution occurred during tubing fabrication.

Higher magnification OLM microstructural morphologies of the cross section region adjacent to the OD surface, the center of cross section region and the region adjacent to the ID surface are presented in Figures 6 to 8, respectively. A greater amount of intergranular precipitates were observed in the center of cross section and adjacent to the OD surface than in the area adjacent to the ID surface. It is believed that the precipitate distribution across the tube cross section is associated with the temperature gradient. Nevertheless, remarkable intergranular precipitation occurred through the entire tube wall.

The appearance of the cross section of Sample C is schematically shown in Figure 9. As indicated in the figure, a lack of fusion exists between the attachment and welds. A photomicrograph showing the microstructural morphology at location "a" in Figure 9 is exhibited in Figure 10. In the weld HAZ at location "b", intergranular type cracking was found. Figure 11 shows the cracking morphology. It is evident that the cracking occurred adjacent to fusion line (experienced a high peak temperature) rather than at a lower temperature (900°C) where stress corrosion/corrosion cracking is most likely to occur. A hardness traverse obtained across the weld HAZ is presented in Figure 12. A slight increase in hardness is noted in the HAZ. It is to be noted that a decrease in hardness adjacent to the fusion line is anticipated. No evidence of liquation formation was observed along the grain boundaries in the HAZ. In addition, the cracking propagated parallel to the fusion line rather than perpendicular to fusion line as would be the case for hot cracking. Thus, from these preliminary observations, the intergranular cracking is defined as most likely due to reheat

cracking. However, more evidence must be obtained before a firm definition of the cracking type is possible.

Figure 13 shows the microstructural morphology at the location where severe general corrosion taken place on the tube OD surface in Sample C. Compared to the mid wall, heavy etched grain boundaries were detected in the region near the OD surface. Under a higher magnification (500X), precipitates can be observed along the grain boundaries were also found in this region and these cracks are most probably be associated with the attack.

SUMMARY AND SUGGESTED FURTHER WORK

From the preliminary investigation, it is clear that coal-ash type corrosion/erosion is responsible for a significant reduction in wall thickness at two locations. Intragranular precipitate dissolution occurred the areas adjacent to both the ID and OD surfaces (with a width of approximately 0.8 mm). Precipitation along the grain boundaries is significant. There is a gradient of precipitation with the greatest amount found near the OD surface region.

Under the attachment welds, intergranular HAZ cracking was observed and is believed to be within the category of reheat cracking. However, additional experiments must be performed in order to verify this conclusion.

Additional experiments suggested for the purpose of verification of the preliminary conclusions include SEM/EDS examination of the intergranular precipitates and localized corrosion sensitivity evaluation (e.g. ASTM 262A and/or EPR methods). SEM/EDS techniques should be enable identification of the type of the precipitates along the grain boundaries. If the precipitates can not be properly defined by using SEM/EDS, TEM metallographic evaluation should be employed.

Figure 1. Photomacrograph showing the tubing examined.

Figure 2. Photomacrograph showing attacked region 1.

Figure 3. Photomacrograph showing attacked region 2.

Figure 4. Schematic illustration of the tube cross section in Sample A.

Figure 5. Micrograph showing the microstructural morphology in the cross section sample where attack occurred. 19X

Figure 6. Higher magnification micrograph showing the microstructural morphology of the region adjacent to the OD surface. 500X

Figure 8. Higher magnification micrograph showing the microstructural morphology in the region near the ID surface. 500X

Figure 7. Higher magnification micrograph showing the microstructural morphology at the center of the tube wall. 500X

Figure 9. Schematic illustration of the tube cross section in Sample C.

Figure 10. Macrograph showing the microstructural morphology at location "a" in Figure 9, 12.5X

(a)

(b)

Figure 11. Weld HAZ cracking morphology at location "b". (a) 100X, (b) 500X.

Figure 12. A hardness traverse across the attachment weld HAZ.

(a)

(b)

Figure 13. Intergranular precipitation/attack morphology near OD surface. (a) 100X, (b) 500X.

Appendix I

Figure A-1. EDS spectrum of powder materials indicated in Figure 1.

TASK 2

Weldability and Metallographic Evaluation of a Newly Developed 310 Tantalum Bearing Stainless Steel: 310TaNb

WELDABILITY AND MICROSTRUCTURAL INVESTIGATION OF NEWLY DEVELOPED 310TaN ALLOY

INTRODUCTION

Several advanced austenitic alloy systems were developed in Japan and the U.S. during the last 15 years [1-3] for applications in power generation systems. At Oak Ridge National Laboratory (ORNL), modified 800H and modified 316 were developed in the early stages of investigation. Both alloy systems showed superior mechanical properties compared to conventional austenitic stainless steels. Many new alloy modifications were made after development of modified 800H and modified 316, the basic concept remained the same, to produce a certain amount of fine MC type carbides to strengthen the materials for both ambient and elevated temperature service. Although the Japanese versions of advanced austenitic alloys, 310HCbN (HR3C) and NF709, have been commercially utilized in heater and reheater tubing, the lack of the information regarding long term microstructural stability and weldability remain. Development and investigation of tantalum bearing 310 alloy were carried out at ORNL and the University of Tennessee (UT) and the 310TaN alloy showed an advantage among the advanced austenitic alloys in terms of microstructural stability, corrosion resistance and mechanical properties. According to the results from weldability and mechanical behavior evaluation, alloy 310TaN should be an elite candidate for power plant piping system applications and the other high temperature service uses.

It is well known that standard 310 stainless steel has a primary austenitic solidification mode, thus, a weld hot cracking tendency is expected. Ta is expected to have an effect on the primary solidification mode in 310 stainless steel similar to Nb which enhances primary ferritic solidification. Therefore, it is expected that the addition of Ta will aid in improving hot cracking resistance. The dissociation temperatures for TaC, TaN and Ta(C,N) are higher than that of

Ti and/or Nb type carbides, nitrides and/or carbonitrides. As a result, this will further enhance matrix weld hot cracking resistance.

MATERIALS AND EXPERIMENTAL PROCEDURES

One heat of laboratory prepared 310TaN was primarily used for this study. The heat was cold rolled from 0.5" to 0.124", then annealed in a vacuum at 1200°C for one hour. The chemical composition was analyzed by ABB-CE and the results indicated in Table 1.

Table 1. Chemical Composition of 310TaN (heat number: EJF23-3624).

C	Mn	P	S	Si	Ni	Cr	Mo	V	Nb
0.044	1.56	0.003	0.001	0.25	20.78	24.15	0.01	0.01	<0.01
Ti	Co	Cu	Al	B	N	O	Ta	Ca	Fe
<0.01	0.04	0.06	0.044	0.001	0.19	24.15	0.01	0.01	Bal.

Varestraint hot crack testing, Gleeble hot ductility testing, Gleeble HAZ simulation and resistance spot welding were employed in the weldability evaluation. Only limited testing could be performed in regard to weldability due to the limited material available.

An Aging study was performed in order to evaluate the long term mechanical and metallurgical behavior at elevated temperature. Two types of samples were evaluated. Samples were extracted from creep tests that were carried out ORNL. These samples with different Time-Temperature parameters and at different stresses conditions were used for metallographic evaluation and microhardness determinations. Aging study samples were extracted from Heat EJF23-3624. The samples were encapsulated in the quartz tube with a positive pressure of Ar. Figure 1 shows the capsulated specimen prior to heat treatment. The capsulated samples were furnace aged at various designed thermal parameters.

Localized corrosion resistance of the base metal, aged samples and Gleeble simulated samples was conducted using ASTM A262A and ASTM 108 G-92. All samples used for corrosion evaluation were polished up to 0.05 μ and cleaned with methanol before testing.

Metallographic evaluations were carried out using OLM, SEM and TEM. Limited particle extractions were performed on samples which were aged. The extracted powder samples were evaluated by X-ray diffraction and EDS.

RESULTS AND DISCUSSION

Varestraint Hot Crack Testing

The Varestraint test results for 310TaN are tabulated in Table 2 in terms of maximum crack length (MCL), total crack length (TCL) and cracked HAZ length (CHL) for the base metal HAZ. Figures 2 to 4 show the Varestraint hot cracking testing results (both maximum crack length and total crack length) in the weld metal HAZ, fusion zone and base metal HAZ, respectively. According to the Varestraint hot cracking testing criteria [1], the hot cracking tendency in all three zones is minimal. The CHL values for two samples, tested at 4% augmented strain, are 0.70 mm and 0.84 mm. The CHL is much smaller than that of other advanced austenitic alloys, such as HR3C, NF709, and modified 800H (5).

Table 2. Summary of Varestraint hot crack testing results for 310TaN.

Strain Rate	Weld Metal HAZ		Fusion Zone		Base Metal HAZ		
	MCL (mm)	TCL (mm)	MCL (mm)	TCL (mm)	MCL (mm)	TCL (mm)	CHL (mm)
4%	0.157	0.461	0.499	4.711	0.039	0.064	0.769
2%	0.099	0.362	0.413	4.623	0.039	0.039	-

As determined previously [4], good hot cracking resistance in 310TaN is expected mainly due to the fact that a favorable type of weld HAZ liquation is in evidence. The Fe-Ta and Fe-Nb phase diagrams are shown in Figures 5 and 6,

respectively. It is clear shown that Fe-Ta and Fe-Nb phase diagrams show the identical shape except for the reaction temperatures.

Figures 7 to 9 show the general microstructural morphologies of the weld metal HAZ, fusion zone and base metal HAZ in a Varestraint hot crack tested sample with 4% augmented strain.

The SEM microstructural morphology of the base metal HAZ in 310TaN (Varestraint hot cracking tested sample) is indicated in Figure 10. An eutectic type microstructure is exhibited along grain boundaries. The EDS results indicate that the particles are mainly Ta, Ta or Cr rich particles (as exhibited in Figures 11 and 12).

Gleeble Hot Ductility Testing

A summary of Gleeble hot ductility test results is listed in Table 3. Gleeble hot ductility and Varestraint hot cracking tests were conducted on 310TaN. Three tests were accomplished for both on-heating and on-cooling conditions during Gleeble hot ductility testing. In general, the 310TaN material showed good hot cracking resistance, especially in regard to base metal HAZ hot cracking resistance.

Figure 13 shows the hot ductility behavior of 310TaN. At a peak temperature of 1175°C, the material showed a ductility of 79%. An approximate estimation in which DRR, RDR and NDR are 15%, 15%, and 20C° is made based upon the limited test results. According to the hot ductility test criteria a good ductility recovery was obtained. Therefore, from the Gleeble hot ductility testing standpoint, 310TaN exhibit a minimal base metal HAZ hot cracking tendency.

Table 3. A summary of Gleeble hot ductility test results for 310TaN.

ZDT ¹ (°C)	ZST ² (°C)	RDR ³ (%)	DRR ⁴ (%)	DRR ⁵ (%)	NDR (C°)
1325	1350	25	44	11	20

- ¹. Zero ductility temperature.
- ². Zero strength temperature.
- ³. Ratio of ductility recover.
- ⁴. Ductility recover rate at rapid dropping temperature on-cooling curve.
- ⁵. Ductility recover rate at 1300°C.
- ⁶. Nil ductility temperature range.

The microstructural morphology of a Gleeble hot ductility tested sample with a test temperature at the ZDT (1325°C) is illustrated in Figure 14. It shows that liquation decorated the interface between particles and the grain matrix and that no significant grain boundary liquation is present. This is another reason that 310TaN has a high weld cracking resistance. A higher magnification micrograph (Figure 14) further illustrates that preferential precipitation in the 310TaN HAZ is located along segregation stringers ("ingotism" during material fabrication). However, it should be pointed out here that this type of segregation pattern may have some influence on high temperature long term service.

HAZ Microstructural Evaluation

It is known that the weld thermal exposure and microstructure in a particular location in a weld HAZ can be expanded in a Gleeble simulated HAZ sample. No grain growth was found in the Gleeble simulated HAZ samples for all the peak temperatures used compared to the base material (ASTM 6). Liquation was only observed in the simulated HAZ samples with a peak temperature of 1320°C and most of the liquation was located along the segregation bands. Grain boundary liquation was not revealed with OLM. These observations indicate that 310TaN has good HAZ weldability characteristics and is expected to show good corrosion resistance. Many researchers [8-11] have indicated that Ta bearing alloys showed a better hot cracking resistance compared to Nb bearing alloys and this agrees with the observations of this investigation.

Microstructural Evaluation and Hardness Assessment

of 310TaN Creep Tested Samples

Eight creep tested 310TaN samples were employed for metallographic and hardness evaluations. All metallographic samples were extracted from creep tested samples near the rupture location. The creep testing conditions for all samples are summarized in Table 4. It should be pointed out that the grain size was a function of distance from the rupture face.

Table 4. Creep test conditions.

Sample	TN27794	TN27796	TN27805	TN27802	TN27811	TN27858	TN27859	TN27873
UT Sample Code	UT1	UT2	UT3	UT4	UT5	UT6	UT7	UT8
Temperature (°C)	650	700	760	816	871	927	982	1038
Stress (MPa)	200	140	100	70	50	35	17.5	12
Time (hour)	3125	1611	1054	539	602	577	1794	845
Average ASTM Grain Size No.	1	4.5	2	2	3	4	3.5	3

Microstructural Evaluation

Figure 15 shows the typical microstructural appearance (sample UT1). The evidence of the precipitates distributed along grain boundaries and segregation inside of grain matrix along the rolling bands is exhibited in the figure. The average size of the fine grain boundary precipitates (mostly spherical) is smaller than $0.5\ \mu$. The larger size particles in the matrix are rod like with an average length of 5 to $10\ \mu$.

Figure 16 shows the microstructure of sample UT2. Clearly, the grain size is smaller and the particle size along the grain boundaries is larger than that observed in sample UT1 (Figure 15).

Figure 17 shows the microstructure of sample UT3. The distribution of secondary phases in terms of size and amount is similar to that observed in sample UT2 (Figure 16) except the matrix grain size (average) is greater than UT2.

Figure 18 shows the microstructure of sample UT4. Microstructurally, UT4 is similar to UT2 and UT3.

Figure 19 shows the microstructure of sample UT5. The size of secondary phases (precipitates along the grain boundaries and in the grain matrix) is greater than that observed in UT2, UT3 and UT4.

Figure 20 shows the microstructure in sample UT6. In this sample, the size of grain boundary precipitates decreases while the size of precipitation in grain matrix increases compared to UT5 (Figure 19).

Figure 21 shows the microstructure of sample UT7. A significant amount of fine precipitates is found in the matrix together with rod like (larger) distributed along the rolling direction.

Figure 22 shows the microstructure of sample UT8. "Bamboo"-like type secondary phase particles are distributed within the grain matrix. It is noticed that UT8 was tested at the highest temperature (1038°C) and with the longest test time (2540 hours).

Based upon the above optical microscopy observations, there is a need to carry out an electron microscopic evaluations in order to identify the precipitates and define their evolution mechanisms.

Microhardness Measurements and Thermal Effects on Microhardness

The microhardness were determined for evaluate the aging samples as summarized in Table 5

Table 5. Microhardness HV (0.5) measurement results.

UT1	UT2	UT3	UT4	UT5	UT6	UT7	UT8
223	184	196	202	169	201	204	236

It is clear from Table 2 that, the microhardness of the group of creep tested sample is within a range of 169 HV to 236 HV. It is assumed that the original microstructure was same for all creep tested samples, and this significant factor in the microstructural evaluation is the thermal effect.

The Larson-Miller parameter can be employed to indicate the thermal effect and Figures 23 and 24 indicate the relationship between hardness and Larson-Miller parameter ($^{\circ}\text{R} * (\text{C} + \log t)$) for a constants of 20 and 30, respectively. Clearly, for both constants (20 and 30), a clear relationship can be found (except for two points which are for the samples with aging temperatures of 650°C and 871°C which are slightly different than the general trend). UT1, the tested material, has the longest aging time and UT8 has the highest aging temperature (1038°C).

HAZ Liquation Behavior Study

From the weldability evaluation, it is apparent that 310TaN shows a superior hot cracking resistance compared to conventional austenitic stainless steels and the advanced austenitic alloys. It was reported by Swindeman [ii] that 310TaN showed an excellent creep strength compared to the other austenitic alloys. In order to reveal the fundamental mechanisms which provide the good weldability response of 310TaN, a melted and solidified weld nugget was made by electric resistance spot welding (a technique which was developed in the early stages of this project). It was found that 310TaN has Type II weld HAZ liquation, (eutectic reaction related liquation), therefore, a low weld metal HAZ hot cracking propensity.

The general morphology of the fusion zone and HAZ of the liquation study sample is shown in Figure 25. The partially melted HAZ (PMHAZ) in this sample is composed of both intergranular liquation and intragranular liquation. In fact, constitutional liquation is the dominant type of liquation in 310TaN which

overshadows low melting point segregation induced grain boundary liquation (in conventional austenitic stainless steel, usually, grain boundary type liquation is dominant).

Figure 26 depicts two "back-filled" HAZ grain boundary liquation. Interestingly, one of the cracks (marked A), was connected to a "liquid pocket" and the crack was completely filled by the liquid metal which flowed from the "liquid pocket." This observation, again, proves the previous hypothesis in which a "liquid pocket" that is caused by eutectic and peritectic reactions behaves as a reservoir and can provides liquid to grain boundaries opened at elevated temperature, thus, healing this.

Microstructural Evaluation of Aged 310TaN

OLM Microstructural Evaluation

The microstructural morphology in a sample aged at 600°C for 10 hours, shows a significant amount of rectangular shaped precipitates with an average length of 50 μ and width of 1 μ . Figure 27 shows the typical microstructural morphology of the sample aged at 600°C for 10 hours. Revealing another type of precipitate, small diameter spherical type carbides with an average diameter of 0.5 μ . Both types of precipitates are essentially intergranular precipitates.

The microstructural morphology in the sample aged at 600°C for 100 hours is shown in Figure 28. There is evidence for growth of the spherical shaped precipitates which were observed in the sample aged at 600°C for 100 hours. The rectangular shaped precipitates observed in the sample aged at 600°C for 10 hours apparently have to change shape and become irregular.

In the sample aged at 600°C for 1000 hours, most of the rectangular shaped precipitates observed in Figure 29 become larger. Significant particle growth and additional precipitation along the grain boundaries was found in this

sample. It was also observed that the grain size increased compared to the other samples aged at 600°C for 100 hours or less.

Figure 30 shows the microstructural condition of the sample aged at 800°C for 1 hour. Clearly, the predominant precipitates in this sample are irregular shaped. Further evidence of the particle shape evolution from rectangular to irregular shape is indicated.

Figure 31 shows the microstructural morphology in the sample aged at 800°C for 10 hours. The dominant precipitates are irregular shaped carbides and the size of the carbides increased as compared to that observed in Figure 30. The size of the grain boundary precipitates were also increased compared to that observed in Figure 30.

No remarkable change was observed in terms of the size of secondary phases in the sample aged at 800°C for 100 hours (Figure 32), as compared the sample aged at 800°C for 10 hours. The extent of precipitate both intergranular and intragranular becomes almost equivalent.

Figure 33 shows the microstructural morphology in the sample aged at 800°C for 1000 hours. In addition to the same types of precipitate which were observed in the sample aged at 800°C for 100 hours, some island shaped precipitates (with fine particles in it) and dark particles with various size were also observed in the sample aged at 800°C for 1000 hours.

Figure 34 shows the microstructural morphology in the 310TaN sample aged at 1000°C for 1 hour. No significant intergranular type precipitates were observed. The dominant precipitates are irregular shaped which are outlined upon etching while the spherical shaped particles were darkened upon etching.

Figure 35 shows the microstructural morphology in the sample aged at 1000°C for 10 hours. A significant increase of the grain boundary precipitates was observed compared to that in the sample aged at 1000°C for 1 hour.

A significant increase in the size of the secondary precipitates for both the irregular and spherical particles is shown in the sample aged at 1000°C for

100 hours as indicated in Figure 36 (especially for the spherical shaped precipitates).

The microstructural morphology in the sample aged at 1000°C for 1000 hours is shown in Figure 37. One observation different from that in the relatively short term aged sample is that the sample aged at 1000°C for 1000 hours showed the precipitates (both spherical and rectangular) to be darkened upon etching.

The microstructural morphology of the sample aged at 1200°C for 1 hour is illustrated in Figure 38. The irregular shaped particles are outlined while the spherical shaped particles are darkened during etching. Only a few intergranular precipitates were detected under this aging condition.

The microstructural morphology of the sample aged at 1200°C for 10 hours is illustrated in Figure 39. More extensive grain boundary segregation was observed in this sample compared to the sample aged at 1200°C for 1 hour. It was also noted that intragranular particles were darkened upon etching in this sample.

The microstructural morphology of the sample aged at 1200°C for 100 hours is illustrated in Figure 40. A similar microstructural and etching response was obtained in this sample compared to that observed in the sample aged at 1200°C for 10 hours.

The microstructural morphology of the sample aged at 1200°C for 1000 hours is illustrated in Figure 41. It is evident that significant grain growth has taken place. Most of particles have regular shapes.

SEM Microstructural Evaluation

The SEM evaluation of the aged samples is carried out in order to characterize the secondary phases. Several samples were selected based upon the information gained from OLM evaluation.

Figure 42 shows the SEM microstructural morphology in the 310TaN aged at 600°C for 10 hours. As observed in OLM, two dominant types of precipitates are rectangular and spherical shaped particles. Typical EDS spectra for the rectangular shaped and spherical shaped particles are exhibited in Figures 43 to 46 (Locations A to D in Figure 42). It is clear that both types of precipitate are rich in Si, Cr and Ta.

Figure 47 shows the SEM microstructural morphology of the 310TaN aged at 600°C for 100 hours. The EDS spectra of particles in Figure 47 are shown in Figures 48 (Location A in Figure 47) and 49 (Location B in Figure 47). The EDS examination results indicated that most of particles are Si, Cr, and Ta rich in this aged condition. The major of the grain boundary particles are of two types in terms of chemical composition. The typical EDS spectrum of a grain boundary precipitate is illustrated in Figure 50 (Location C in Figure 47). The EDS spectrum for the matrix is shown in Figure 51 (Location D in Figure 47).

Figure 52 shows the SEM microstructural morphology in the 310TaN sample aged at 600°C for 1000 hours. An increased amount of grain boundary precipitates was detected in this sample as compared to the samples aged at 600°C for 10 and 100 hours. The amount of Cr rich precipitates in this aged condition was enhanced although Si, Cr, and Ta rich particles are still predominant. The typical EDS spectrum for Cr rich and Si, Cr, and Ta rich particles are indicated in Figures 53 (Location A in Figure 52), 54 (Location B in Figure 52) and 55 (Location C in Figure 52).

Figure 56 shows the SEM microstructural morphology in the 310TaN sample aged at 800°C for 100 hours. Significant amount of intergranular type precipitates along the grain boundaries was found in this sample. It was also found, as expected, that Cr rich precipitates become the predominant precipitates under this aging condition. Figures 57 (Location A in Figure 56), 58 (Location B in Figure 56) and 59 (Location C in Figure 56) present the typical EDS spectra for particles as pointed out by arrows in Figure 57.

Figure 60 shows the SEM microstructural morphology in a 310TaN sample aged at 1000°C for 1000 hours. The EDS spectra for the particles and matrix are presented in Figures 61 to 65 (Locations A to E in Figure 60). Under this aging condition, a significant amount of fine intragranular precipitates was observed. Mainly, three types of the precipitates were in terms of the shape of the particles namely, irregular shaped, spherical shaped and blocky, respectively. Most of precipitates for all shapes are Si, Cr, and Ta rich particles.

To summarize the SEM/EDS evaluation results in the aged samples, two conclusions are obvious. One, the Si, Cr, and Ta rich particles are the predominant precipitates in 310TaN aged base metal. Secondly, all precipitates are thermodynamically stable (no significant growth of the precipitates were observed up to 1200°C for 1000 hours). This is characteristic of a material designed for elevated temperature applications.

Preliminary Assessment on Precipitation Kinetics of Secondary Phases in 310TaN

Based on above described microstructural investigations, the following precipitation evolution process for two types of particles can be can be illustrated in Figure 66. It is indicated that the maximum formation rate for Cr_{23}C_6 type carbides in 310TaN occurs at approximately 850°C. σ phase can form after aging above 1000°C in 310TaN.

Localized Corrosion Resistance Evaluation of 310TaN

It is well known that austenitic stainless steels may be prone to localized corrosion attack during service. the most common mode of localized corrosion attack in austenitic stainless steels are knife-line, intergranular corrosion (IGC) / intergranular stress corrosion cracking (IGSCC), pitting corrosion and crevice corrosion. For a materials which are subject to applications requiring welded fabrication, the development of the alloy should consider weld HAZ localized

corrosion resistance. The localized corrosion resistance of 310TaN was evaluated by the practices below.

ASTM A262 Practice A

ASTM A262 Practice A is a pass-fail type test, therefore, it is wise to conduct this practice prior to more extensive corrosion tests. The basic information derived from A262 Practice A is the tendency and level of the sensitization. Therefore, it not only applies to weld HAZ sensitization assessment, but also to base metal sensitization behavior which depends on the heat treatment and service exposure conditions. The preliminary results indicated that 310TaN passes ASTM A262 Practice A evaluation for all of the weld HAZ samples and for the base metal (solution treated condition).

A base metal sample and five Gleeble simulated HAZ samples were used for carry out this evaluation. Based upon the oxalic acid electrolytically etched microstructures observed, the sensitization tendency of the samples evaluated can be ranked as follows: HAZ (900°C), base metal, HAZ (1320°C), HAZ (1200°C), HAZ (1250°C) and HAZ (1100°C). Among them, only the HAZ sample with a peak temperature of 900°C and the base metal samples exhibited ditched grain boundaries although ditches did not completely surrounded a whole grain.

The metallographic morphologies of all above samples tested with A262 Practice A are presented in Figures 67 to 72. Clearly, the microstructures in these samples are classified "acceptable". Only the base metal and HAZ (900°C) samples showed evidence of grain boundary attack, however, the grain boundary attack did not completely surrounded a single grain, i.e. a dual structure. This result indicates that the solution treatment for the base metal was not optimum. When the weld HAZ peak temperature exceeds 1100°C, the Cr rich carbide ($M_{23}C_6$) start to dissolve into the matrix, a phenomenon which was observed in the Nb-bearing HR3C materials [12].

ASTM G108 (EPR) Evaluations

The results of electrochemical reactivation (EPR) evaluations of 310TaN agreed with that of ASTM A262 Practice A. It is certain that the all the Gleeble simulated weld HAZ sample and base metal solution treated (1200°C, 1 hour) passed with little HAZ sensitization tendency. The EPR results of 310TaN is summarized in Table 6 along with a data obtained from literature [3].

Table 6. EPR testing results of 310TaN.

	Test 1 Coulombs/cm ²	Test 2 Coulombs/cm ²	Average Coulombs/cm ²
Base Metal	0.074	0.044	0.059
HAZ (PT: 900°C)	0.038	0.158	0.098
HAZ (PT: 1100°C)	0.000	0.035	0.018
HAZ (PT: 1200°C)	0.002	0.059	0.031
HAZ (PT: 1250°C)	0.006	0.039	0.023
HAZ (PT: 1320°C)	0.022	0.069	0.046

* Solution treated at 1200°C for 1 hour.

The EPR testing results of 310TaN for all the Gleeble simulated samples and solution treated base are plotted in Figures 73 to 78. It is clear that, all the Gleeble simulated HAZ and base metal samples showed only a small reactivation tendency during the down scan. The sensitization tendency of 310TaN in terms of the EPR test can be ranked as: HAZ/900°C, base metal, HAZ/1320°C, HAZ/1100°C, HAZ/1250°C and HAZ/1200°C. The surface morphologies of the EPR tested samples are shown in Figures 79 to 80 for HAZ/900°C and HAZ/1100°C, respectively. HAZ sensitization tendency is closely related to microstructural changes during welding thermal exposure, mainly, secondary phase dissolution and redistribution. TaC and TaN possess high dissolution temperatures and are stable compared to Nb rich MC/NC type

secondary phases, therefore, 310TaN shows an enhanced sensitization resistance.

CONCLUSIONS

1. Alloy 310TaN possesses an excellent weld hot cracking resistance based upon the results from Varestraint hot crack testing, Gleeble hot ductility testing, and the weld HAZ liquation study.
2. The reason for the lower weld HAZ cracking tendency is associated with the eutectic reaction related type HAZ liquation exhibited in the 310TaN weldments.
3. 310TaN possesses excellent microstructural stability in terms of secondary phases evolution compared to other advanced austenitic alloys, such as modified 316 and HR3C. This consequence is partially due to the fact that TaN and/or TaC have a higher dissolution temperatures compared to the Nb and Ti rich nitride and carbides in the other system.
4. Preliminary microstructural investigation indicates that the precipitation evolution process is a very important factor affecting the base metal mechanical properties. Microhardness difference in the creep tested samples are basically associated with thermal effects (as evidenced using the Larson-Miller parameter). Ta, Si, and Cr rich fine precipitates were detected in both aged and solution treated samples. This type of precipitate can play a significant role in strengthening.
5. It was revealed through the corrosion evaluations of 310TaN that this alloy possesses excellent corrosion resistance, especially, localized corrosion resistance for the both base metal and the weld HAZ.

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Figure 1 Capsulated specimen prior to heat treatment.

Weld Metal HAZ Hot Cracking Behavior

Figure 2 Varestraint hot cracking testing results in 310TaN weld metal HAZ.

Fusion Zone Hot Cracking Behavior

Figure 3 Varestraint hot cracking testing results in 310TaN fusion zone.

Figure 4 Varestraint hot cracking testing results in 310TaN base metal HAZ.

Figure 5 Fe-Nb phase diagram.

200X

400X

Figure 7 Microstructural morphology of weld metal HAZ in 310TaN
Varestraint hot crack tested sample with 4% augmented strain.

200X

400X

Figure 8 Microstructural morphology of fusion zone in 310TaN
Varestraint hot crack tested sample with 4% augmented strain.

200X

400X

Figure 9 Microstructural morphology of base metal HAZ in 310TaN
Varestraint hot crack tested sample with 4% augmented strain.

Figure 10 SEM microstructural morphology of base metal HAZ in 310TaN Varestraint hot cracking tested sample.

Figure 11 EDS spectrum on the location A in Figure 10.

Figure 12 EDS spectrum on the location B in Figure 10.

Hot Ductility Behavior of 310TaN

(95/07/31)

Figure 13 Hot ductility behavior of 310TaN.

100X

400X

Figure 14 Microstructural morphology of the Gleeble hot ductility tested 310TaN sample with a test temperature of ZDT (1325°C).

100X

400X

Figure 15 Typical microstructure in creep tested 310TaN sample UT1.

100X

400X

Figure 16 Typical microstructure in creep tested 310TaN sample UT2.

100X

400X

Figure 17 Typical microstructure in creep tested 310TaN sample UT3.

100X

400X

Figure 18 Typical microstructure in creep tested 310TaN sample UT4.

100X

400X

Figure 19 Typical microstructure in creep tested 310TaN sample UT5.

100X

400X

Figure 20 Typical microstructure in creep tested 310TaN sample UT6.

100X

400X

Figure 21 Typical microstructure in creep tested 310TaN sample UT7.

100X

400X

Figure 22 Typical microstructure in creep tested 310TaN sample UT8.

Figure 23 Hardness as a function of Larson-Miller parameter for 310TaN sample with a of 20.

Figure 24 Hardness as a function of Larson-Miller parameter for 310TaN samples with a of 30.

100X

400X

Figure 25 Microstructural morphology of the fusion zone and HAZ of 310TaN liquation study sample.

400X

Figure 26 Microstructural morphology of two back filled HAZ grain boundary liquation cracks.

100X

1000X

Figure 27 Microstructural morphology of 310TaN sample aged at 600°C for 10 hours.

100X

1000X

Figure 28 Microstructural morphology of 310TaN sample aged at 600°C for 100 hours.

100X

1000X

Figure 29 Microstructural morphology of 310TaN sample aged at 600°C for 1000 hours.

100X

1000X

Figure 30 Microstructural morphology of 310TaN sample aged at 800°C for 1 hour.

100X

1000X

Figure 31 Microstructural morphology of 310TaN sample aged at 800°C for 10 hours.

100X

1000X

Figure 32 Microstructural morphology of 310TaN sample aged at 800°C for 100 hours.

100X

1000X

Figure 33 Microstructural morphology of 310TaN sample aged at 800°C for 1000 hours.

100X

1000X

Figure 34 Microstructural morphology of 310TaN sample aged at 1000°C for 1 hour.

100X

1000X

Figure 35 Microstructural morphology of 310TaN sample aged at 1000°C for 10 hours.

100X

1000X

Figure 36 Microstructural morphology of 310TaN sample aged at 1000°C for 100 hours.

100X

1000X

Figure 37 Microstructural morphology of 310TaN sample aged at 1000°C for 1000 hours.

100X

1000X

Figure 38 Microstructural morphology of 310TaN sample aged at 1200°C for 1 hour.

100X

1000X

Figure 39 Microstructural morphology of 310TaN sample aged at 1200°C for 10 hours.

100X

1000X

Figure 40 Microstructural morphology of 310TaN sample aged at 1200°C for 100 hours.

100X

1000X

Figure 41 Microstructural morphology of 310TaN sample aged at 1200°C for 1000 hours.

Figure 42 SEM microstructural morphology in 310TaN aged at 600°C for 10 hours.

Figure 43 EDS spectrum on the location A in Figure 43.

Figure 44 EDS spectrum on the location B in Figure 43.

Figure 45 EDS spectrum on the location C in Figure 43.

Figure 46 EDS spectrum on the location D in Figure 43.

Figure 47 SEM microstructural morphology in 310TaN aged at 600°C for 100 hours.

Figure 48 EDS spectrum on the location A in Figure 47.

Figure 49 EDS spectrum on the location B in Figure 47.

Figure 50 EDS spectrum on the location C in Figure 47.

Figure 51 EDS spectrum on the location D in Figure 47.

Figure 52 SEM microstructural morphology in 310TaN aged at 600°C for 1000 hours.

Figure 53 EDS spectrum on the location A in Figure 52.

Figure 54 EDS spectrum on the location B in Figure 52.

Figure 55 EDS spectrum on the location C in Figure 52.

Figure 56 SEM microstructural morphology in 310TaN aged at 800°C for 100 hours.

Figure 57 EDS spectrum on the location A in Figure 56.

Figure 58 EDS spectrum on the location B in Figure 56.

Figure 59 EDS spectrum on the location C in Figure 56.

Figure 60 SEM microstructural morphology in 310TaN aged at 1000°C for 1000 hours.

Figure 61 EDS spectrum on the location A in Figure 60.

Figure 62 EDS spectrum on the location B in Figure 60.

Figure 63 EDS spectrum on the location C in Figure 60.

Figure 64 EDS spectrum on the location D in Figure 60.

Figure 65 EDS spectrum on the location E in Figure 60.

Figure 66 Diagram for precipitation evolution

200X

400X

Figure 67 Metallographic morphology of an ASTM-A262 (Practice A) tested 310TaN sample: base metal.

Figure 68 Metallographic morphology of an ASTM-A262 (Practice A) tested 310TaN sample: HAZ (900°C).

200X

400X

Figure 69 Metallographic morphology of an ASTM-A262 (Practice A) tested 310TaN sample: HAZ (1000°C).

Metalllographic morphology of an ASTM-A262 (Practice A) tested
310TiN sample: HAZ (1200°C).

Figure 70

400X

200X

200X

400X

Figure 71 Metallographic morphology of an ASTM-A262 (Practice A) tested 310TaN sample: HAZ (1250°C).

200X

400X

Figure 72 Metallographic morphology of an ASTM-A262 (Practice A) tested 310TaN sample: HAZ (1320°C).

Data from "TaNBM96"

Figure 73 EPR testing result of 310TaN base metal with solution treated condition.

Figure 74 EPR testing result of 310TaN Gleeble simulated HAZ (900°C).

Figure 75 EPR testing result of 310TaN Gleeble simulated HAZ (1100°C).

Figure 76 EPR testing result of 310TaN Gleeble simulated HAZ (1200°C).

Data from "TaN1250"

Figure 77 EPR testing result of 310TaN Gleeble simulated HAZ (1250°C).

Figure 78 EPR testing result of 310TaN Gleeble simulated HAZ (1320°C).

Figure 79 Surface morphology of the EPR tested 310TaN Gleeble simulated sample (900°C).

Figure 80 Surface morphology of the EPR tested 310TaN Gleeble simulated sample (1100°C).

TASK 3

Iron Aluminide Cladding on Stainless Steel Tubing

GTA IRON ALUMINIDE CLADDING ON AUSTENITIC STAINLESS STEEL TUBING

ABSTRACT

Iron aluminide GTA overlay clad deposits were made on austenitic stainless steel plates and tubes with a variety of Al contents. The bonding between the iron-aluminide and the matrix was excellent. Cracking always occurred in room temperature overlay clad deposits when the Al content was greater than 18.4 at. %. The study of the effect of Al content and preheat/postweld heat treatments on cracking shows that at a 21-25 at. % Al content in the overlay clad deposits with a preheat of 300°C followed by a postweld heat at 600°C for one hour is optimum for cladding deposition.

Alloying additions of Ti, Si, Mo, and Mg were evaluated but each addition increased cracking tendency. The greatest increase in cracking tendency occurred with the addition of Mg. The Ti addition resulted in finer grains and overlay clad deposits with Ti and Ti+Si additions were welded successfully at a preheat of 300°C and postweld heat of 600°C for one hour.

An austenitic stainless steel tube was clad successfully with an iron aluminide of a deposit composition of 22.7 at. % Al.

INTRODUCTION

Although Fe-Al system alloys have been of interest since the 1930's [1], due to their high oxidation and corrosion resistance, low material density, and low cost, actual industrial structural applications have not been utilized due to the limited ductility at ambient temperatures and a decrease in strength above 600°C [2,3]. In recent years, low NO_x burners have been installed in many fossil fuel power plants in order to comply with new clean air regulations [4].

Due to the operating characteristics of these burners, boiler tube sulfidation corrosion is often enhanced and premature tube failures can occur. Failures due to oxidation and solid particle erosion are also a concern. Due to increasing demand for greater energy savings and further protection of the environment there is a strong demand to improve the efficiency of fossil fuel power plants [5]. The prevention of high temperature corrosion damage to boiler tubes has been a major topic of concern in order to ensure a stable supply of power. Therefore, a continuous effort on Fe-Al system alloys has been retained because of their attractive properties. Most research efforts have been devoted to understanding and improving the fabrication and mechanical properties of iron aluminides [6, 7, 8, 9]. Melting and fabrication of iron aluminides can be accomplished by many different methods including air induction melting, vacuum induction melting, vacuum arc remelting, and electroslag remelting [10, 11, 12]. Recent studies have shown that the mechanical properties of iron aluminides are sensitive to many factors including aluminum content, order, heat treatment, test temperature, alloying additions, environment, microstructure, and defects [13]. Some efforts have focused on cladding with iron aluminides on Cr-Mo steels and stainless steels [14, 15]. W.R. Mohn and M.J. Topolski investigated cladding of austenitic (304) stainless steel piping with iron aluminide by using hot extrusion techniques [16]. Although under-coating layer cracks were found in the Fe-Al coated tubing, the bonding between the iron aluminide and stainless steel was basically successful. However, it is evident that this type of tubing can not be used in any of the welding required fabrications since the joint structure will be different from both base matrix and coating. On the contrary, using GTA and GMA welding processes, an Fe-Al cladding can be performed in the last stages of structural fabrication, which eliminates many fabrication problems. In recent years, The University of Tennessee and the Oak Ridge National Laboratory investigated cladding of 304 stainless steels and Cr-Mo steels with iron aluminides using GTA and GMA processes [17].

Composite filler wires were developed and overlay clad deposits of iron aluminides have been successfully made on both 304 stainless steels and Cr-Mo steels. However, more work is needed to unravel the details of the effect of Al content in the overlay clad deposits and the effect of preheat and postweld heat parameters on cracking. It is necessary to optimize the Al content in overlay clad deposits such that high oxidation and corrosion resistance as well as good weldability is retained.

EXPERIMENTAL PROCEDURE

Materials

A tubular Fe-Al composite filler wire was used throughout this investigation consisting of an iron sheath of 0.825 mm thickness surrounding a core wire of 1.40 mm diameter pure aluminum as shown in Figure 1.

Table 1. Al and Alloying Compositions (filler material) for Overlay Clad Deposits (at. %) (Balance Fe)

	Al Content	Alloying Additions
GTA 1	16.3	--
GTA 2	20.3	--
GTA 3	22.6	--
GTA 4	24.3	--
GTA 5	27.8	--
GTA 6	31.1	--
GTA 7	32.6	--
GTA 8	34.0	--
GTA 9	36.7	--
GTA 10	27.8	2.12 Ti
GTA 11	27.8	0.68 Si
GTA 12	27.8	0.97 Mo
GTA 13	27.8	0.84 Mg
GTA 14	27.8	2.12 Ti+0.68 Si
GTA 15	27.8	0.97 Mo+0.68 Si

Austenitic stainless steel plates were used as the base metal (4"x2"x3/8") in the initial trials. Pure aluminum (ER1100) was added along the composite filler wire during welding in order to evaluate welds with differing Al content. Three side-by-side bead-on-plate overlay passes were GTA deposited on the stainless steel plates. Ti, Si, Mo, and Mg alloying element additions were made along with the Fe-Al composite filler wire in order to examine the effect of these alloying element additions on cracking. Table 1 shows the Al and alloy composition in the filler materials for the fifteen overlay clad deposits evaluated.

Test methods

The composite cladding fillers were deposited on the austenitic stainless steel using GTA welding. Filler metal was pre-placed on the plate. Welding conditions were varied in order to evaluate the effect of heat input on dilution in the overlay clad deposit. The welding conditions used are shown in Table 2.

Table 2. GTA Welding Parameters for Bead-On-Plate Clad Deposits

	GTA 1	GTA 2	GTA 3	GTA 4	GTA 5
Voltage (v)	16	17	17	18	18
Current (A)	160	180	200	220	240
Speed (in/min)	10	10	10	10	10
Heat Input (kJ/in)	15.4	18.4	20.4	23.8	25.9

RESULTS AND DISCUSSION

The effect of heat input on dilution

Five welding conditions were used to evaluate the effect of heat input on dilution. The results are shown in Figure 2, in which it is evident that dilution increases with an increase in heat input. Al content in overlay clad deposits depends on dilution when identical Al content filler wire is used. Therefore, it is

necessary to adopt the proper heat input to control dilution in order to obtain a clad deposit with optimum Al content. A welding heat input of 20.4 kJ/in. was used in this study (18.5% dilution) to optimize fusion and minimize dilution. Figure 3 shows the macrostructure of an overlay clad deposit with 20.4 kJ/in. welding heat input. Using the same welding heat input (20.4 kJ/in.), the Al contents of overlay clad deposits (taking into account dilution) with differing filler material Al contents are listed in Table 3.

Table 3. Al Content (at. %) in Overlay Clad Deposits Accounting for Dilution (Balance Fe)

	Al in filler	Al in deposit
GTA 1	16.3	13.3
GTA 2	20.3	16.5
GTA 3	22.6	18.4
GTA 4	24.3	19.8
GTA 5	27.8	22.7
GTA 6	31.1	25.3
GTA 7	32.6	26.6
GTA 8	34.0	27.7
GTA 9	36.7	29.9

* Welding heat input: 20.4 kJ/in.

Alloying additions of Si, Ti, Mo & Mg overlay clad deposits with 22.7 at. % Al are listed in Table 4.

Table 4. Alloying Additions (at. %) in Overlay Clad Deposits Accounting for Dilution (Balance Fe)

	Alloy Addition in Filler Material	Alloy Addition in Deposit
GTA 10	2.12 Ti	1.73 Ti
GTA 11	0.68 Si	0.55 Si
GTA 12	0.97 Mo	0.79 Mo
GTA 13	0.84 Mg	0.68 Mg
GTA 14	2.12 Ti + 0.68 Si	1.73 Ti+0.55 Si
GTA 15	0.97 Mo+0.68 Si	0.79 Mo + 0.55 Si

* Welding heat input: 20.4 kJ/in.

** Al content: 22.7 at. %

The effect of Al content on cracking

Iron aluminide GTA overlay clad deposits were made on stainless steel plates with the filler material Al content ranging from 16.3 at. % to 36.7 at. % (13.3-29.9 at. % including dilution). Four different combinations of preheat/postweld heat conditions were adopted: no preheat or postweld heat, a preheat of 250°C/postweld heat of 400°C for 1 hour, a preheat of 300°C/postweld heat of 600°C for 1 hour, and a preheat of 350°C/postweld heat of 750°C for 1 hour. Figure 4 shows the effect of Al content on cracking for the different welding heat conditions. The variation in cracking tendency with Al content for the room temperature (no preheat or postweld heat) deposition conditions is almost the same as for a preheat of 250°C/postweld heat of 400°C for 1 hour. A peak in the cracking tendency occurred at a deposit Al content of 19.8 at. % Al. When the deposit Al content was increased to 22.7 at. %, the cracking tendency decreased. Upon increasing the deposit Al content beyond 22.7 at. %, the cracking tendency again increased. It is evident that a 22.7 at. % deposit Al results in a minimum cracking tendency for deposit Al contents greater than 19.8 at. %. C.G. McKamey mentioned a similar result in Fe-Al binary alloys in regard to mechanical properties [18].

From the Fe-Al phase diagram (Figure 5) [19,20], it can be seen that the iron-rich iron aluminides in the range of 15-30 at. % Al content can be divided into three regions: a disordered solid solution of aluminum in iron (a) which is stable at lower Al content (up to approximately 20 at. % Al), a disordered solid solution (a) with two ordered phases (Fe_3Al with the DO_3 structure and FeAl with an imperfectly ordered B2 structure) in the range of 20-24 at. % Al content, two ordered phases (DO_3 and B2) at an Al content greater than 24 at. %.

Figures 6-9 show the microstructures of the overlay clad deposits with 13.3 at. %, 16.5 at. %, 18.4 at. %, and 19.8 at. % Al contents respectively. As

mentioned above, all of the microstructures with four different Al contents are the disordered solid solution α . They exhibit similar grain sizes and fine inclusion distributions within the matrix and thus there is little difference in microstructure. Figure 10 shows the microstructure of an overlay clad deposit with 22.7 at. % Al. It can be seen that there is an increase in the number and size of the inclusions distributed in the disordered solid solution α with two ordered phases DO_3 and B2 matrix. Figures 11-14 show the microstructures of the overlay clad deposits with 25.3 at. %, 26.6 at. %, 27.7 at. %, and 29.9 at. % Al contents respectively. From the Fe-Al phase diagram, it is apparent that all of the structures are in the ordered DO_3 and B2 phases region. With the exception of the increase in size and number of inclusions in the matrix, a change in structures can not be distinguished using OLM.

Figures 15-17 show the cracks which occurred in the overlay clad deposits with 19.8 at. %, 22.7 at. %, and 29.9 at. % Al contents respectively. Most cracks are transgranular. Figure 18 shows the results of PT inspection. It is evident that the overlay clad deposits with 29.9 at. % Al content are the most susceptible to cracking.

The Effect of Preheat and Postweld Heat Treatment on Cracking

As discussed in Section 3.2, when the Al content was greater than 18.4 at. %, cracking occurred in iron aluminide overlay clad deposits for room temperature welding. (However, the Al content should be greater than 20 at. % in the overlay for good corrosion resistance.^[15]) Therefore, it is necessary to adopt suitable preheat and postweld heat treatments to obtain a crack free optimum iron aluminide overlay clad deposit with over 20 at. % Al.

Table 5 shows the effect of preheat and postweld heat treatment on cracking.

Table 5 Effect of Preheat and Postweld Heat Treatment on Cracking

Al Content in Deposit [atm%]	Total Crack Length (mm)			
	Preheat: 25°C Postheat: 25°C	Preheat: 250°C Postheat: 400°C	Preheat: 300°C Postheat: 600°C	Preheat: 350°C Postheat: 750°C
13.3	0	0	0	0
16.5	0	0	0	0
18.4	12	0	0	0
19.8	99	43	0	0
22.7	52	8	0	0
25.3	80	26	0	0
26.6	131	49	0	0
27.7	142	80	20	0
29.9	191	140	82	12

When the Al content was less than 16.5 at. % there was no cracking. With a preheat of 250°C and a postweld heat of 400°C for 1 hour the Al content can be increased to 18.4 at. % without cracking. Further increasing the preheat temperature to 300°C and the postweld heat temperature to 600°C for 1 hour resulted in crack free overlay clad deposits with Al content up to 26.6 at. %. When the Al content was greater than 29.9 at. %, there was cracking even when the preheat and postweld heat temperatures were increased to 350°C and 750°C respectively. (Further increase in the preheat and postweld heat temperatures would be difficult for industrial applications.) As can be seen from the above results, a preheat of 300°C and a postweld heat of 600°C with an Al content from 21-25 at. % should be considered optimum for crack free clad deposits with suitable corrosion resistance.

Figures 19-20 show the microstructures with 19.8 at. % Al content in a preheat of 250°C/postweld heat of 400°C for 1 hour and a preheat of 300°C/postweld heat of 600°C for 1 hour respectively. As compared with the microstructure for the room temperature welding condition (Figure 9), there is no

difference. Figures 21-22 show the microstructures with 22.7 at. % Al content with a preheat of 250°C/postweld heat of 400°C for 1 hour and a preheat of 300°C/postweld heat of 600°C for 1 hour respectively. The microstructures with 29.9 at. % Al content for three different welding conditions are shown in Figures 23-25. It is evident that preheat and postweld heat treatments have no effect on microstructure. However, the hardness in the overlay clad deposits increased upon increasing the temperature of preheat and postweld heat treatments as shown in Figure 26. Much work has shown that iron aluminide is susceptible to environmental hydrogen embrittlement. The possible reasons that preheat and postweld heat treatment reduced cracking tendency is that preheat reduced the moisture on the surface of Fe-Al composite filler wire and austenitic stainless plate and preheat and postweld heat treatments improved the level and distribution of the strain induced by welding.

The effect of alloying additions on cracking

Significant work has shown that the mechanical, corrosion and welding properties of iron aluminides are sensitive to alloying additions. Many reviews have mentioned the results of past studies [18, 21]. In this study, the effect of Ti, Si, Mo, and Mg on cracking upon welding was investigated. It is evident from the results shown in Figure 27 that all alloying additions increased the cracking tendency in both room temperature and at a preheat of 250°C/postweld heat of 400°C for 1 hour. When the preheat temperature was increased to 300°C and the postweld heat temperature to 600°C for 1 hour, there was no cracking in the overlay clad deposits with 2.12 at. % Ti, and 2.12 at. % Ti + 0.68 at. % Si respectively. The greatest cracking increase occurred with a Mg addition even with a preheat of 300°C/postweld heat of 600°C for 1 hour.

Figures 28-29 show the microstructure with 1.73 at. % Ti, and 1.73 at. % Ti + 0.55 at. % Si additions. It is evident that the grain size is smaller as

compared to the grain size of the microstructure with only 22.7 at. % Al. This grain size refinement is the reason that Ti and Ti+Si additions were added. Figure 30 shows the microstructure with 0.68 at. % Mg, in which it can be seen that cracking propagates along grain boundaries. Figures 31-33 show the microstructure with 0.55 at. % Si, 0.79 at. % Mo, and 0.79 at. % Si + 0.55 at. % Mo additions. There were no differences in grain size, inclusion size or inclusion distribution as compared to the microstructure with 22.7 at. % Al.

Austenitic Stainless Steel Tube Cladding

In an effort to further substantiate the aforementioned data, an iron aluminide GTA overlay clad deposit was made on an austenitic stainless steel hot reheat tube (HR3C). The tube was 2 3/8" OD with a 1/4" wall thickness. In order to maximize the corrosion resistance and eliminate cracking within the overlay clad deposit, an Al content of 22.7 at. % was chosen in addition to a preheat of 300°C and postweld heat of 600°C for 1 hour heat treatment. Alloy elements were not utilized because of the lack of beneficial results pertaining to crack prevention. These parameters resulted in a successfully welded overlay clad sample that was crack free. Figure 34 shows the hot reheat tube (HR3C) overlay clad sample.

CONCLUSIONS

1. Crack free, iron aluminide overlay clad deposits on austenitic stainless steel tubing at 22.7 at. % Al were successfully produced by the GTA process. This approach provides a promising opportunity for industry application of iron aluminides.
2. Preheat and postweld heat treatments are necessary to prevent iron aluminide overlay clad deposits from cracking when the Al content is greater than 18.4 at. %. Cracking tendency decreased with increasing

- preheat and postweld heat temperatures. A preheat of 300°C and postweld heat of 600°C for 1 hour welding condition in which overlay clad deposits with 21-25 at. % Al contents were welded without cracking was suggested.
3. The cracking tendency was high when the Al content was less than 19.8 at. %. Increasing Al content to 22.7 at. % resulted in a reduction in cracking tendency. When the Al content was increased beyond 22.7 at. %, the cracking tendency increased. There was a minimum cracking tendency at 22.7 at. %.
 4. Dilution in the welding fusion zone depended on welding heat input. Dilution increased with increasing welding heat input. Based on dilution, the Al content in the overlay clad deposit was estimated.
 5. Alloying additions of Ti, Si, Mo, and Mg increased cracking tendency. The greatest increase in cracking tendency occurred with the of Mg additions. The Ti addition resulted in finer grains and overlay clad deposits with Ti and Ti+Si additions were welded successfully at a preheat of 300°C and postweld heat of 600°C.

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Figure 1. Cross-sectional photomicrograph of the Fe-Al composite filler wire.

Figure 2. Effect of heat input on dilution.

Figure 3. Macrostructure of overlay clad deposit (Welding heat input: 20.4 kJ/in.).

Figure 4. Effect of Al Content on Cracking Tendency.

Figure 5. Fe-Al phase diagram.

Figure 6. Microstructure of the overlay clad deposit with 13.3 at. % Al content (room temperature).

Figure 7. Microstructure of the overlay clad deposit with 16.5 at. % Al content (room temperature).

Figure 8. Microstructure of the overlay clad deposit with 18.4 at. % Al content (room temperature).

Figure 9. Microstructure of the overlay clad deposit with 19.8 at. % Al content (room temperature).

Figure 10. Microstructure of the overlay clad deposit with 22.7 at. % Al content (room temperature).

Figure 11. Microstructure of the overlay clad deposit with 25.3 at. % Al content (room temperature).

Figure 12. Microstructure of the overlay clad deposit with 26.6 at. % Al content (room temperature).

Figure 13. Microstructure of the overlay clad deposit with 27.7 at. % Al content (room temperature).

Figure 14. Microstructure of the overlay clad deposit with 29.9 at. % Al content (room temperature).

Figure 15. Cracks in the overlay clad deposit with 19.8 at. % Al content (room temperature).

Figure 16. Cracks in the overlay clad deposit with 22.7 at. % Al content (room temperature).

Figure 17. Cracks in the overlay clad deposit with 29.9 at. % Al content (room temperature).

Figure 18. PT inspection of overlay clad deposits with different Al contents (room temperature). 1.7X

Figure 19. Microstructure of the overlay clad deposit with 19.8 at. % Al content (preheat of 250°C, postweld heat of 400°C for 1 hour).

Figure 20. Microstructure of the overlay clad deposit with 19.8 at. % Al content (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 21. Microstructure of the overlay clad deposit with 22.7 at. % Al content (preheat of 250°C, postweld heat of 400°C for 1 hour).

Figure 22. Microstructure of the overlay clad deposit with 22.7 at. % Al content (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 23. Microstructure of the overlay clad deposit with 29.9 at. % Al content (preheat of 250°C, postweld heat of 400°C for 1 hour).

Figure 24. Microstructure of the overlay clad deposit with 29.9 at. % Al content (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 25. Microstructure of the overlay clad deposit with 29.9 at. % Al content (preheat of 350°C, postweld heat of 750°C for 1 hour).

Figure 26. Effect of preheat and postweld heat treatments on hardness.

Figure 27. Effect of alloying addition on cracking.

Figure 28. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 1.73 at. % Ti addition (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 29. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 1.73 at. % Ti + 0.55 at. % Si additions (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 30. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 0.68 at. % Mg addition (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 31. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 0.55 at. % Si addition (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 32. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 0.79 at. % Mo addition (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 33. Microstructure of the overlay clad deposit with 22.7 at. % Al content and 0.79 at. % Mo + 0.55 at. % Si additions (preheat of 300°C, postweld heat of 600°C for 1 hour).

Figure 34. Tube overlay cladding sample. 1X

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