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**PROCESSING AND STRUCTURE OF
INSITU Fe-Al ALLOYS PRODUCED BY
GAS TUNGSTEN ARC WELDING**

February 14, 1997

Report Prepared by

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**PROCESSING AND STRUCTURE OF INSITU Fe-Al ALLOYS PRODUCED BY
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1.0 EXECUTIVE SUMMARY

Iron aluminide weld overlays are being investigated for corrosion and erosion protection of boiler tubes in low NO_x burners. The primary objective of the research is to identify overlay compositions which can be deposited in a crack-free condition and provide corrosion protection in moderately reducing environments. In the current phase of work, Fe-Al alloy weld overlays were produced by depositing commercially pure aluminum wire on to low carbon steel substrates using Gas Tungsten Arc Welding. A systematic variation of the wire feed speed and current, two major factors affecting dilution, resulted in a variation in aluminum contents of the welds ranging from 3-42 wt% aluminum. The aluminum content was observed to increase with wire feed speed and a decrease in the current. The aluminum content was also found to affect the cracking susceptibility of the overlays. At 10wt% aluminum, few to no cracks were observed in the deposits. Above this value, cracking was prevalent throughout the weld. In addition, two types of microstructures were found correlating to different concentrations of aluminum. A homogeneous matrix with second phase particles consisting of coarse columnar grains was found for low aluminum concentrations. With higher aluminum contents, a two-phase constituent was observed to surround primary dendrites growing from the substrate. The transition of the microstructures occurred between 24 and 32 wt% Al.

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2.0 INTRODUCTION

At present, new clean air regulations have required electric utility companies to decrease NO_x emissions, leading to a reducing/sulfidizing condition within the boiler. With these new low NO_x boiler environments, protective oxide scales once formed by typical low alloy Cr-Mo steels are being replaced by less protective sulfide scales [1]. As a result, the conventional materials used for the boiler tubes are experiencing unacceptable corrosion rates resulting in forced outages. In order to reduce corrosion of the boiler tubes and preserve their integrity, new material systems are being investigated. It has been shown that of the materials tested, iron aluminides possess *both* excellent sulfidation and oxidation resistance in very aggressive environments [2,3].

However, iron aluminides are unsuitable for direct replacement of present boiler tubes. Limited room temperature ductility leads to difficulty in the manufacturing and a sharp drop in strength above 600°C results in the material being inadequate during service [4]. Therefore, coatings are being applied to the existing tubes to override these obstacles. Two primary application methods are thermal spray and weld overlay processes. While these two types of processing have similar advantages (i.e. relatively inexpensive, shop and field application, wide range of material systems), there are distinctions which separate them. Thermal sprays, while initially the most economical, contain inherent oxides and porosity due to processing [5]. If interconnected, localized corrosion of the substrate may occur due to transport of the corrosive specie through the coating. In addition, due to the coating having only a mechanical bond with the substrate, spallation may occur in service [6]. Weld overlays, on the other hand, have metallurgical bonds that provide better strength in thermal cyclic environments. Added to fewer inherent processing defects, weld overlays appear to be the most evident choice for application of iron aluminide coatings.

The overall objective of this work is to study various iron aluminide weld overlay compositions, specifically between 8-16 wt% Al, and relate their processing and microstructure to properties which are critical in low NO_x boiler environments: weldability, oxidation, sulfidation, and erosion resistance. These results will be compared with other weld overlay candidates presently

being considered. The goal of the current research presented in this paper is to produce Fe-Al alloy weld overlays by depositing commercially pure aluminum wire on to low carbon steel substrates using GTAW. A systematic variation of the wire feed speed and current, two major factors affecting dilution [7], will yield varying aluminum contents in the weld. The results will indicate the maximum aluminum content present in the weld overlay that does not produce cracks. Future investigation will revolve around extensive laboratory evaluation of the above properties on selected coatings in order to compare them to other coating systems.

3.0 EXPERIMENTAL PROCEDURE

Samples were produced using Gas Tungsten Arc Welding (GTAW) conducted in a fully automated welding research laboratory at Lehigh University. Commercially pure aluminum wire (99.96%) with a 1.2mm diameter was directly fed into the weld pool of a low carbon steel substrate with dimensions of 12" x 4" x 0.25". The substrates were ground with 120 grit silicon carbide paper and cleaned with alcohol to remove any surface oxides. Bolts located at each corner were used to constrain the plate to the table. Pre-heat and post-weld heat treatments were not conducted. A single weld bead was placed in the center of each plate with a constant torch travel speed of 2.0 mm/sec. The voltage was kept constant at approximately 12V while the wire feed speed and current were varied. The plates were not released until they cooled to room temperature, at which point, a dye penetrant technique was used to assess cracking of the weld beads. Cracks within 1" of the beginning or end of the weld were not counted due to inconsistent microstructures with the rest of the weld overlay due to the start-up and end of processing.

Samples were removed from the bulk using an abrasive cut-off wheel and cross-sections mounted in cold-setting epoxy. Preparation of the mounts consisted of grinding to 600 grit silicon carbide paper and a subsequent polish, finishing with 0.04 μ m colloidal silica. For microstructural evaluation, a solution of 60ml methanol, 40ml nitric acid, and 20ml hydrochloric acid was used. Microstructural characterization was performed with both light optical microscopy (LOM) and scanning-electron microscopy (SEM).

Cross-sectional areas of the weld overlay (A_o) and melted substrate (A_s) were measured using a LECO 2001 Quantitative Image Analysis (QIA) system. The weight percentages of each element can be calculated using the cross-sectional areas measured from above:

$$\text{Overlay aluminum wt\%} = \frac{A_o \times \rho_{Al}}{(A_o \times \rho_{Al}) + (A_s \times \rho_{Fe})} \quad (1)$$

where ρ_{Al} and ρ_{Fe} are the density of aluminum and iron, respectively.

Electron probe microanalysis (EPMA) was conducted on a JEOL 733 SuperProbe equipped with wavelength dispersive spectrometry. The accelerating voltage and probe current were set at 15kV and 21nA, respectively. K_α x-ray lines were analyzed and counts converted to weight percentages using a ϕ (ρz) correction scheme. A line trace from the weld overlay surface into the substrate was performed. In addition, at least five probe scans were conducted in random areas through out the weld overlay at a magnification of 10,000x to ensure the overall homogeneity of the deposit. Weld overlay compositions were calculated by averaging the probe scans, as well as the probe points that were within the bulk of the overlay.

4.0 RESULTS AND DISCUSSION

A sample matrix was produced to obtain a wide range of aluminum contents in the weld overlay and determine the minimum aluminum wt % to cause cracking. Wire feed speeds used were 10, 15, 20, 25, 30, 35, and 55 mm/sec while the current was varied between 250 and 300A with steps of 25A. Single weld beads were deposited to ensure that the microstructure and compositions of the overlays were not complicated by overlapping passes.

I. Microstructure

Figure 1 shows the experimental matrix for this research. Samples were labeled as either cracked or not cracked. The number of cracks that occurred are located next to the symbols, with **M** signifying more than 15 cracks in the sample. A combination of high feed speed and low current

was found to produce cracked overlays. This processing scheme results in high aluminum in the overlay due to reduced mixing between the aluminum and iron [7]. During deposition, current flowing through the arc is the main source of heat that melts the base metal and filler wire. While in the liquid state, mixing occurs to produce an alloy of the two constituents with a composition depending upon the volume of each material that was melted. For a fixed wire feed speed, decreasing the current results in a smaller volume of melted substrate that can mix with the melted wire, thus leading to higher aluminum concentrations in the overlay. Conversely, for a fixed current, increasing the wire feed speed allows for more melted aluminum to mix with the base metal, again resulting in higher aluminum contents. The high aluminum overlays have previously been found to produce unsound deposits [8,9]. Samples located at the crack/no crack boundary were reproducible.

Figure 1: Experimental matrix produced by varying aluminum wire feed speed and current. Each data point (box) represents an overlay deposited at that processing condition, and was subsequently labeled as cracked or no crack. Numbers next to the cracked data points signify how many cracks occurred for the sample, whereas samples having more than 15 cracks have **M**.

Cracking was primarily found to occur during the process and while the substrate was cooling down to room temperature. In addition, cracking was also observed several days after welding. This type of failure is referred to as cold cracking and normally associated with environmental or hydrogen embrittlement [10] of the alloy. Typical macrographs of the samples after application of the dye technique can be seen in Figure 2. Figure 2a shows a weld produced in the “no crack” regime and 2b with multiple cracks. The majority of the cracks are found in one specific direction, i.e. perpendicular to the direction of welding.

Cross-sectional micrographs of the weld overlays showed two different types of microstructures. For samples deposited with a wire feed speed less than 55mm/sec, a homogeneous matrix with second phase particles was found, Figure 3. The second phase was found both in the coarse columnar grains and the grain boundaries. It was observed that cracking of the overlay was both inter- and transgranular. Figure 4 shows the second type of microstructure seen for the welds deposited with a wire feed rate of 55mm/sec. At very low magnification, a change in microstructure can be seen throughout the weld overlay. Large grains near the fusion line in the partially mixed zone precede dendrites that continue to grow into the weld pool. At higher magnifications (Figure 4b), a two-phase constituent is observed surrounding the primary dendrites. Though not quantitatively measured, the percentage of the two phase constituent was observed to decrease with an increase in current (i.e. decreasing aluminum content), Figure 5. Again, cracking was not confined to between grains, but can also be seen traversing through the dendrites, Figure 5b.

a

b

Figure 2: Typical macrographs of single bead weld overlays after application of the dye penetrant technique. a) not cracked, b) cracked

Figure 3: Light optical micrograph showing a) the as-polished homogeneous matrix with second phase particles and b) the etched microstructure consisting of coarse columnar grains. This type of microstructure was found for all samples deposited with wire feed speeds less than 5mm/sec.

Figure 4: Back-scattered images showing the microstructure found for all samples deposited with wire feed speeds of 55mm/sec. Coarse columnar grains near the fusion line (**L**) precede the dendritic structure (**D**) surrounded by a two-phase constituent (**C**).

Figure 5: Scanning electron micrographs showing a qualitative increase in the volume percent of primary dendrites with increasing current (i.e. decreasing aluminum concentration) at a constant wire feed speed of 55mm/sec. a) 41.8wt%Al with 250A, b) 38.4wt% Al with 275A, c) 32.8wt% with 300A

II. Composition

As an initial check on the processing parameters, a volume balance equation for aluminum was used [7]:

$$A_w \times S_w = A_o \times S_T \quad (2)$$

where A_w is the cross-sectional area of the wire, and S_w and S_T are the wire feed speed and the travel speed of the torch, respectively. The left hand side of the equation represents the amount of aluminum being delivered during processing, while the right side represents the amount of aluminum deposited. Since A_w , S_w , and S_T are either constant or designated values, the area of the weld overlay should be easily calculated. Table I summarizes the results for selected samples. Reasonable agreement between the calculated and measured values was found. The most probable error lies in the wire feed speed being different from that programmed into the computer parameters, since a feed back mechanism was not utilized for these samples. At present, experiments are being conducted to determine if this is the case. Another possible error may be in the assumption that the cross-sectional areas of both the weld and the melted substrate are constant.

Table I: Calculated and experimental cross-sectional areas (in mm²) of weld overlays (A_o)

Feed Rates (mm/sec)	Calculated areas from eq. (2)	Measured areas for 250A	Measured areas for 275A	Measured areas for 300A
15	8.5	10.6 ± 1.1	11.9 ± 0.9	11.1 ± 1.2
35	19.8	20.5 ± 1.2	19.3 ± 1.0	20.5 ± 1.1
55	31.1	27.5 ± 1.0	27.5 ± 1.0	29.4 ± 0.8

Using the areas of the weld deposit and melted substrate, an estimate for the aluminum content in the weld overlay can be determined using eq. (1). Table II summarizes the results. Again, reasonable agreement was found between the two values. The trend found in the table is expected. As discussed above, moving from bottom to top and from left to right in the table will result in a decrease in the weight % aluminum of the overlay due to mixing and dilution produced by these processing conditions.

Table II. Estimated weight percentages of aluminum according to the cross-sectional areas. The bold numbers use A_0 calculated from equation (2) and the others use the measured A_0 .

Feed Rates (mm/sec)	Measured areas for 250A	Measured areas for 275A	Measured areas for 300A
15	8.7 10.7	7.1 9.7	5.4 6.9
35	24.2 24.8	18.6 18.2	17.4 17.9
55	32.5 29.8	36.4 33.6	34.6 33.3

Through use of EPMA, the nominal compositions of the weld deposits can be determined. Figure 6 shows the wt% of Al in the bulk of the overlay as a function of the welding parameters. As expected, the aluminum content increases as the wire feed rate increases and the current decreases. It can be seen that samples with less than 10wt% Al did not crack, while those just above had very few cracks, refer back to Figure 1. Above this value, cracks were prevalent as expected. In addition, the change in microstructure observed can be related to aluminum composition in the weld overlay. This transition occurs between 24 and 32 wt% Al.

Figure 6: Weight percent aluminum in the weld overlay as a function of the processing parameters. Deposits produced around 10wt% aluminum had few to no cracks.

A typical microprobe trace across a weld with a homogeneous matrix is seen in Figure 7a. Both the aluminum and iron contents are relatively homogenous through out the bulk of the weld until the partially mixed zone (PMZ) is encountered. This signifies good mixing between the iron and aluminum during processing and accounts for the low standard of deviation in figure 6. Through the PMZ, the aluminum content sharply decreases until 0% is reached in the substrate. A probe trace through a two phase weld is observed in Figure 7b. The shorter depth of the homogeneous portion and wide region of the PMZ indicates poor mixing during deposition. The PMZ of the weld is seen to have a gradual decrease in aluminum content into the substrate. EPMA data indicated that the dark single phase and dendrites, from Figure 4b, have a composition of approximately 45 and 33 wt% aluminum, respectively. The solidification process of these microstructures is presently under investigation.

a)

b)

Figure 7: Typical probe trace across a) homogeneous matrix weld overlay showing a sharp decrease of aluminum weight percent in the partially mixed zone (PMZ), b) multiphase weld overlay showing a gradual decrease in aluminum content into the substrate.

5.0 CONCLUSIONS

The goal of this current phase of research was to produce Fe-Al alloy weld overlays, with varying wt% of Al, by depositing commercially pure aluminum wire on to low carbon steel substrates using the GTAW process. These alloys were produced by varying the processing parameters of wire feed speed and current. Under these processing conditions, the following conclusions can be drawn:

- 1) A correlation was found between the processing parameters and aluminum content in the weld overlay. The wt% of Al in the deposit was observed to increase with wire feed speed and a decrease in the current.
- 2) The cracking susceptibility of the overlays was found to be a function of the aluminum content. At 10wt% aluminum, few to no cracks were observed in the deposits. Above this value, cracking was prevalent throughout the weld.
- 3) The microstructure was related to the aluminum content of the deposits. A homogeneous matrix with second phase particles consisting of coarse columnar grains was found for low aluminum concentrations. With higher aluminum contents, a two-phase constituent was seen to surround primary dendrites growing from the substrate. The transition of the microstructures occurs between 24 and 32 wt% Al.

6.0 FUTURE WORK

Future investigation will revolve around extensive laboratory evaluation of the single bead Fe-Al weld overlays deposited, as well as multiple pass overlays to be produced. The required alloy composition for low NO_x applications will be determined by exposing overlays with varying aluminum concentrations to simulated low NO_x boiler environments using a thermogravimetric balance. Other properties to be studied include thermal cycling and erosion resistance. These results will then be evaluated versus other coating systems presently being considered. In addition, further studies to develop an understanding of the solidification mechanism found in the high aluminum wt% weld overlays will be conducted.

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