Surface Modification of 4340 Steel with Iron Aluminides Using High-Energy-Density Processes

G. Muralidharan¹, C. A. Blue¹, V. K. Sikka¹, and N. B. Dahotre¹,²

¹Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN-37831-6083
²Department of Materials Science and Engineering, University of Tennessee, Knoxville, TN 37932

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Abstract

Iron aluminides possess several attractive properties including excellent resistance to oxidation and sulfidation in aggressive environments, and hence appropriate compositions of iron aluminides could find applications as high-temperature coatings. Iron aluminides can also be reinforced with hard, wear-resistant TiB₂ particles thus achieving a combination of superior oxidation/sulfidation properties and excellent wear-resistance. In this paper, results of recent work involving the use of a high-powered laser and a high-density infrared plasma arc lamp to coat a representative steel, 4340, with iron aluminides, will be presented. It was observed that iron aluminide could be retained on the surface when either processing technique was used but its retention was a strong function of the processing parameters. In this paper, particular emphasis will be placed on the relationship between the structure and composition of the coatings, their properties, and processing parameters.

Introduction

Iron aluminides possess several attractive properties thus resulting in significant research and development efforts over the past years [1]. Fe₃Al and FeAl show excellent resistance to oxidation and sulfidation in aggressive environments [1-6]. Due to their excellent properties and low cost, appropriate compositions of iron aluminides could find applications as coatings for more traditional high-strength materials which suffer from inferior high temperature corrosion-resistant properties [1-6]. Previous studies on the corrosion resistance of weld overlays of iron aluminides on stainless steels have shown that weld overlays with at least 25-30 at. % Al provide adequate corrosion resistance in oxidizing/sulfidizing atmospheres [5]. In other experiments, bulk iron aluminides have demonstrated superior wetting characteristics with hard particles such as TiB₂ [7]. Thus, iron aluminide coatings reinforced with hard, wear-resistant TiB₂ particles could potentially possess the desired combination of superior oxidation/sulfidation properties and excellent wear-resistance.

The purpose of this study was to explore the use of two high-powered heat sources, a Nd-YAG laser and a plasma arc lamp, to synthesize iron aluminide coatings on 4340 steel. Substrates made of 4340 steel were initially coated with iron aluminide-based precursor coatings. Subsequently, these coatings were fused with the substrate using high-energy-density heating processes. Preliminary experimental work along with earlier work on weld overlays [3-7], showed that the aluminum content in the final coatings would be less than that in the precursor iron aluminide coatings due to loss of aluminum through evaporation, and dilution that occurred during processing. Hence, in addition to iron aluminide powders, aluminum was added to the precursor coatings. This paper will outline the effect of the process parameters used to fuse the coatings to the substrate, on the microstructure and properties of the coating and the substrate.

Experimental Method

Argon gas-atomized FeAl alloy powder (Fe-41.7Al-0.18Mo-0.04Zr-1.1C-0.02B, in at.%), -100 mesh particle size, was mixed with pure Al powder resulting in an overall compositions of 75 mole % of Al in the FeAl-Al mixture. This mixture was subsequently blended with hydroxypropyl methyl cellulose, a water-based organic solvent, and sprayed as a precursor coating onto coupons of 4340 steel (Nominal composition: Fe-1.8C-0.74Cr-0.7Mn-0.11Mo-1.7Ni-0.39Si, in at.%) using a manual spray gun. An industrial grade 2.5kW Hobart HLP 3000 continuous wave Nd:YAG laser (1.06 µm wavelength) was employed for the synthesis of FeAl coatings and their diffusion bonding to the substrates. Laser optics was configured to provide a 3.5-mm-wide beam on the sample surface. Such configuration provides a large sweeping coverage, and reduces overlap between successive laser passes [8, 9]. In this series of experiments, three different laser power settings of 1250 W, 1500 W, and 1750 W were used, while the travel speed was kept constant.
The plasma arc lamp at ORNL utilizes a unique technology to produce high-energy-density infrared radiation with the peak in the spectrum occurring over the wavelength region of 0.2 – 1.40 \( \mu m \). Instead of using an electrically heated resistive element, a controlled, contained plasma is used to provide the radiant energy. Power densities of up to 3.5 kW/cm\(^2\) can be achieved using the lamp. Processing is performed with the sample placed in an environmentally controlled box with an IR-transparent quartz cover [10]. In this work, the plasma arc lamp was used to synthesize FeAl coatings. Results from two sets of processing conditions are presented in this paper with each of these sets consisting of two sequential scans on the sample. The processing conditions used were: 1) First scan at a power of 2025 W/cm\(^2\) and a scan speed of 8 mm/sec, followed by an immediate second scan at 2350 W/cm\(^2\) and a speed of 8 mm/sec.; 2) First scan at 2025 W/cm\(^2\) and a scan speed of 6 mm/sec and the second at 2350 W/cm\(^2\) and a speed of 8 mm/sec.

Cross-sectional optical microscopy was carried out on the processed samples after etching with a 2 vol. % Nital solution. Electron Probe Micro Analysis (EPMA) was carried out on the cross-sectioned samples to understand the variation in composition as a function of position within the coating and in the substrate. These observations were supplemented with microhardness tests (Knoop hardness, 100 g normal load) with the indents placed at equal intervals of 50 \( \mu m \). X-ray diffraction measurements were used to identify phases present in the samples.

**Results and Discussion**

**Samples Processed using the Nd-YAG Laser**

Figures 1 – 3 show typical cross-sectional optical microscopy images of the samples coated using the laser, operating at power settings of 1250W, 1500W and 1750W, respectively. As can be seen from the figures, the integrity of the coating layer in each sample is excellent and the interface between this layer and the heat-affected zone in the substrate is free of cracks and voids. Note that there exists a near-surface band in each sample, which appears bright when etched with the 2 vol. % Nital solution. The size of this band increases with increasing power as is observable from the figures.

Figures 4-6 show the Fe and Al concentrations as a function of position as obtained from EPMA for the samples processed using the three different power levels. All three profiles clearly show an Al-rich region at the surface. Comparing the extent of this region with that of the observed band shown in figures 1-3, leads to the inference that the bright-etched layer corresponds to the region in which there is enrichment of Al. Figure 4 shows that this region is approximately 100 \( \mu m \) thick in the sample processed at the lowest power of 1250 W, while it increases to about 275 \( \mu m \) in the sample processed with the highest power. Figures 4-6 also show that the increased power levels resulted in a decrease in the maximum Al contents observed at the surface. Note that the maximum Al content at the surface in the sample processed at the lowest power is about 31 at. % while it decreases to about 15 at. % in the sample processed at the highest power.

Figures 7-9 show the X-ray diffraction patterns obtained from samples processed using power levels of 1250W, 1500W, and 1750W, respectively. As observed from the figures, all samples show the presence of aluminum oxide on the surface. The presence of the oxide can be explained by the fact that the laser processing was conducted in air. In addition, FeAl peaks are present only in the sample processed at the lowest power level. Electron microscopy work is in progress to identify if other intermetallic phases are present in this sample. In the samples processed at the higher power levels, X-ray diffraction shows the presence of only a B.C.C. Fe-rich solid solution with a lattice parameter larger than that of pure Fe. Another important trend observed in figures 7-9 is that with increasing power levels, phases typically observed in 4340 (austenite, and martensite) start to appear in the near surface region, indicating that the effect of the addition of aluminum decreases with increasing dilution.

Some of the above observations can be explained on the basis of the Fe-Al phase diagram. It is known that the Fe-rich solid solution can accommodate up to 45 at.% Al, the FeAl phase is stable over a composition range of 23.3 to 55 at.% Al, and the Fe\(_3\)Al phase is stable over the composition range of ~23 to ~34 at.% Al [11]. Figure 10 also shows that the solubility of Al in Fe is approximately 21 at.% at 400°C. In this work, intermetallic compounds were observed only in the samples in which the aluminum concentration was 31 at. % at the surface, while solid solutions were observed in the other two samples in which the maximum aluminum concentrations were less than 21 at.%, thus consistent with the known solubility of Al in Fe.
Figure 1: A cross-sectional optical micrograph of a 4340 steel substrate with Fe-Al alloy coatings processed using the laser operating at a power of 1250 W.

Figure 2: A cross-sectional optical micrograph of a 4340 steel substrate with Fe-Al alloy coatings processed using the laser operating at power of 1500 W.

Figure 3: A cross-sectional optical micrograph of a 4340 steel substrate with Fe-Al alloy coatings processed using the laser operating at a power of 1750 W.
Figure 4: Variation in Fe and Al concentration within the near surface region obtained using EPMA for a sample processed using a power of 1250 W.

Figure 5: Variation in Fe and Al concentration within the near surface region obtained using EPMA for a sample processed using a power of 1500 W.

Figure 6: Variation in Fe and Al concentration within the near surface region obtained using EPMA for a sample processed using a power of 1750 W.
Figure 7: An X-ray diffraction pattern from the sample processed with the laser using a power of 1250 W.

Figure 8: An X-ray diffraction pattern from the sample processed with the laser using a power of 1500 W.

Figure 9: An X-ray diffraction pattern from the sample processed with the laser using a power of 1750 W.
Samples Processed using the Plasma Arc Lamp

Figure 11 shows a cross-sectional optical micrograph of the sample processed using condition 1, while figure 12 shows the corresponding X-ray diffraction pattern. Analysis of the diffraction pattern shows the presence of FeAl. Further work is necessary to determine the presence/absence of other intermetallic compounds. As in the laser-processed samples, an oxide layer is observed on the surface. Figure 13 shows a cross-sectional optical micrograph of the sample processed using condition 2, while figure 14 shows the corresponding diffraction pattern. Peaks that would indicate the presence of intermetallic compounds were not observed in the diffraction pattern. It should be noted that the only difference between the two sets of parameters is that in the second set of processing parameters, the scan at the lamp power of 2025 W/cm² occurred at a speed of 6 mm/sec instead of the speed of 8 mm/sec used in set 1. The slower scan speed resulted in a larger heat input to the sample, thus causing increased dilution and the disappearance of the intermetallic compound. Observations on the effect of heat input in laser processing showed a similar effect of power levels, with the higher power levels resulting in greater dilution and in the loss of intermetallic compounds at the surface.
Figure 11: A cross-sectional optical micrograph of a 4340 steel substrate with Fe-Al alloy coatings processed using the plasma arc lamp. Two sequential scans were used, the first at a power of 2025 W/cm² and a speed of 8 mm/sec and the second at 2350 W/cm² and a speed of 8 mm/sec (Processing condition 1).

Figure 12: An X-ray diffraction pattern from the sample processed with the plasma arc lamp using conditions shown in figure 11 (Processing condition 1).
Microhardness testing

Figures 15 and 16 show the variation in Knoop hardness values as a function of position within the coating, the heat-affected zone, and the substrate for samples processed using the laser and the plasma arc lamp, respectively. Note that in the case of the laser-processed samples, the hardness values within the coating are much larger (Knoop hardness values >400) than that obtained for the base material (Knoop hardness values < 300) for all the process conditions used in the study. In the case of the samples processed with the plasma arc lamp, the hardnesses vary with the processing conditions. The sample processed using parameter set 1 reveals hardness values within the coatings layer that are higher than those processed using set 1. Intermetallic compounds present in the sample processed using set 1 may be responsible for the higher hardness values. A significant feature in both samples processed using the plasma arc lamp is that a region with high hardness occurs at a distance of about 100 µm to 300 µm from the surface of the sample. Presence of a martensitic region, as observed in previous studies of laser processing of 4340 substrates [12-14], results in high hardness values. It should be noted that hardness values were measured at intervals of 50 µm only. Any variation over length scales smaller than this would not be observed in figures 15 and 16. Further work is on-going to study variations over smaller length scales.
Conclusions

From this study, it can be concluded that

1. Both laser processing and plasma arc high density IR lamp can be used to coat Fe-Al alloys on 4340 steel.
2. Process conditions and composition of precursor coating have to be carefully selected to retain iron aluminides on the surfaces of the samples.
3. Under appropriate processing conditions, coatings free of porosity and cracks can be obtained using both techniques.
4. Hardnesses of the coatings and the heat-affected zones are a strong function of the processing parameters and hence can be tailor-made for the application.
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References


