

MICROSTRUCTURE AND PROPERTIES OF HVOF-SPRAYED PROTECTIVE COATINGS

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ABSTRACT

Coatings of iron based aluminides have been deposited on steel substrates using the high velocity oxygen-fuel method (HVOF). The coatings are highly dense, contain relatively low fraction of oxides and have compressive residual stress in the as-deposited condition. While the microstructures are stable during corrosion testing in either oxidizing or simulated combustion gas environments the state of stress in the coating and the bonding to the substrate can change significantly as a result of corrosion or thermal cycling and this potentially leads to coating failure. Mechanical properties of free standing HVOF processed specimens have been characterized and compared to material that remained attached to substrates to separately determine the influence of microstructure evolution and constraint due to residual stress arising from the coating-substrate interaction on the cracking of coatings. A series of thermal cycling tests is being developed and the influence on corrosion resistance and mechanical properties will be determined.

INTRODUCTION

Alloys based on intermetallic compositions are being developed for advanced fossil fired power plants due to their potential to operate at higher temperature and resistance to environmental effects. Properties of these materials have shown considerable improvement after nearly two decades of development, however, they continue to face a number of challenges with respect to ductility and high temperature strength.^[1-4] One approach to take advantage of the environmental resistance of these materials while minimizing their limitations is to create coatings on more conventional structural materials.

Coatings can be applied by a variety of methods, including weld-overlay, reaction synthesis, physical vapor deposition and thermal spray processes.^[5-14] Thermal spray processing has the advantage of being able to deposit materials with widely varying melting points from power precursors. It is only recently, however, that processes like plasma spraying and high-velocity oxygen fuel (HVOF) thermal spray deposition have begun to move from an Edisonian approach to more science based process design and control. It is becoming increasingly common to control the properties of metallic and intermetallic coatings through measurement and control of the properties (size, temperature and velocity) of droplets in-flight from the spray torch during coating deposition. Through systematic control of the particle velocity and temperature it is possible to largely control the state of residual stress in the coating and the density and macrostructure.^[15]

The environmental resistance of coatings can be different from bulk materials due to chemical alteration of the powder during coating deposition giving rise to areas of alloy depletion and oxide inclusions,

porosity, and micro-cracking of the coating. In addition to environmental effects, the performance of the coating can also be influenced by residual stress, the nature of the bond between the coating and substrate, and the potential for poor bonding between layers in thick coatings built up through multiple spray passes. Stresses between the coating and substrate can be the result of thermal expansion mismatch between the coating and substrate during the high temperature excursion associated with coating deposition, or as a result of repeated thermal cycling representative of service conditions.^[15]

Laboratory testing of coating durability is problematic because of the difficulty with replicating service conditions. In addition, durability is not an intrinsic property of the coating or substrate that can be unambiguously measured. Coating durability can be separated in a general way into components relating to bonding to the substrate and cracking of the coating. The bonding is typically measured in the as-deposited condition or after simulated service conditions using a simple pull test as described in ASTM C 633. The resistance of the interface to cracking can be determined using modified fracture mechanics methods, however, this type of testing has not been widely applied due to difficulty in performing the tests and the fact that results are difficult to apply in design.^[16]

Resistance to cracking that might arise due to tensile loading is relatively straightforward to measure by loading coating-substrate couples until cracking is noted. The load at which cracking occurs can be determined using acoustic emission or visual means during testing or by post-mortem metallographic analysis. The fracture properties measured in this type of test are for the coating-substrate couple and not necessarily intrinsic to the coating material itself. Although this type of laboratory testing can be of value for scaling relative coating performance, determining cracking behavior due to service conditions is much more difficult since it can involve creep, cyclic behavior, or environmentally assisted failure mechanisms.

This paper reports on development of performance tests for HVOF sprayed intermetallic coatings. Testing focused on iron aluminide compositions sprayed onto substrates with differing thermal expansion coefficients. Thermal spray parameters were varied to alter the residual stress in the as-deposited coatings and the influences of test geometry and thickness of the coating on the propensity toward cracking were characterized.

EXPERIMENTAL METHODS

As reported previously, Fe₃Al coatings for coating fracture strain testing were prepared on stainless steel substrates using the HVOF process with kerosene and oxygen as the combustion gasses.^[17,18] Two different particle velocities, 560 and 620 m/s were used and coating thickness was varied from 50 to 410 μm. The microstructure of as-deposited coatings was characterized using metallographic examination of polished and etched cross-sections of the coating.

The room temperature cracking behavior of coatings was measured by tensile testing coated substrates. The substrates were rectangular dog-bone type specimens with coating material applied to the reduced section. Substrate strain to failure was previously reported for 500 μm specimens of Fe₃Al on several different substrate materials. Cracking of the coating was monitored by two acoustic emission sensors attached to the substrate in the grip section at both ends of the specimen. The experimental set-up is shown schematically in Figure 1 and Figure 2.

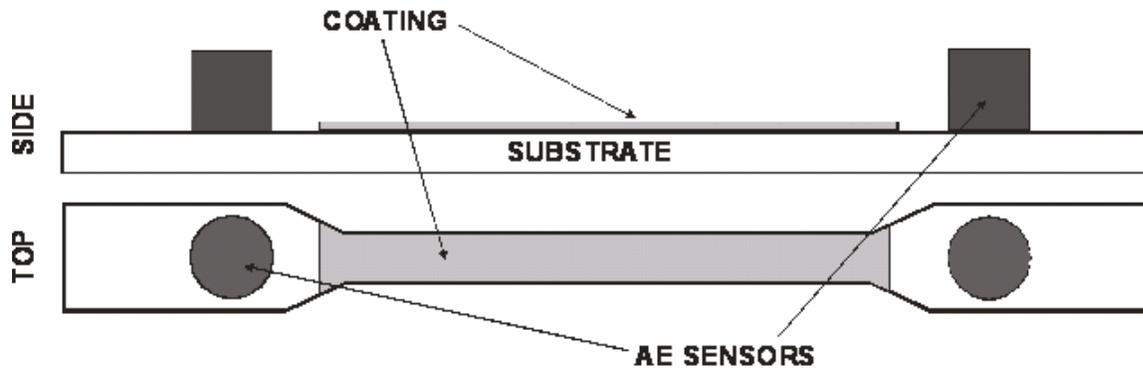


Figure 1. Schematic illustration of the test configuration for determining coating cracking strain.

The acoustic emission sensors were used to indicate a major cracking event in the sample. Visual examination was used during tests and in post-mortem examination. For the thickest coatings cracking was always clearly indicated by high-energy acoustic events. For less-thick coatings visual examination indicated a high density of microcracks or minor spallation of the coating could occur without clear high-energy acoustic events being recorded.

RESULTS AND DISCUSSION

The microstructure of coatings deposited with particle velocities of 570 and 630 m/s are shown in Figure 3a and b, respectively. In both cases the coating is fully dense and appears to be well bonded to the substrate. Some fraction of particles is unmelted in both coatings as indicated by hemispherical features that are flat on the impact side of the particle (i.e., the side of the particle that is oriented toward the substrate). The coatings illustrated in figure 3 were deposited under nominally identical conditions except for the particle velocity, the coating formed using the higher velocity particles was considerably less thick. It has been shown previously that higher velocity particles tend to fragment to a greater degree upon impact with the substrate and a significant volume of the fragmented material rebounds from the substrate and is not incorporated into the coating. This results in reduced deposition efficiency; the ratio of material that is incorporated into the coating compared to the amount that is feed through the torch is reduced with higher particle velocity. Thus, to form coating with equivalent total thickness a larger number of coating passes was necessary with higher velocity particles.

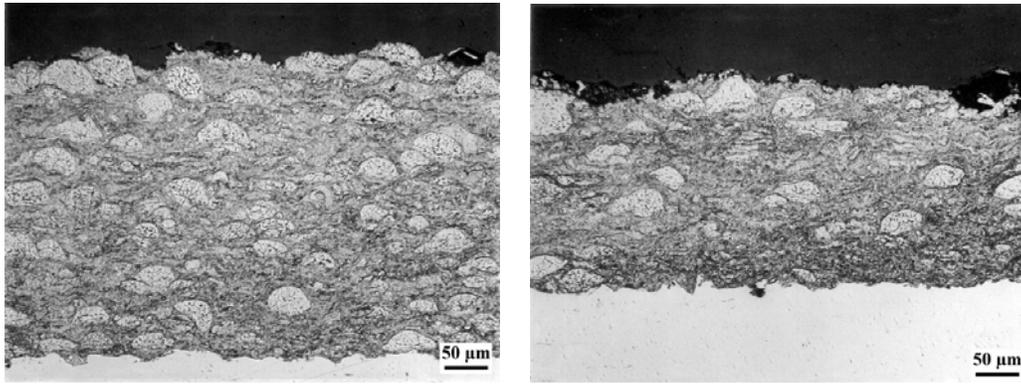
Photographs of the coating surface after tensile loading to a strain resulting in coating failure are shown in Figure 4 for Fe_3Al coatings with three different thicknesses, 50, 130 and 410 μm . The 410 μm thick coatings displayed single large cracks at failure, similar to those reported previously for coatings 500 μm in thickness. Very thin coatings, 50 μm in thickness, exhibited no significant cracking. It can be noted in Figure 4, however, that there are patches along the edge that are much brighter than the surrounding coating material. These bright features are regions where small amounts of coating have spalled from the coating with fracture parallel to the coating-substrate interface.



Figure 2. Photograph of a strain to failure test with the acoustic emission apparatus and clip gage attached to measure attached. This 500 μm thick Fe_3Al coating failed with a single major through-thickness crack.

Through-thickness cracking is life limiting since it will allow the corrosive environment access to the coating-substrate interface. For protective coatings that are prone to cracking during thermal cycling as a result of accumulated strain due to thermal expansion mismatch this observation suggests that thin coatings will provide greater service life. For general corrosion, however, it is evident that a thick coating will provide a greater corrosion allowance during service. It is well known that the HVOF coating process results in coatings that have significant compressive residual stress in the as-deposited condition. This compressive stress is a result of peening from high velocity particle impact during deposition. While the compressive deposition stresses might be expected to mitigate service induced stresses from thermal cycling, the Fe_3Al coatings have sufficiently low yield strength at expected service conditions that the initial stress is relieved during the first thermal cycle above about 700°C.

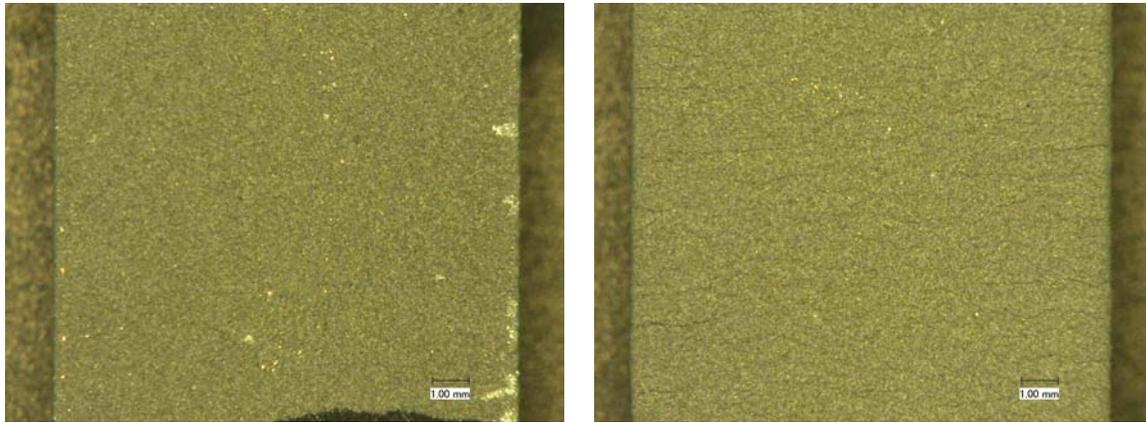
It is evident from Figure 4 that the spallation in thin coatings and microcracking in moderately thick coatings are initiated at the edge of the dog-bone tensile specimens. The influence of edge effects on laboratory measurement of strain to failure is difficult to quantify. It seems likely that strain to failure measured with this type of specimen is reduced compared to expected service conditions on large sections or tubular geometry. A laboratory test is being developed to eliminate the influence of edge effects and to allow rapid thermal cycling. Round bar substrate specimens have been obtained and Fe_3Al coatings will be deposited on the cylindrical reduced gage section for testing. Thermal cycling will be accomplished with induction heating with the specimen clamped in a heat sink so that the thermal cycle can be varied from times as short as a few hundred seconds. This test configuration is shown schematically in Figure 5. Based on the tests to date, visual inspection will be used to determine the strain to failure in these round bar tests as it appears to be the preferred method to identify microcracks.



(a)

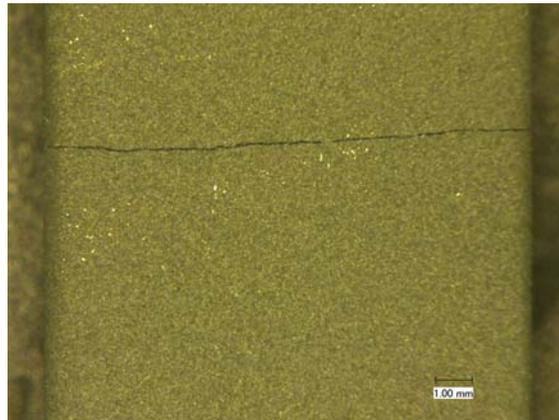
(b)

Figure 3. Microstructure of Fe_3Al coatings produced using HVOF with particle velocity of (a) 570 and (b) 630 m/s.



(a)

(b)



(c)

Figure 4. Macrographs of the surface of coatings strained to failure in tension; (a) 50 μm , (b) 130 μm and (c) 410 μm in thickness.

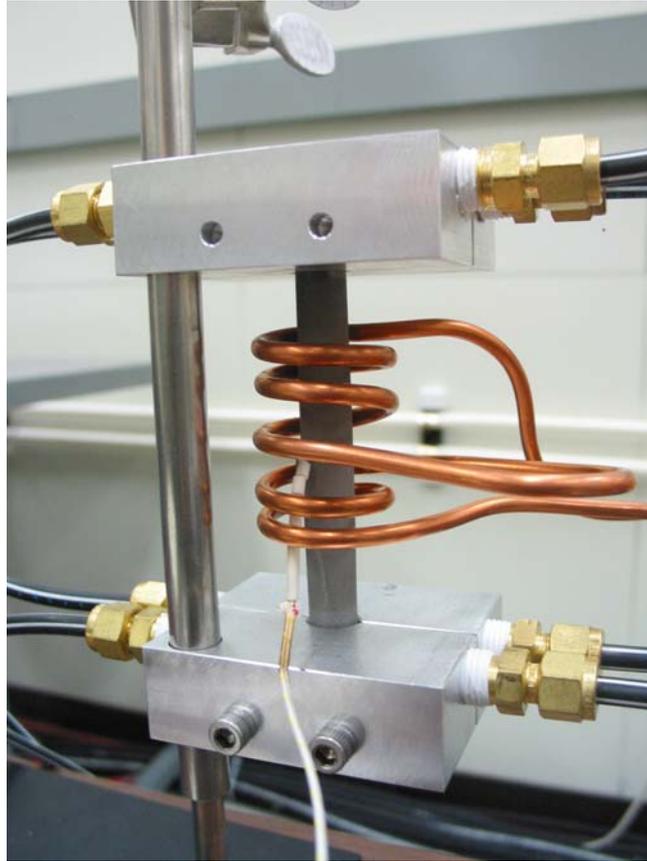


Figure 5. Induction heated round bar geometry for performance testing of coating cracking in simulated service without the influence of edge cracking.

CONCLUSIONS

Fully dense and adherent Fe_3Al coatings have been produced using the HVOF process. Methods to characterize the performance of coatings under expected service conditions are being developed. Tensile testing of coatings on flat dog-bone type specimens has shown that $50\ \mu\text{m}$ thick coatings show a small amount of coating spallation, while $410\ \mu\text{m}$ coatings fail as a result of one major through-thickness crack. Coatings with intermediate thickness show a high density of microcracks. While this type of test may be suitable for screening coating behavior, cracks are initiated at the edge of the specimen. Behavior from the flat specimen geometry may not be a reasonable approximation of behavior expected under service conditions where large panels or tubular geometry will prevail. A round bar type of specimen capable of being rapidly thermally cycled using induction heating is being developed to address this issue.

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