

**Oxide Dispersion Strengthened Iron Aluminide by CVD
Coated Powders**

Final Report

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FOREWORD

This report was prepared by the Powdermet Inc, Euclid, Ohio for the DOE I&I proposal Category 2 entitled “Oxide Dispersion Strengthened Iron Aluminide by CVD coated Powders” Award Number DE-FG36-03GO13012. Fabrication and Evaluation of the Mechanical and Physical Properties of Iron Aluminide composites and its thermal sprayed coatings using chemical vapor deposition (CVD) aluminum, iron and aluminum oxide coated iron powders and iron, aluminum coated aluminum oxide powders were evaluated as part of a DOE I & I Category 2 Program following the successful completion of a preceding SBIR Phase-I assignment on the same subject. The objective of this program was to develop and optimize CVD aluminum, iron and aluminum oxide coated iron powders and iron, aluminum coated aluminum oxide powders for thermal spray application and optimize the fabrication of this Iron Aluminide composites, and it’s coatings and evaluate the performances of these new materials for potential use as high temperature, oxidation, corrosion and carburization resistant material aimed for future power generation and boiler equipment. The program developed CVD coated powders demonstrated the fabrication of oxide dispersion strengthened (ODS) Fe₃Al composites with a tensile value of 121 KSI and oxidation, carburization and corrosion resistant thermal sprayed coating The Prime Contract manager was Mr. Asit Biswas, of Powdermet, Inc., and the subject DOE I & I Program was monitored by HQ I&I Technology Manager: Lisa Barnett, Project Officer: Deborah Weems, Project Monitor: Tony Newville, and Financial /Officer: Michael Schledorn.

ABSTRACT

This I &I Category2 program developed chemical vapor deposition (CVD) of iron, aluminum and aluminum oxide coated iron powders and the availability of high temperature oxidation, corrosion and erosion resistant coating for future power generation equipment and can be used for retrofitting existing fossil-fired power plant equipment. This coating will provide enhanced life and performance of Coal-Fired Boilers components such as fire side corrosion on the outer diameter (OD) of the water wall and superheater tubing as well as on the inner diameter (ID) and OD of larger diameter headers. The program also developed a manufacturing route for readily available thermal spray powders for iron aluminide coating and fabrication of net shape component by powder metallurgy route using this CVD coated powders.

This coating can also be applied on jet engine compressor blade and housing, industrial heat treating furnace fixtures, magnetic electronic parts, heating element, piping and tubing for fossil energy application and automotive application, chemical processing equipment, heat exchanger, and structural member of aircraft. The program also resulted in developing a new fabrication route of thermal spray coating and oxide dispersion strengthened (ODS) iron aluminide composites enabling more precise control over material microstructures.

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1.0 PROGRAM OBJECTIVES

The objective of this program for Powdermet was to fabricate and evaluate key mechanical and physical properties of iron aluminide composite fabricated by cold isostatic preseed (CIP) /sinter, CIP/extrusion and thermal sprayed coating process with the objective of examining the potential for use of such material in the construction of certain power generation components, such as boiler tubes, thermal sprayed coating to enhance life and performance of Coal-Fired Boilers components such as fire side corrosion on the OD of the water wall and superheater tubing as well as on the ID and OD of larger diameter headers. In executing this task it should be recognized that with the Powdermet aluminide coating and its composites being a new material whose processing has partially optimized but not fully optimized, more than one processing iteration and pathway or method had to be evaluated, while keeping in mind downstream considerations such as scale-up, property balance, temperature tolerance, etc. This evaluation may therefore be considered as an initial evaluation aimed at guiding the path towards further optimization and technology maturation of innovative ODS iron aluminide composite, iron aluminide, alumina reinforced iron aluminide and its coating to unique applications for future power generation structures.

2.0: INTRODUCTION

The high efficiency power plant of the 21st century will consist of a pressurized fluidized bed (PFB), combined cycle (CC) power plant, or an integrated gasification combined cycle (IGCC). These PFB/CC and IGCC systems burn high sulfur coal in an environmentally acceptable way more efficiently than any other coal burning systems currently under development. However, operation and efficiencies of PFB and other combined cycle power generation systems are limited by one or more of the following factors:

Combustor and heat exchanger designs

Temperature limits on piping, as well as non-uniformity and “hot spots” created in the bed and tube bundles limit operating temperatures. This is particularly true of superheater and preheater bundles.

Low gas turbine inlet temperatures

PFB exit temperatures are limited to 1500-1700°F, largely due to gas cleanup, sulfur removal, and piping limitations. More advanced designs will use a topping cycle to increase gas turbine inlet temperatures to 2300°F

Non-optimum steam conditions

Increasing steam temperatures and pressures improved efficiency of the steam turbine cycle. Current systems operate at steam temperatures of 550°C (1032°F). Next generation systems will operate at 650-700°C (1202-1292°F), and design goals now call for 750-850 degree C (1382-

1562°F) steam temperatures. These increased temperatures will be generated in superheaters and reheaters in both low emission boilers, PFB's, and firetube boilers built into IGCC's. This will push power cycle efficiencies to over 50% in combined cycle plants.

These limitations are all related to availability of oxidation and corrosion resistant materials are capable of withstanding temperatures of 1300-1700°F (705-927°C) – well above the temperature limits of currently *affordable* materials. **Iron aluminides** offer a potential solution to materials challenges for these next generation (and retrofit) fossil fuel powered utility power islands. **Iron aluminides** have the following unique properties, making them ideal for use in 1100-1700°F temperature regimes:

- Low cost compared to cobalt- and nickel-based superalloys
- Excellent oxidation resistance
- Good hot corrosion/sulphidation resistance (intermediate Cr alloys)
- Excellent carburization resistance (low to intermediate Cr alloys)
- High yield and creep strengths at elevated temperatures
- Acceptable toughness and ductility at low temperatures

Drawbacks of current iron aluminides include the following:

- Poorer than desired material ductilities
- Limited availability, processing capabilities and field exposure experience
- Lower than desired creep resistance and high temperature strength
- Poor weldability

Lack of approved minimum design property values (ASTM Boiler Code)

IGCC conditions are typically consist of:

- Gasification temperature: 2300-2500°F
- Syngas exit temperature (Wabash): 1900°F
- Syngas cooler steam generator (Wabash): 1600psig

Hot gas cleanup gas temperature: 800-1100°F

Typical conditions encountered in PFBC and integrated gasification combined cycle (IGCC) systems are described as follows:

- The gas environment in a PFBC system is an oxidizing combustion stream, with an operating temperature in the range of 840-950°C (1544-1742°F)
- Operating pressures are typically 10-16atm.
- Ash particle loadings may range from 500 to 10,000ppm by weight
- The system contains volatile chloride, sulfur, and alkali species

Materials Needs

Based on projected operating conditions in future utility generating systems, the following components would tremendously benefit from advanced materials use:

- Carbonizer/synfuel cooler heat exchanger tubes (1500-1900°F; reducing/carburizing atmosphere containing H₂ and H₂S)
- Hot gas filters (1100-1500°F; can be oxidizing or reducing)
- Superheater and reheater tubes (1100-1500°F; oxidizing)
- Thermocouple sheaths, injectors, clamps, valves, supports, fasteners, ferrules, and other components

Two primary approaches exist to solving these materials problems:

- (1) Developing improved coatings that enable current materials such as nickel-based alloys and stainless steels to be used in these highly oxidizing and/or corrosive environments.
- (2) Developing new alloys with improved high temperature strength and corrosion resistance.

Iron aluminide materials based on Fe₃Al and FeAl compositions show potential for use in all of the above-mentioned components as both coatings and as new alloys (given improvements in material compositions/microstructures and processing/fabrication capabilities).

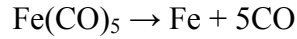
3.0: EXPERIMENTAL WORK, RESULTS AND DISCUSSIONS:

The initial target was to coat the iron powders or alumina particles with a thin layer of iron, followed by a thin layer of aluminum and aluminum oxide, such that these would produce an ODS iron aluminide alloy or alumina reinforced iron aluminide after the consolidation step. The scope was expanded to include two more alloy additions for this study — chromium and boron to increase room temperature ductility and grain boundary strength. But it was realized that chromium and boron coating by CVD process is not a cost effective process. As a result iron-chromium initial powder was chosen for coating of iron, aluminum and in case of iron, aluminum coated alumina powders, no chromium was blended before consolidation step or thermal spray deposition.

A *critical* technical repeatability and reproducibility issues were demonstrated applying uniform, nanoscale, CVD oxide coatings with aluminum, iron, having controlled morphology. To accomplish this, deposition studies were conducted on aluminum, iron, chromium and boron coating systems. Though chromium and boron were not deposited on the final powders due to cost issue, Powdermet did experimental/developmental run for chromium and boron deposition.

The iron coatings were deposited using standard carbonyl precursor materials. Iron

pentacarbonyl was used as the starting precursor. Iron deposition was performed in a small bed reactor, where the particles were fluidized and then CVD coated with the desired amount of iron via the following reaction:



Iron pentacarbonyl was directly fed to the reactor as liquid and vaporized at 200 °C

Iron deposition was subsequently followed by CVD deposition of aluminum onto the iron surface. Aluminum deposition was done using tri-ethylene aluminum (TEAL) decomposition at 460-475°C. The key difficulty with aluminum deposition from TEAL was obtaining high density nucleation, and TEAL was vaporized (175 °C) before entering the reactor. However, at the end of the program nucleation problems were solved within this short period of the program.

Tri-ethylene aluminum (TEAL/H₂O) were selected as suitable precursors for alumina, and deposition investigations were conducted. Deposition investigations included temperature, pressure and partial pressure effects on oxide deposition. Alumina deposition results indicated that alumina could be successfully deposited from TEAL.

Aluminum, iron, alumina encapsulation was conducted on iron-chromium alloy powders and aluminum, iron encapsulation were done on alumina particles using Powdermet, Inc.'s patented, R&D 100 award winning (2000) Recirculating Fast-fluidized Bed (RFFB), as shown in Figure 1. Then iron was deposited via chemical vapor deposition from iron carbonyl onto fluidized iron-chromium and alumina powders. The iron carbonyl was vaporized in the reactor and iron-chromium alloy powders were fluidized using argon/hydrogen gas flow. Fe-Cr was produced according to the following reaction:

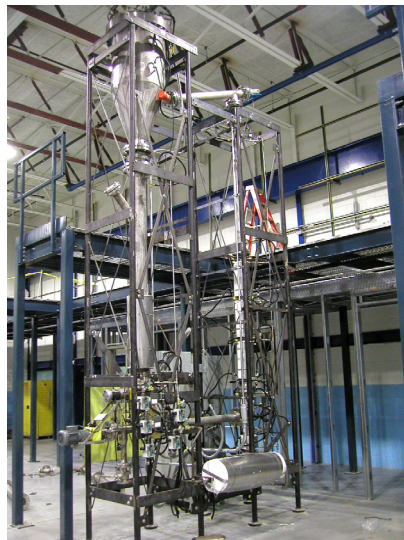
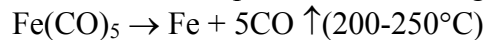
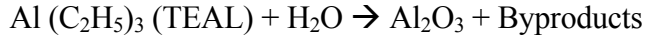


Figure 1. Powdermet's Fluidized Bed Reactor

Aluminum and aluminum oxide, was coated onto Fe-Cr alloy powders according to following reactions:



Similarly iron and aluminum was coated on fluidized alumina particles.

Powdermet uses metal salts, alkyls and carbonyls to produce coated powders. For metal carbonyls (Mond Process used in nickel refining), Powdermet has vertically integrated to manufacture its own precursors for cost-effective manufacture of coated powders. Figure 2 shows a carbonyl production unit installed at Powdermet's Euclid, Ohio manufacturing plant. Coating summary is presented in Table 1

Table 1. CVD Coating Run Summary

Precursor	Temp. Ranges	Theoretical Yield (%)	Actual Yield (%)	Comments	Pressure (in hg)
TEAL (for Al deposition)	250-300°C	23.7%	19.5-20%	Oxygen control	-25
TEAL/H ₂ O (for alumina)	200-350°C	25	22.0	H ₂ O/H ₂	-20 to -25
Iron Carbonyl (for iron)	175-200°C	28.6	25.0	Carbon/oxygen Control	-20 to -25
Diborane (for boron)	500-600°C	79	55.0	Boron concentration	-20 to -25
Chromium Carbonyl (for chromium)	350-500°C	23	20.0	Oxygen/carbon Control	-25



Figure 2. Powdermet's Carbonyl Production Unit

3.1 Powder Coating: 5-20 micron iron-chromium powders and 30 micron size alumina particles were coated with iron/aluminum/alumina and iron/aluminum. Figures 3-6 are a SEM photomicrograph of the powders.

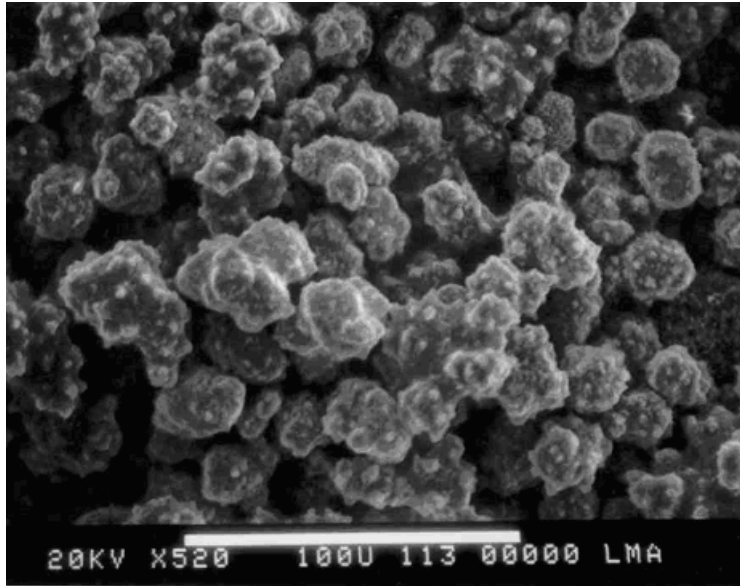


Figure 3: Aluminum and iron coated Iron Powders for thermal spray coating fabricated at Powdermet X520

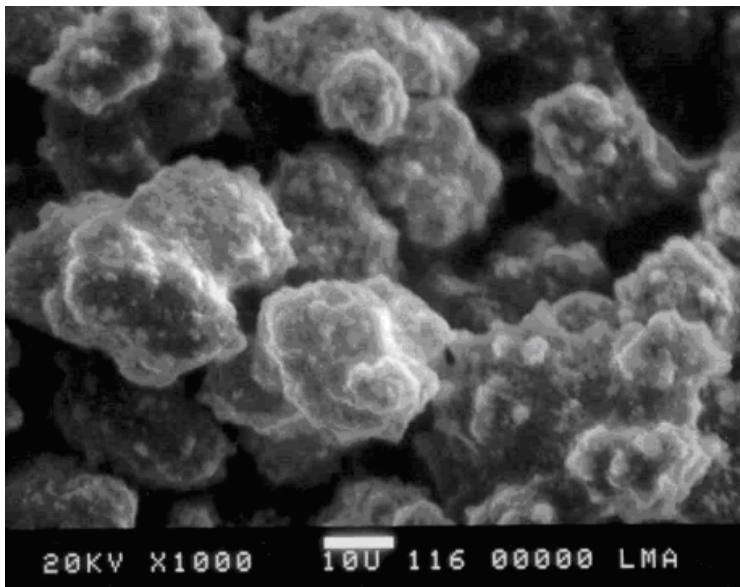


Figure 4: Aluminum and iron coated Iron Powders for thermal spray fabricated at Powdermet X1000

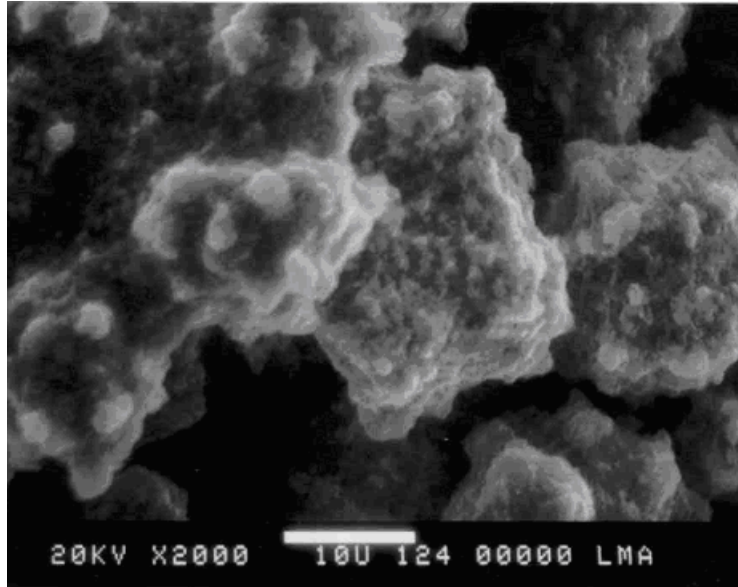


Figure 5: Aluminum and Iron coated Iron Powders for thermal spray fabricated at Powdermet X2000

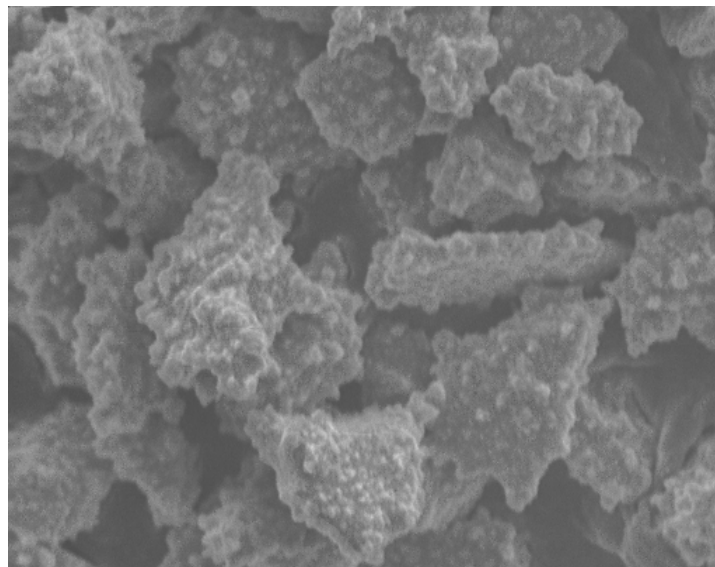


Figure 6: Fe, Al Coated Alumina Thermal Spray Powders fabricated at Powdermet

This is one of the biggest achievements of the program that Powdermet successfully fabricated CVD iron, aluminum, aluminum oxide coated iron and iron, aluminum coated alumina powders for fabricating composites and thermal spray coating for future power generation equipment.

3.2 Consolidation by CIP/Sinter Method

Mechanically pressed powders from small coupons were placed into a furnace and were

evacuated prior to heating. The samples were heated to 200°C and held for 1 hour to remove adsorbed moisture. The furnace ramped up to the sintering temperature (800-1300°C) for 1-4 hours. To develop the sintering process, coated powders were compacted into pellets and sintered in a vacuum at 1300°C for 1 hour. Inspecting the sintered microstructures revealed that sintered samples fabricated by aluminum coated iron iron powders showed better densification and microstructure than the sample fabricated by aluminum/aluminum oxide coated and iron, aluminum coated alumina powders. The 1300°C, 1 hour sintering samples sintered the best.

Powdermet demonstrated netshape fabrication of tube by powder metallurgy technique. and Figures 8 represent sintered microstructures of the alumina reinforced iron aluminide



Figure 7: Showing alumina reinforced iron aluminide tube fabricated by CIP/sinter process and net shape component by powdermetallurgy technique demonstrated using this CVD coated powder

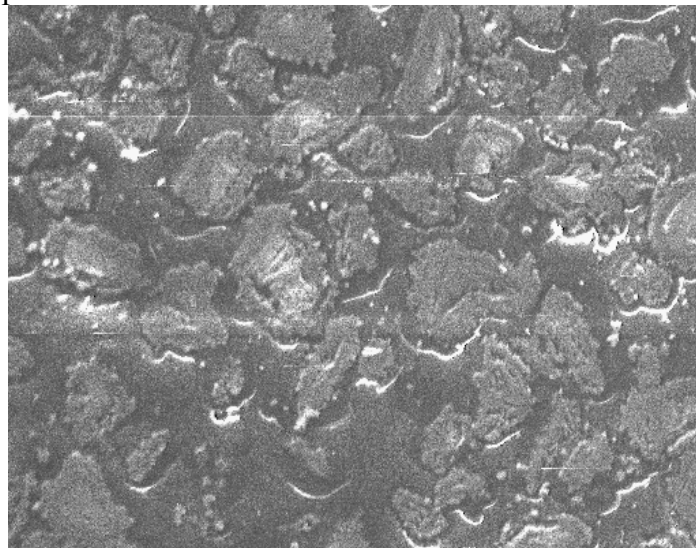


Figure 8: Sintered microstructure of alumina reinforced iron aluminide

3.3 Consolidation By Extrusion: Powders shown in figure 9 and 10 were coated with iron, aluminum and aluminum oxide on iron and then cold isostatically pressed and extruded. No rolling was done after extrusion. Extrusion of canned and evacuated cold isostatically pressed bars was done at 1000 °C at a 10:1 extrusion ratio. At first bars with Fe-16Al composition were cold isostatically pressed at a pressure of 60,000psi, canned in mild steel and evacuated. Extrusion conditions produced a fully dense product. Extruded bars were cut into pieces and the steel cans were removed from the extruded bars. Tensile testing specimens were machined from the can-free extruded ODS iron aluminide bars. Figure to Figure represent CVD aluminum coated iron powders.

Optical micrographs of transverse and longitudinal sections of extruded ODS iron aluminide bars are presented in Figures 11 and 12.

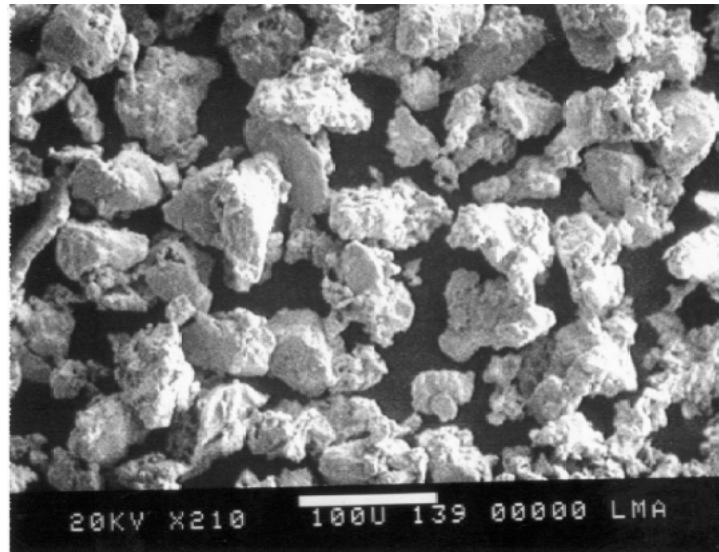


Figure 9: Aluminum, iron Coated Iron Powders X210

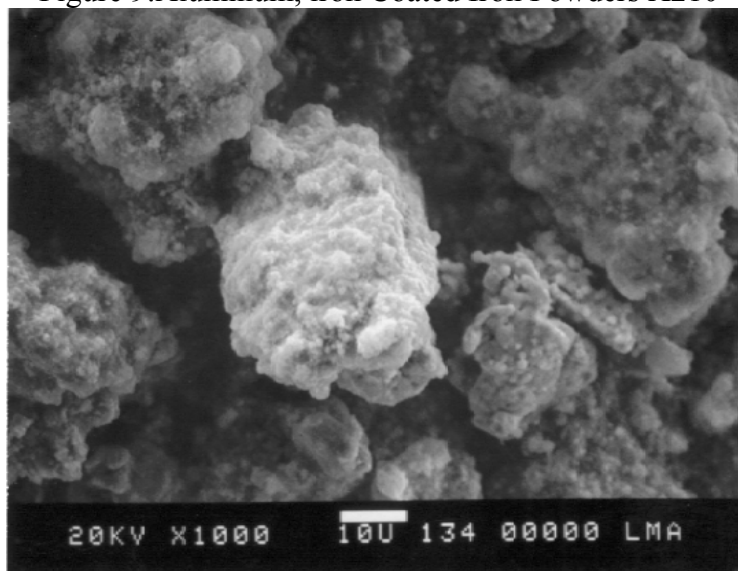


Figure 10: Aluminum, iron coated iron powders X1000

Figures 11 and 12 represent optical micrographs of transverse and longitudinal sections of extruded ODS iron aluminide bars.

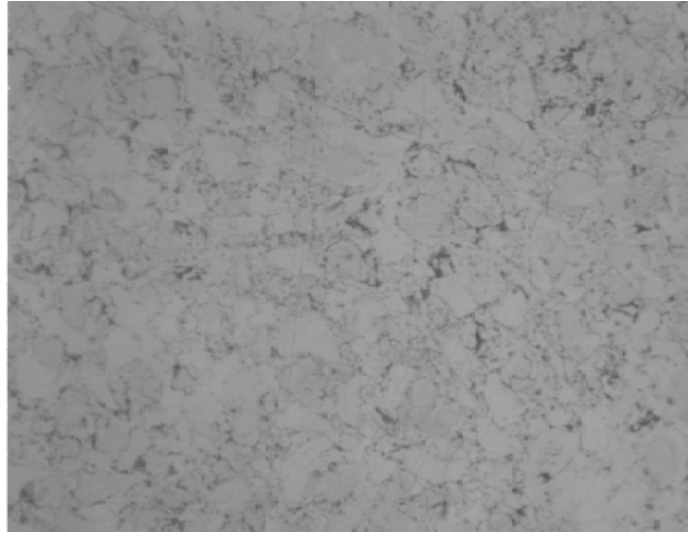


Figure 11. Optical Micrograph of Extruded (10:1) Iron Aluminide Transverse (200X)



Figure 12. Showing Optical Micrograph of Extruded (10:1) Iron Aluminide Longitudinal (200X)

3.4 Mechanical Property Testing

For ODS iron aluminide, tensile testing was conducted only at room temperature and only few tests were performed due to difficulty in machining samples. Samples were breaking frequently during fabricating tensile test samples. Samples were breaking in the threaded neck region of ODS iron aluminide during tensile test sample fabrication. As a

result, not many tensile samples were fabricated and tested. Same thing happened in a previous program.

Despite this problem, a few tensile tests were performed at room temperature for ODS iron aluminide. Table 2 represents extruded ODS iron aluminide mechanical test results.

Table 2. Mechanical Properties of Extruded Bars

Particle Size	Test Temperature	Yield Strength, KSI	Elongation (%)	ULT KSI
40 Micron	RT	105	4.1	118
	RT	104	3.7	121
	RT	-	Specimen fractured	101

The ultimate tensile strength (UTS) of the extruded ODS iron aluminide alloy was 101-121KSI. The UTS of MA 754 is 140KSI with yield strength of 85KSI. Yield strength and UTS of MA 758 are 81KSI and 138KSI, respectively. The MA 956 alloy possesses 75 KSI yield strength and 95KSI UTS. Comparing these mechanical property data, it can be concluded that mechanical properties obtained in this study of ODS iron aluminide using the CVD coated powder approach shows potential.

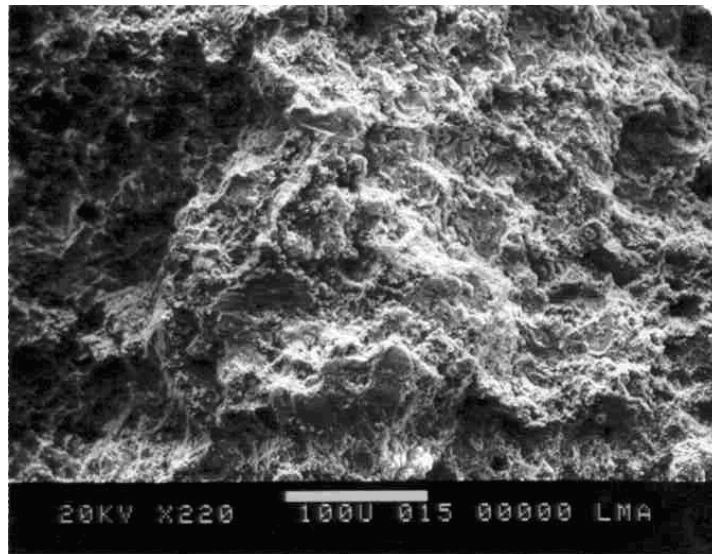


Figure 13. Fracture Surface of Extruded (10:1) Iron Aluminide showing mixed mode fracture comprised of brittle and ductile

But a limited attention was given to CIP/extrusion consolidation process before it was abandoned due to poor room temperature properties, breaking of samples while machining, needed for room temperature property modifications, further consolidation development and financial need. In a previous program almost same results were obtained. So with the permission of DOE I & I HQ Technology Manager, Project Officer

and that time Project Monitor, the CIP/extrusion or CIP/sinter process route had to be sacrificed (at the program outset) for the more critical processing-microstructure-property optimization effort and the more readily available thermal spray coating method using this powder for oxidation, carburization and sulfidation coating.

3.5 Thermal Spray Coating:

Thermal spray deposition

Thermal spraying is a well-established process for fabricating freestanding structures from a wide range of metals, alloys and ceramic materials. It is a complex process involving rapid melting and high velocity impact deposition of melted powder droplets usually referred as “splats.” Due to the rapid solidification or quenching of materials, the coating evolution process is also complex; usually leading to fine-grained and metastable microstructure of deposited materials. A unique feature of thermal spraying is that it combines melting, quenching, and consolidation in to a single operation and therefore retains the rapidly quenched structure of materials.

These splats form the “building blocks” of the coatings, and the conditions of these splats prior to impact on the substrate — namely, their temperature, velocity, size and also properties of the substrates such as temperature, chemistry and roughness —determine the droplets’ spreading or flatness and rate of solidification. Depending upon these factors, the resulting splats form different shapes, thickness and adhesion to the substrates with important consequences on the coating microstructure and on other physical and mechanical properties. It must be noted that thermal spray is a “line of sight” process where the arriving droplets from which the coating is formed deposit only on to the surfaces that are directly in line with the droplets direction. Due to the high plasma flame temperature (greater than 15,000°K), thermal spray processes have a relatively high rate of materials’ deposition in comparison to CVD and PVD coating processes. Thermal spray deposited coatings exhibit no mixing of substrate material to the substrate material, thus preserving base material composition.

The common major processing parameters in thermal spray methods are jet-velocity and temperature profiles, both derived from gas heating and flow parameters which determines whether enough energy or time is available to melt the powder particles. In combustion and plasma spray, a particle dwell time determines the degree of particle heating and is defined as the time a particles spends in the jet in a temperature zone, which is above the melting point of particle. Dwell time varies among the processes and even in a single jet as thermal spray jets have a distribution of gas velocities and temperatures. The second important parameter is the particles velocities which is dependent on the gas velocity distributions, with the resultant final particle velocity being determined by the jet/particle drag force. It is accepted that higher thermal spray jet velocities generally lead to higher “average” particle velocities. Particle velocities are important as it is accepted that higher particle velocity imparts higher impact energy, which leads to higher degree of splats deformation, better coating bonds and higher coating densities. On the other hand, higher particle velocities lead to shorter dwell times

which results in less time for particle heating and may lead to lower coating densities, and a higher percentage of un-melted or partially melted phases present in the coating materials.

Experimental Processing

In this investigation, the following parameters were investigated and optimized to understand the splat formation, microstructure development and quenching stress in the thermal sprayed coatings:

- 1) Gun power and gas flow rate.
- 2) Powder feed rates for different types of powders.
- 3) Gun standoff distance and relative speed between the gun and the coating surface.
- 4) Substrate heating/cooling before, during, and after spraying

The iron, aluminum coated powders were sprayed using Plasma Spraying in presence of hydrogen argon atmosphere and High Velocity Oxygen Fuel (HVOF) processes. In this program plasma spray process was used for the spraying of alumina reinforced iron aluminide layers coating for producing the high temperature, corrosion, wear, carburization, sulfidization and oxidation resistant coating in one design. For this evaluation, a Sulzer-Metco 9MB type spray torch was used with an external powder injection system employing a No. 6 powder port set-up. The primary plasma gas was argon, with hydrogen used to control plasma voltage.

The major difficulty encountered in spraying the micro-engineered powders was achieving powder flow during spraying. Table 3 represents plasma sprayed coating composition and thickness during initial development run.

Table 3. Layer Thicknesses of iron Aluminide Coating

Hardness (VHN)	Aim Thickness, mm	Actual Thickness, mm
420	6-8 mil	9 mil



Figure 14: Technical Approach of Thermal Spray Coating on 718 and 304 Substrate

Figure 14 represents the technical approach for thermal spray coating. Figure 15 to Figure 16 represent the scanning electron microscope (SEM) micrograph/ Energy Dispersive Spectroscopy (EDS) analysis of inconel 718 substrate before thermal spray coating and EDS analysis reveals that the substrate is comprised of Ni, Fe, Cr, Ti and Al.

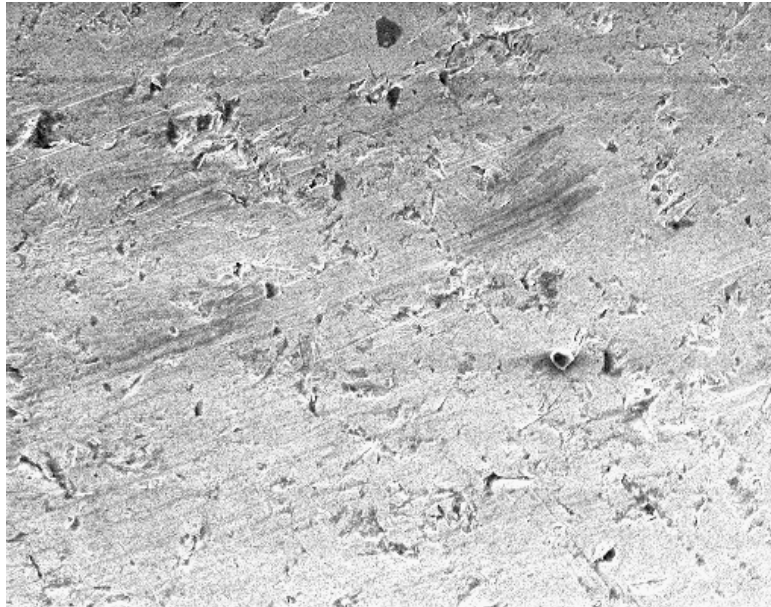


Figure 15: SEM Micrograph of Substrate Inconel 718 X160

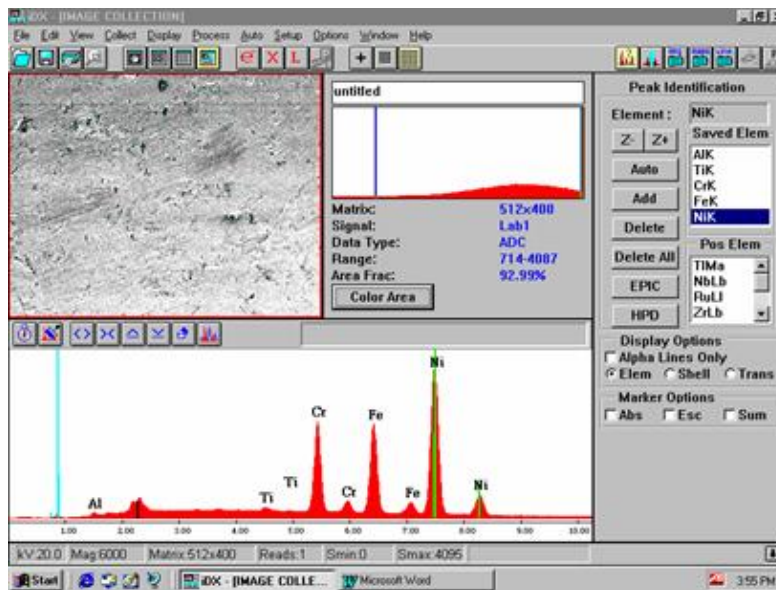


Figure 16: EDS analysis show Ni, Fe, Cr, Al in inconel 718

Figure 17 to Figure 18 represent the SEM micrograph, EDS analysis and X ray mapping of as sprayed (Plasma Spray) structure on inconel 718. EDS analysis and X ray mapping confirmed that the inconel substrate is nicely covered by iron aluminide sprayed structure as there was no nickel or chromium peaks found in the EDS analysis.

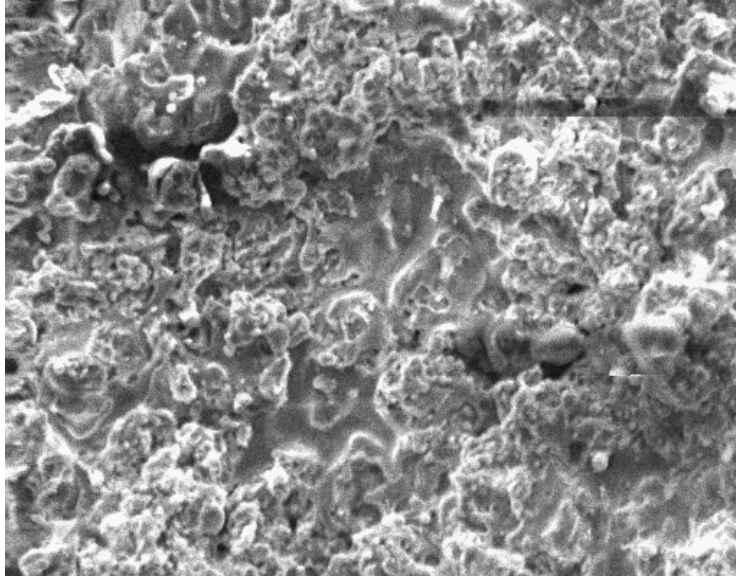


Figure 17 : SEM photomicrograph of as sprayed structure X430

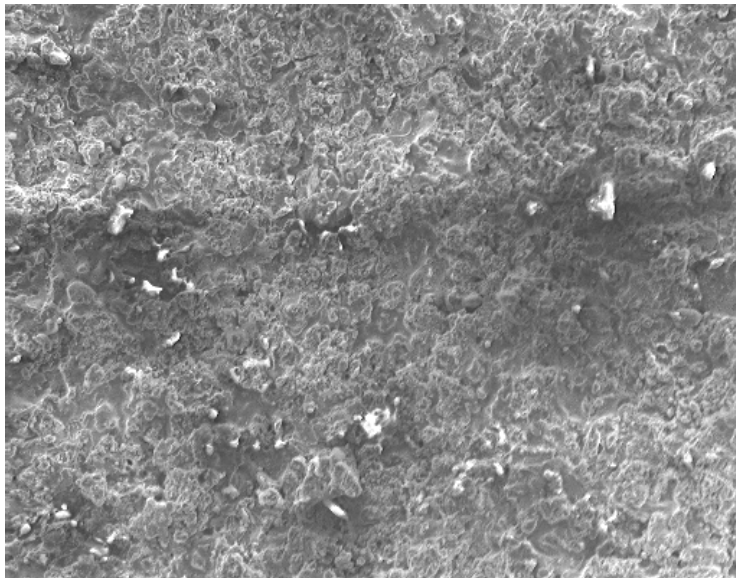


Figure 18: SEM micrograph of as sprayed structure X160

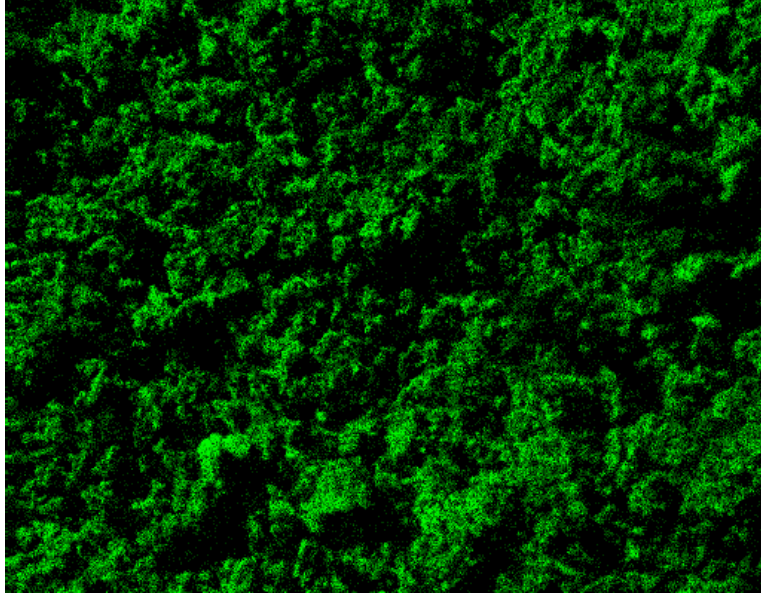


Figure 19: X ray mapping of Aluminum

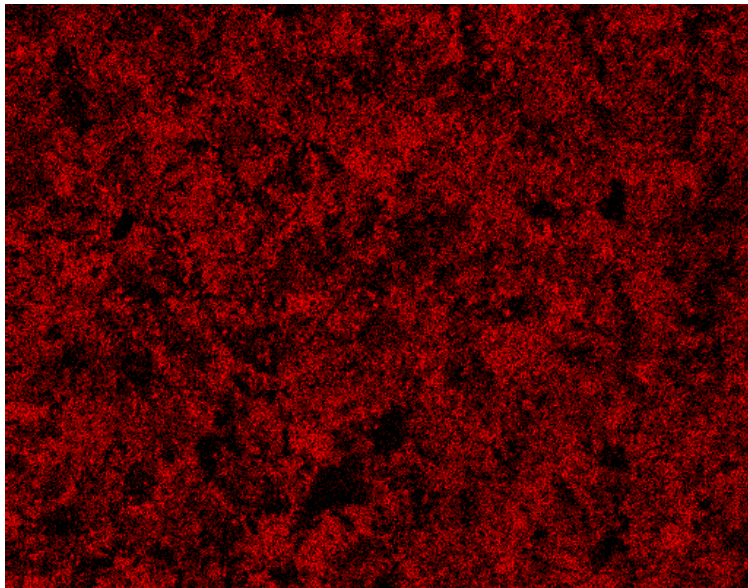


Figure 20: Xray mapping of Iron

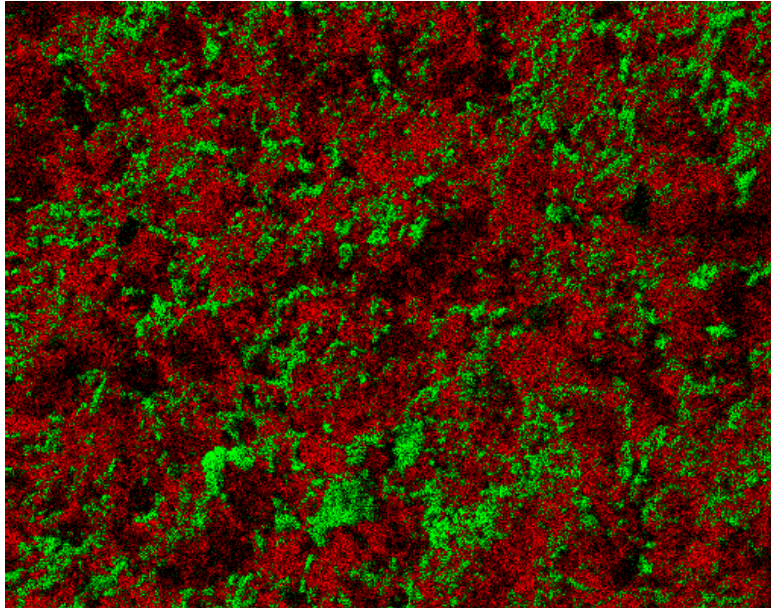


Figure 21: EDS analysis of Overlaid Iron-Aluminum

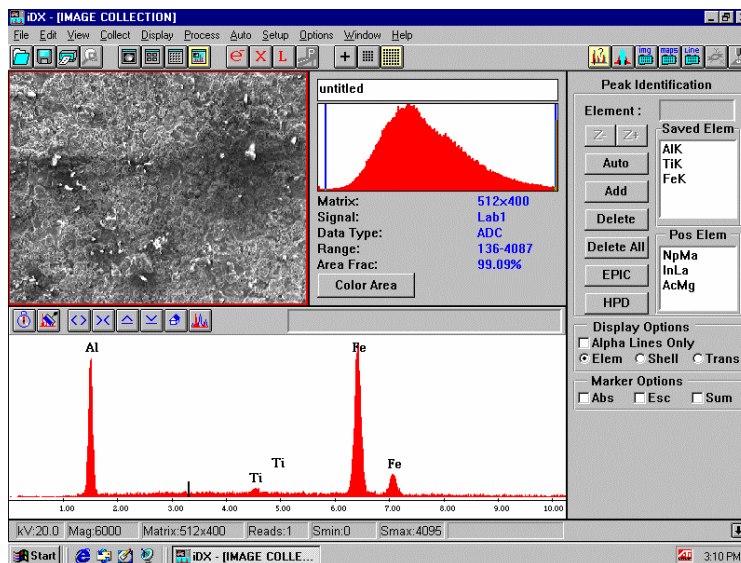


Figure 22: EDS analysis of as sprayed structure show no nickel and chromium from the substrate, which confirm the coverage

Figure 16 to Figure 26 represent the polished optical micrograph, SEM micrograph, EDS analysis and Xray mapping of sprayed (Plasma Spray) structure and inconnel 718 substrate. EDS analysis and X ray mapping confirmed that the inconnel substrate is nicely covered by iron aluminide sprayed structure with good interface bonding at the substrate and sprayed structure

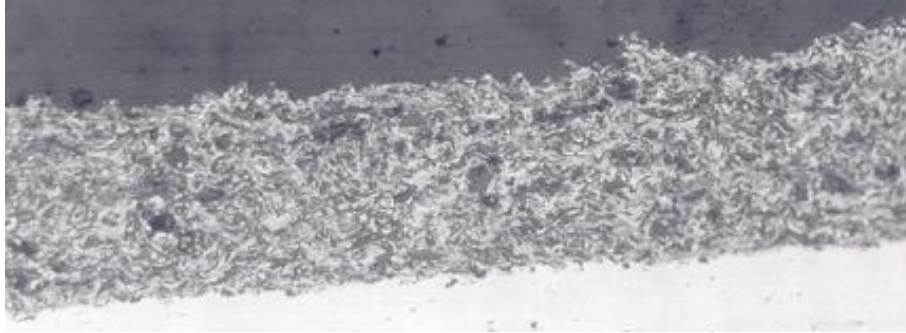


Figure 23: Polished/Optical micrograph of alumina reinforced iron aluminide plasma sprayed layer

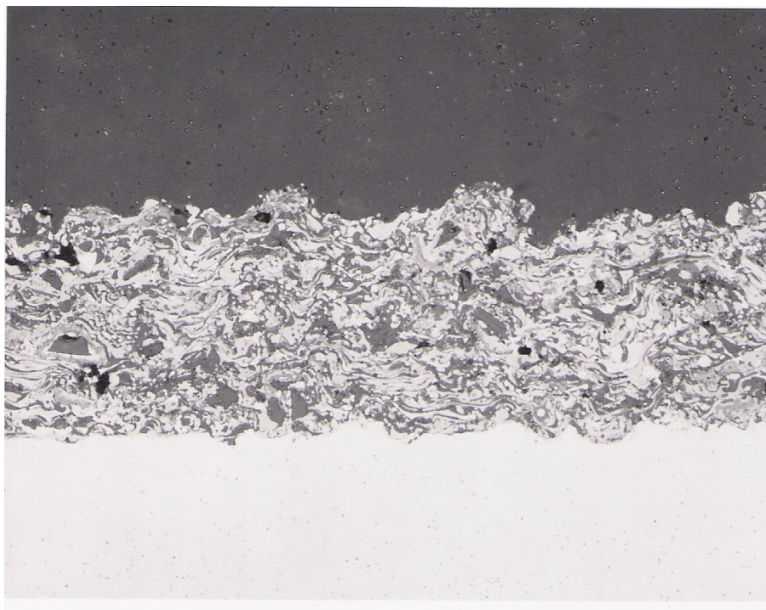


Figure 24: Polished/Optical micrograph of alumina reinforced iron aluminide plasma sprayed layer

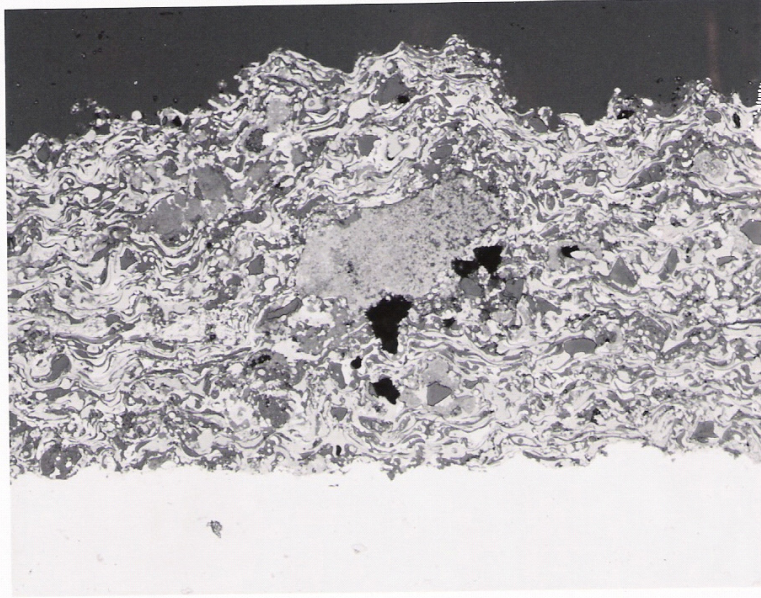


Figure 25: Polished/Optical micrograph of alumina reinforced iron aluminide plasma sprayed layer-show some agglomerates

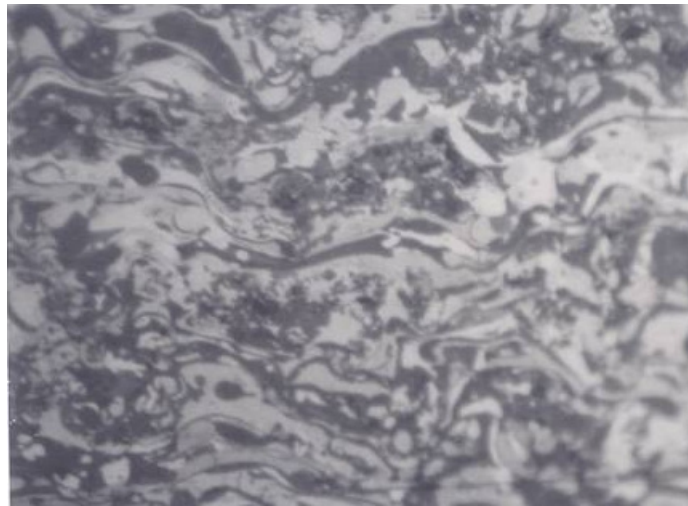


Figure 26: Polished/Optical micrograph of alumina reinforced iron aluminide plasma sprayed layer

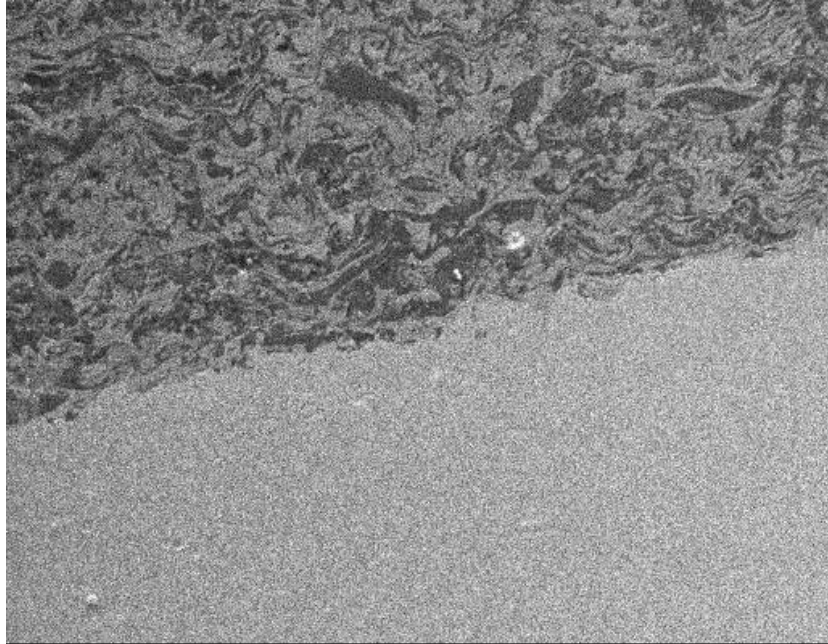


Figure 27: SEM micrograph of alumina reinforced iron aluminide plasma sprayed structure showing good interface bonding with substrate

Powdermet optimized and fine tuned spray parameters and finally plasma sprayed and HVOF sprayed coating on Inconel 718 and 304H stainless steel substrate using following spray parameters. Table 4 represents plasma sprayed coating composition and thickness.

Table 4 : Plasma Sprayed Fe₃Al and Alumina reinforced Fe₃Al Coating on 718 and 304 H substrate

Substrate Composition	Coating Layer Composition	Coating Thickness (mil)
1 Inconel 718	Alumina reinforced Fe ₃ Al	9-11
2 Inconel 718	Fe ₃ Al	9-11
3 304H Stainless Steel	Alumina reinforced Fe ₃ Al	9-12
4 304H Stainless Steel	Fe ₃ Al	9-11

Figure 28 to figure 35 represent iron aluminide and alumina reinforced iron aluminide coating on 304H and IN 718 substrate by plasma spray and HVOF process. Figure show adherent and diffect free coating.

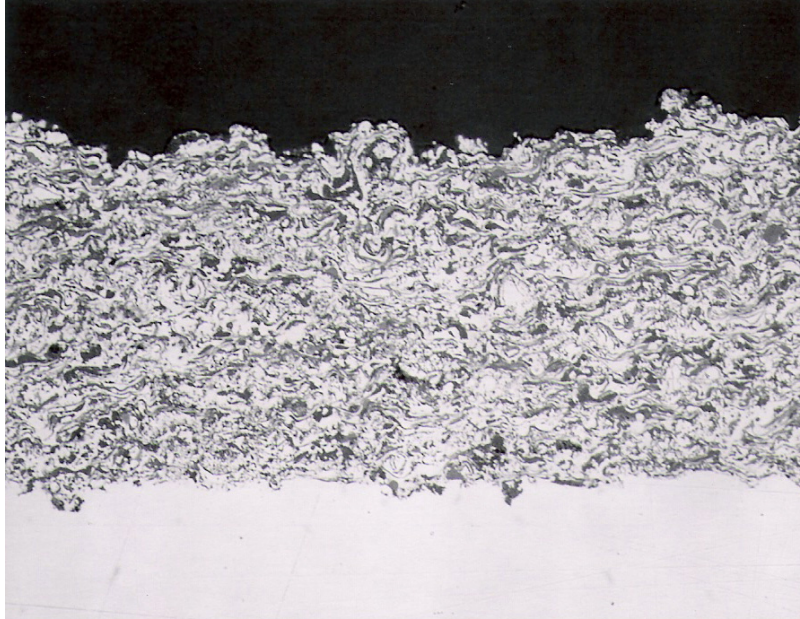


Figure 28: Plasma Sprayed Fe_3Al on 304H, coating thickness .009-.0105 inch X200

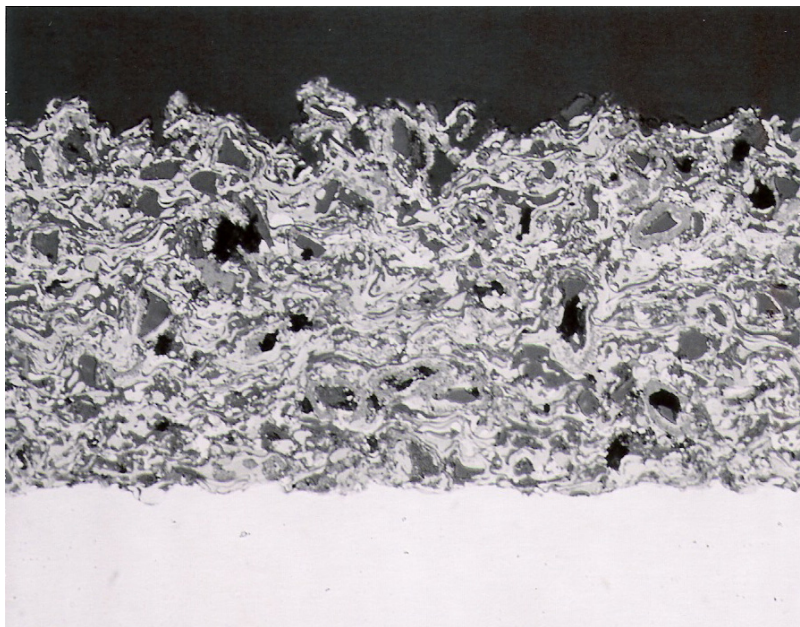


Figure 29: Plasma Sprayed Alumina reinforced Fe_3Al on 304H, coating thickness .009-.012 inch X200

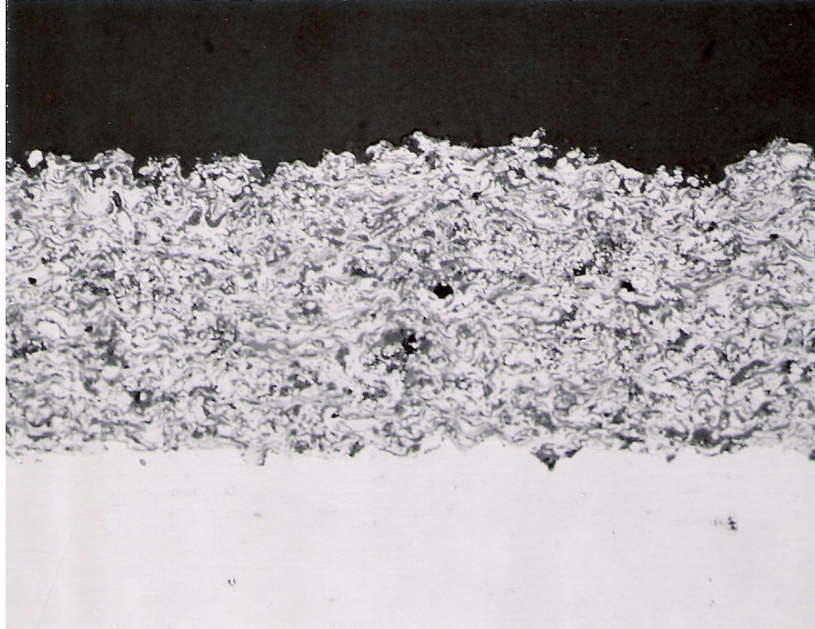


Figure 30: Plasma Sprayed Fe₃Al on 718 coating thickness: .0075-.009 inch X200

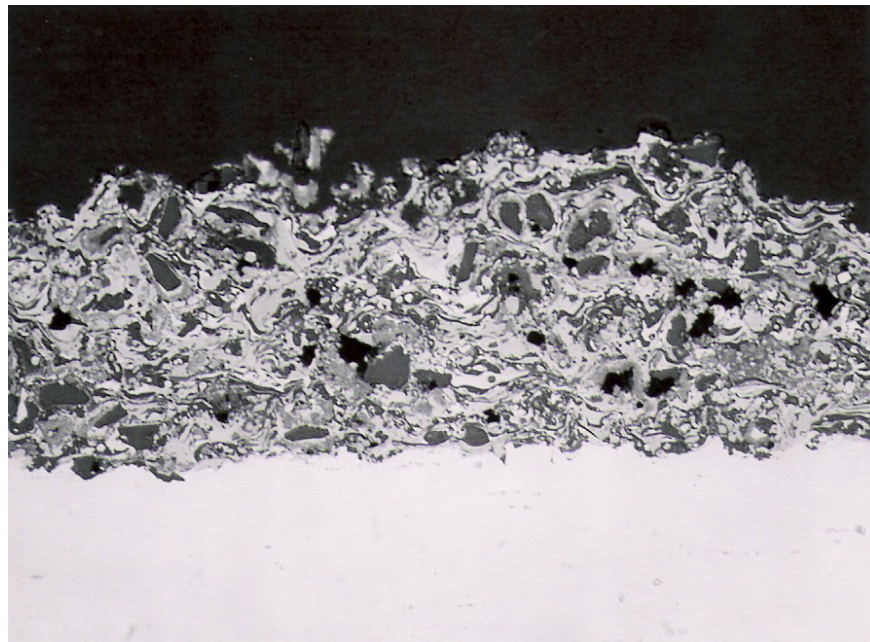


Figure 31: Plasma Sprayed Alumina reinforced Fe₃Al on 718, coating thickness .006-.008 inch X200

Similarly Fe₃Al and alumina reinforced Fe₃Al coatings were sprayed on 304H and IN 718 substrate by HVOF. But Fe₃Al could not be sprayed more than 2 mils in case of HVOF process in case of spraying 9-10 micron size aluminum iron coated iron powders. Probably spray drying is necessary. Table 5 represents HVOF sprayed coating composition and thickness

Table 5: HVOF Sprayed Fe₃Al and Alumina reinforced Fe₃Al Coating on IN 718 and 304 H substrate

Substrate Composition	Coating Layer Composition	Actual Coating Thickness (mil)
1 718	Alumina reinforced Fe ₃ Al	10-11
2 718	Fe ₃ Al	2
3 304H	Alumina reinforced Fe ₃ Al	10-11
4 304H	Fe ₃ Al	2

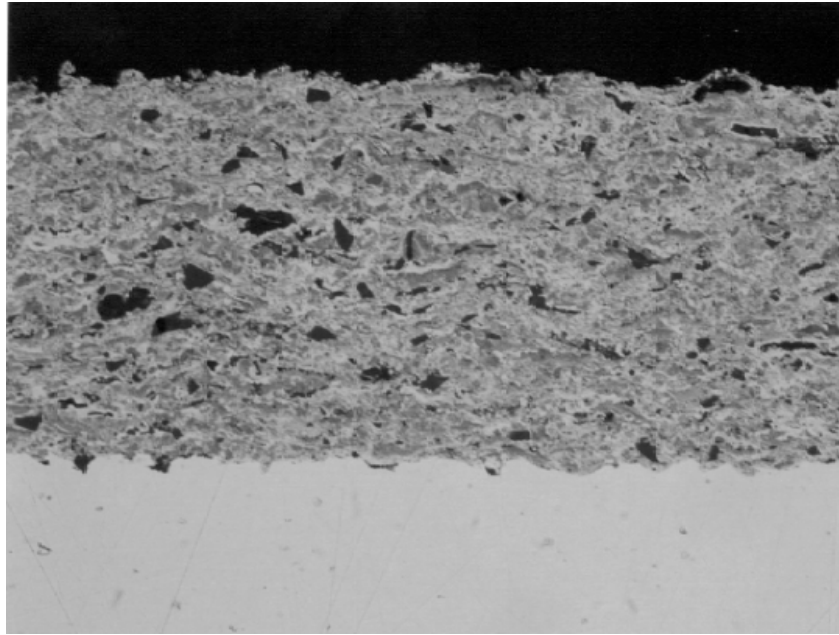


Figure 32: HVOF alumina reinforced Fe₃Al on 718 - coating thickness .010/.011 inch
X200

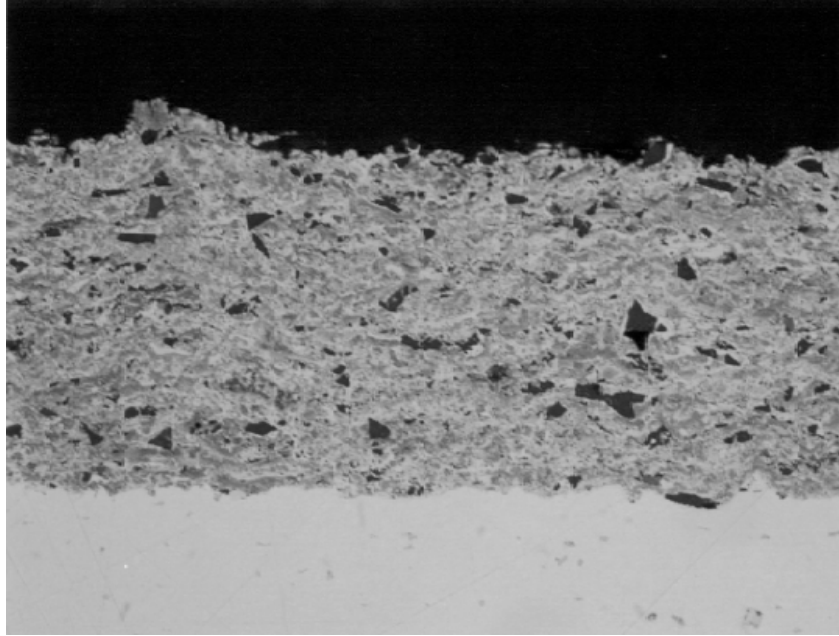


Figure 33: HVOF alumina reinforced Fe_3Al on 304 X200 coating thickness .010/.011 inch

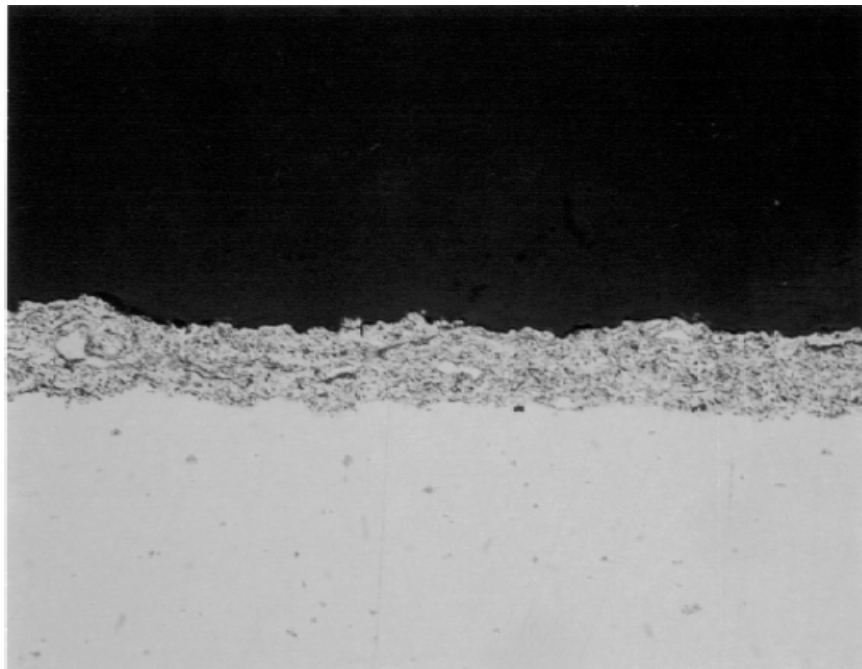


Figure 34: HVOF Fe_3Al on 304 -coating thickness .002 inch X200

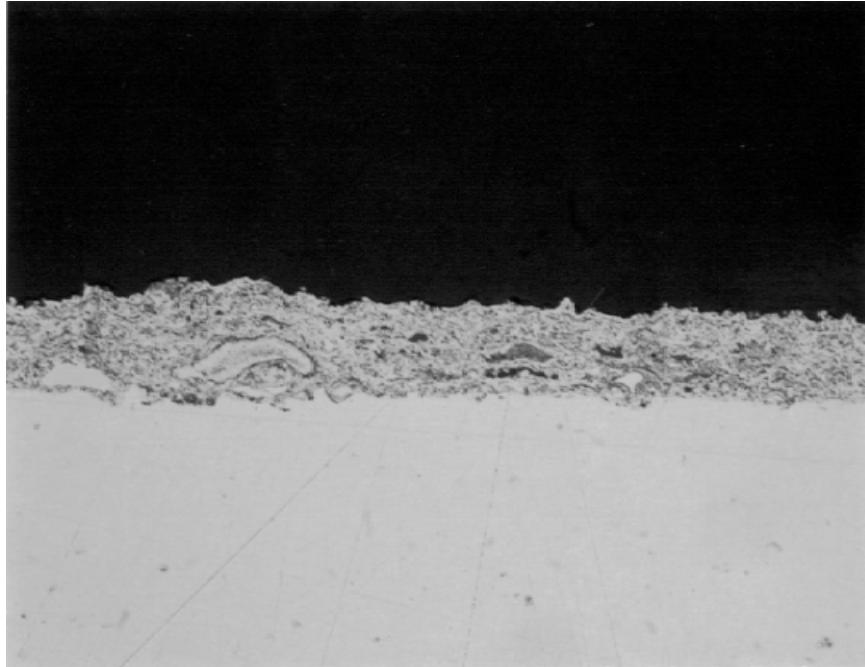


Figure 35: HVOF Fe₃Al on 718 X200 coating thickness .002 inch

Hardness Test: Hardness was measured using a Vickers diamond pyramid indenter at 300 gram loads.

Plasma Sprayed Alumina reinforced Fe₃Al coating on 718 – Average hardness 422.4 Vickers hardness number (VHN), Thickness = .006/ .0085”

Plasma Sprayed Fe₃Al coating on 718– average hardness was 425.8 VHN, thickness = .0065/ .009”

Plasma Sprayed Alumina reinforced Fe₃Al coating on 304, average hardness was 372.4 VHN, thickness = .009/ .012”

Plasma Sprayed Fe₃Al coating on 304 Average hardness was 395.6, thickness = .0091/ .0105”

Detailed hardness results are presented in table 6 to table 13. Hardness value of HVOF sprayed coating are higher than plasma sprayed coating

Table 6: Hardness of Plasma sprayed Alumina Reinforced Fe₃Al on 718

Test #	Coating Thickness	VHN	Load	Average Hardness (VHN)
1		518.6		
2		454.1		
3		533.2		
4	.006/.0085	278.4	300 g	422.4
5		459.3		
6		411.9		
7		331.7		
8		472.8		
9		343.3		
10		421.0		

Table 7: Hardness of Plasma Sprayed Fe₃Al on 718

Test #	Coating Thickness	VHN	Load	Average Hardness
1		534.8		
2		371.4		
3		373.3		
4	.0065/.009	280.3	300 g	425.8
5		616.0		
6		229.3		
7		405.2		
8		489.8		
9		529.9		
10		428.0		

Table 8: Hardness of Plasma Sprayed Oxide reinforced Fe₃Al coating on 304H

Test #	Coating Thickness	VHN	Load	Average Hardness
1		458.0		
2		385.2		
3		345.9		
4	.0091/.012	290.6	300 g	372.4
5		340.0		
6		307.2		
7		298.0		
8		312.3		
9		557.1		
10		430.4		

Table 9: Hardness of Plasma Sprayed Fe₃Al coating on 304H

Test #	Coating Thickness	VHN	Load	Average Hardness
1		413.0		
2		446.4		
3		381.2		
4	.0091/.0105	330.1	300 g	395.6
5		384.2		
6		399.8		
7		482.6		
8		424.5		
9		313.1		
10		381.2		

HVOF process:

Table 10: Hardness of HVOF sprayed Alumina Reinforced Fe₃Al on 718

Test #	Coating Thickness	VHN	Load	Average Hardness
1		400.9		
2		464.6		
3		376.2		
4	.0101/.011	459.3	300 g	490.03
5		518.6		
6		529.9		
7		564.2		
8		562.4		
9		434.0		
10		590.2		

Table 11: Hardness of HVOF Sprayed Fe₃Al on 718

Test #	Coating Thickness	VHN	Load	Average Hardness
1		506.2		
2		582.6		
3		421.0		
4	.002	482.6	300 g	522.2
5		518.6		
6		492.7		
7		538.2		
8		566.0		
9		531.5		
10		582.6		

Table 12: Hardness of HVOF Sprayed Oxide reinforced Fe₃Al coating on 304H

Test #	Coating Thickness	VHN	Load	Average Hardness
1		518.6		
2		531.5		
3		575.1		
4	.0101/.011	518.6	300 g	518.43
5		550.1		
6		452.8		
7		510.8		
8		513.9		
9		492.7		
10		520.2		

Table 13: Hardness of HVOF Sprayed Fe₃Al coating on 304H

Test #	Coating Thickness	VHN	Load	
1		495.7		
2		489.8		
3		513.9		
4	.002	488.4	300 g	504.01
5		531.5		
6		601.9		
7		488.4		
8		491.3		
9		426.8		
10		512.4		

Erosion Test: Grid blast erosion test was done by alumina grit

Parameters used for the test are described below:

Erodent: SS White Alumina Powder

Erodent Size: 50 – 60 microns

Erodent Mass Load: 600 gm

Air Pressure: 24.5 psi

Vibrator Pressure: 17 psi

Distance from nozzle: 4 inches

Fe₃Al density: 6.5 g/ cm³

Al₂O₃ reinforced Fe₃Al density: 5.5 g/cm³

Test Time: 103-106 seconds

Table 14 represents Erosion Test Results of Plasma Spray and HVOF sprayed Fe₃Al and Alumina Reinforced Fe₃Al on IN 718 and 304H Substrate

Table 14: Erosion Test Results of Plasma Spray and HVOF sprayed Fe₃Al and Alumina Reinforced Fe₃Al on IN 718 and 304H Substrate

Erosion data for plasma sprayed samples

Angle	Coating	Time (sec)	Ri (μin)	Rf (μin)	Wi (gm)	Wf (gm)	Wi-Wf (gm)	Erosion rate mg/g
90°	Al ₂ O ₃ -Fe ₃ Al / Inco 718	107.9	284	25	18.1826	17.998	0.1846	0.3076667
	Fe ₃ Al / Inco 718	104.8	184	67	17.8937	17.7608	0.1329	0.2215
	Al ₂ O ₃ -Fe ₃ Al / 304H	104.6	248	27	15.3563	15.1434	0.2129	0.3548333
	Fe ₃ Al / 304H	111.8	183	71	15.2154	15.0776	0.1378	0.2296667

Erosion data for HVOF coated samples

Angle	Coating	Time (sec)	Ri (μin)	Rf (μin)	Wi (gm)	Wf (gm)	Wi-Wf (gm)	Erosion rate mg/g
90°	Al ₂ O ₃ -Fe ₃ Al / Inco 718	109.7	159	32, 58	27.2279	27.0915	0.1364	0.2273333
	Fe ₃ Al / Inco 718	107.5	113	25	25.8163	25.7264	0.0899	0.1498333
	Al ₂ O ₃ -Fe ₃ Al / 304H	109.9	164	36, 49	27.3911	27.2541	0.137	0.2283333
	Fe ₃ Al / 304H	105.1	124	25	25.6919	25.5997	0.0922	0.1536667

Ri= Initial Roughness before Erosion test

Rf= Roughness after Erosion test

Thermal sprayed coating surface became smoother after erosion test. But coating remained intact after the test. Figure 36 to figure 41 represent the SEM photo micrograph and EDS analysis after Erosion test

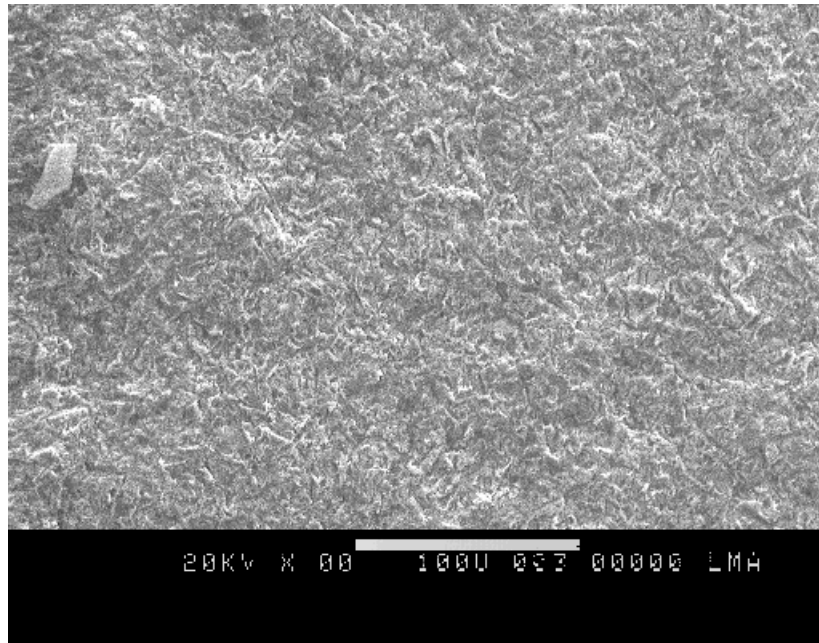


Figure 36: Fe₃Al Coating on 304H substrate after erosion test. Coating remained intact and surface roughness reduced after erosion testX370

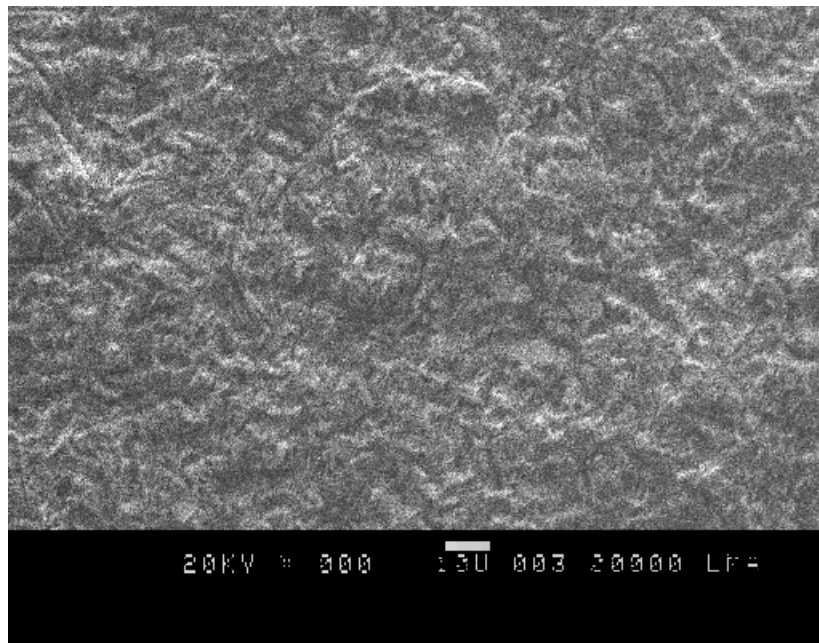


Figure 37: Fe₃Al sprayed coating after Erosion Test X700

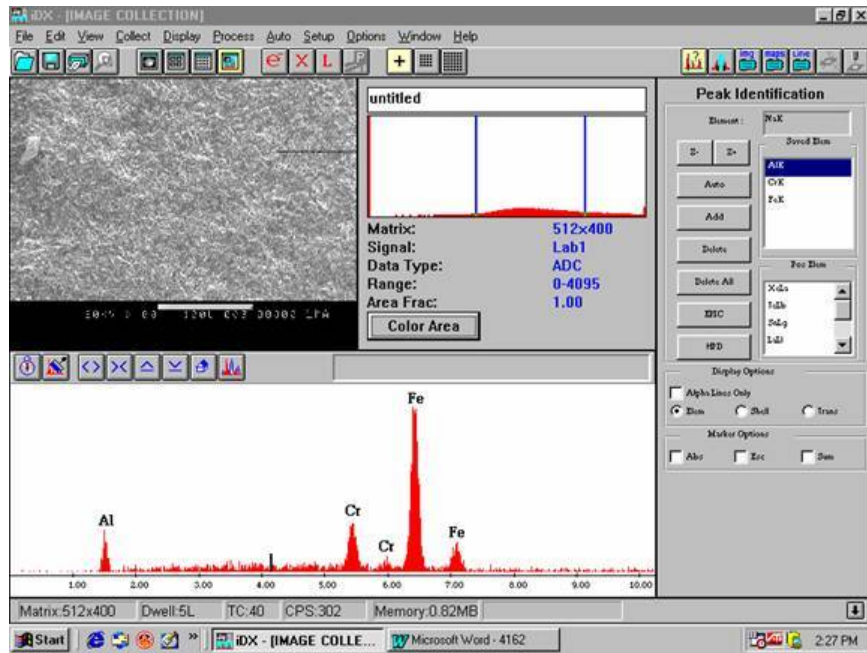


Figure 38: EDS analysis show alumina reinforced iron aluminide coating

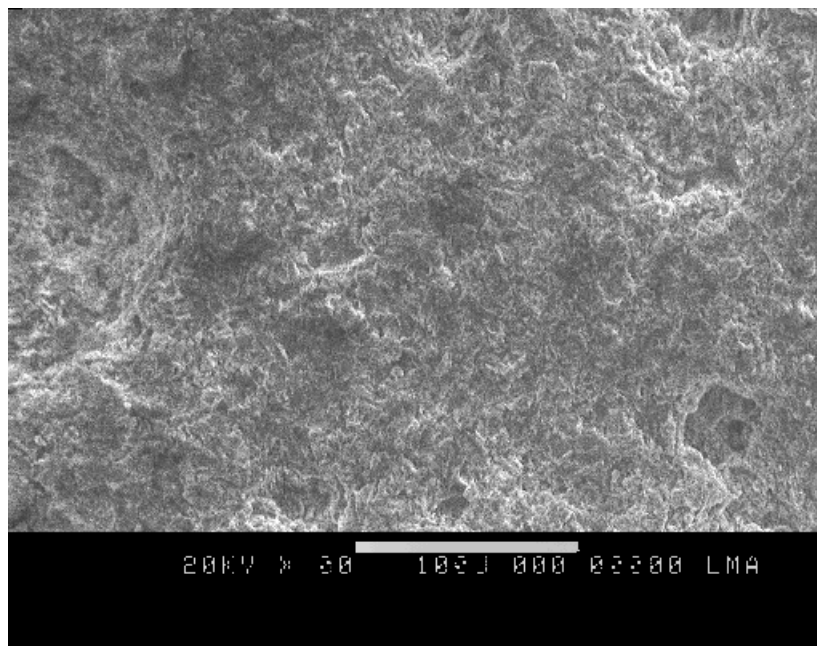


Figure 39: SEM micrograph showing alumina reinforced Fe_3Al coating after erosion test showing eroded more X370

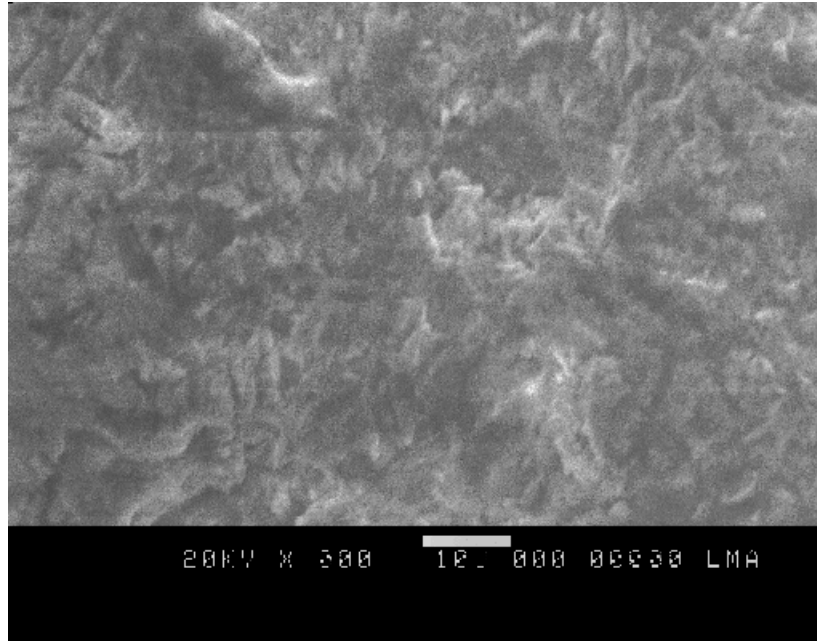


Figure 40 Alumina reinforced Fe₃Al after erosion test X1400

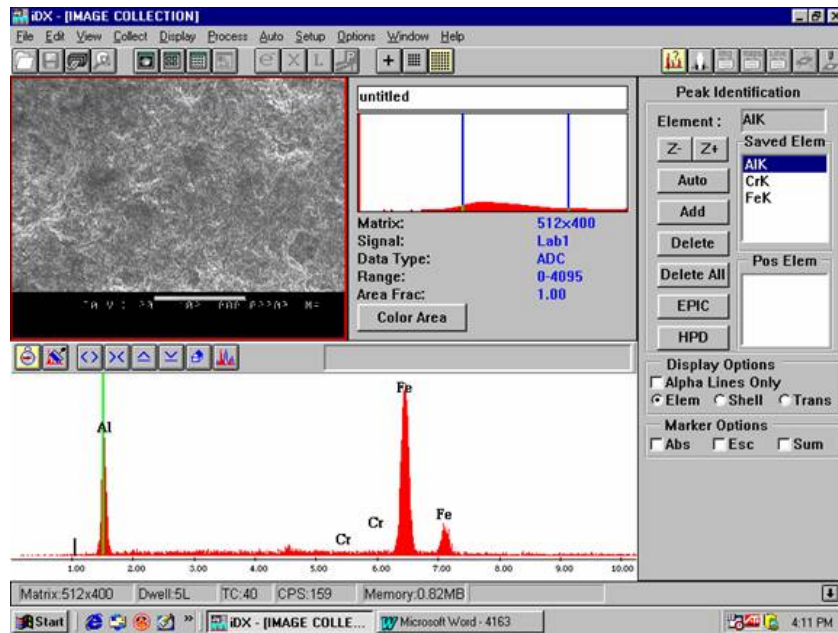


Figure 41: EDS analysis showing that alumina reinforced iron aluminide coating

From the SEM microscopy and erosion test results it was concluded that the Fe_3Al and alumina reinforced Fe_3Al coating sprayed on IN 718 and 304H was adherent to substrate and did not delaminate during test. Erosion rate of alumina reinforced iron aluminide coating was higher than the iron aluminide coating sprayed on both substrate and erosion rate of plasma spray coating is higher than the HVOF coating. But once again both coating passed the erosion test

Preparation of Oxidation, Carburization and Corrosion Test Samples:

Thermal sprayed coatings were characterized for carburization, Oxidation, cyclic oxidation, sulfidation/corrosion and Erosion Test:

Uncoated IN 718, 304 H , steel , Fe_3Al and alumina reinforced Fe_3Al thermal Sprayed IN 718 and 304H samples were cut into small pieces. The samples five faces were covered with alumina paste, dried and only one face was exposed for oxidation, carburization and corrosion tests. Figure 42 to Figure 44 represent how samples were prepared with alumina paste and made ready for the oxidation, carburization and sulfidation/corrosion tests.

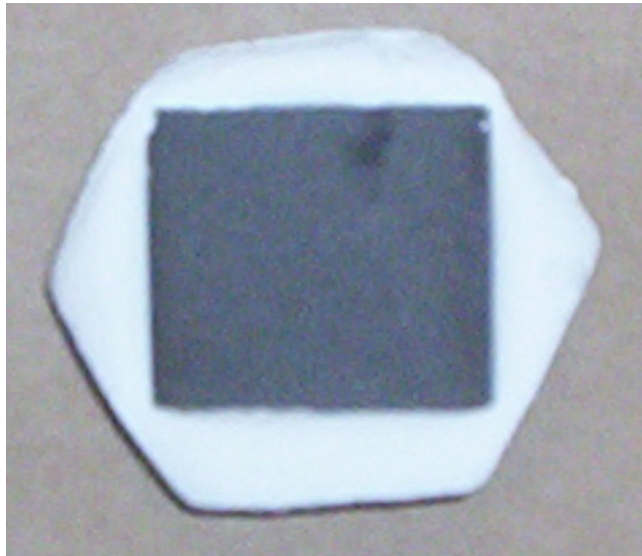


Figure 42: Showing Thermal Sprayed Exposed, other faces are covered in Alumina



Figure 43: Side view show Thermal Sprayed Sample one face is open for exposure, other faces are covered in Alumina



Figure 44: Side view show Thermal Sprayed Sample one face is exposed, other faces are covered in Alumina

Carburization resistance test was done in an atmosphere of hydrogen containing 2% methane by exposing the test samples at 1000C for 100 hours and weight gain (mg/cm^2) with a function of time was compared with Incoloy 718, alloy 304 H. Figure 45 to 47 represents the test samples after carburization test. Table 15 represents the carburization test results. Results revealed that Thermal sprayed iron aluminide coating is a carburization resistant material



Figure 45: Free carbon deposited on samples after carburization

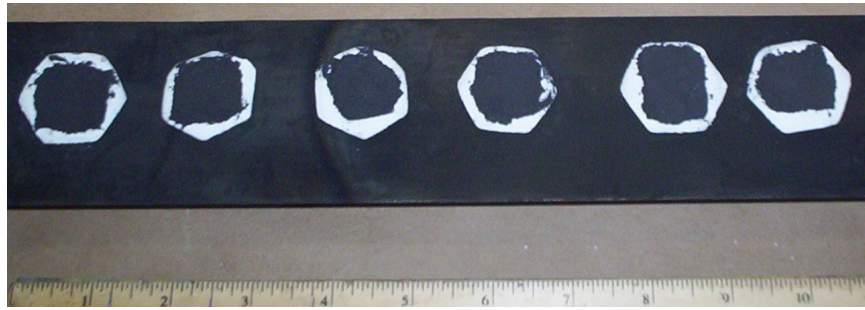


Figure 46: Uncoated and thermal sprayed Samples after Carburization tests



Figure 47: Showing free carbon accumulated on thermal sprayed tested sample

Table 15: Carburization Test Results of Fe₃Al and Alumina Reinforced Fe₃Al Coating on 304H and IN 718 Substrate

Sample	Type of Thermal Spray Coating	Weight Gain (mg/cm ²) after 1000 Hrs Carburization at 1000 °C
Fe ₃ Al on 304 H	Plasma Spray	0.105
Alumina reinforced Fe ₃ Al on 304 H	Plasma Spray	0.102
Fe ₃ Al on 718	Plasma Spray	0.098
Alumina reinforced Fe ₃ Al on 718	Plasma Spray	0.097
Fe ₃ Al on 304 H	HVOF	0.103
Alumina reinforced Fe ₃ Al on 304 H	HVOF	0.101
Fe ₃ Al on 718	HVOF	0.0975
Alumina reinforced Fe ₃ Al on 718	HVOF	0.0965
Uncoated 718	No Coating	0.185
Uncoated 304 H	No Coating	0.198
Steel	No coating	0.255

From the carburization test results it was concluded that weight gain of uncoated steel, IN 718 and 304 H were more than Fe₃Al and alumina reinforced Fe₃Al coating sprayed on IN 718 and 304H. But still coatings were adherent to substrate, did not spall off during carburization test and the coating protected the substrate from the carburization. It was also found that there was little difference between carburization rate of alumina reinforced iron aluminide coating and the iron aluminide coating sprayed on both substrates by both the processes plasma spray and HVOF. But once again both coating showed carburization resistant properties and protected the substrate.

Coal Ash Corrosion Test:

Coal ash corrosion involves the reaction of alkali sulfate deposits that form an overlaying product on the metal surface. The reaction between the sulfates and the surrounding environment results in a liquid **alkali-iron trisulfate** (AIT) phase forming beneath the sulfate layer. This liquid phase has a high solubility for oxides (e.g. Fe_xO_y, Al_xO_y, Ni_xO_y), is in direct contact with the metal surface and performs a fluxing breakdown of the protective scale. The synthetic ash that has been labeled as the worst case is the one composed of 25 wt% Fe₂O₃-37.5 wt% Na₂SO₄-37.5 wt% K₂SO₄. This particular composition is composed solely of corrodents that are used in the AIT reaction.

Uncoated 304H , IN718 and steel sample, Fe₃Al and alumina reinforced Fe₃Al thermal sprayed (Plasma and HVOF) coating on 304H and IN 718 were exposed with Fe₂O₃-37.5 wt% Na₂SO₄-37.5 wt% K₂SO₄ slag at 1000 °C for 100 hours. Uncoated 304H, IN 718 steel sample was severely attacked by the slag , But Fe₃Al and alumina reinforced sprayed sample substrate was not attacked but coating was partially attacked but it still protected the underneath substrate. Figure 48 to figure 50 represent photographs of test samples after corrosion tests

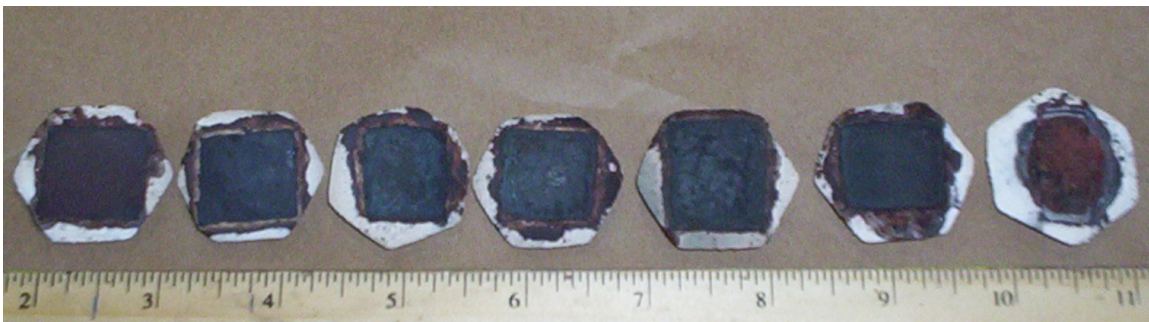


Figure 48: Showing Samples after Corrosion Tests in presence of Fe₂O₃-37.5 wt% Na₂SO₄-37.5 wt% K₂SO₄ for 100 Hours at 1000 °C

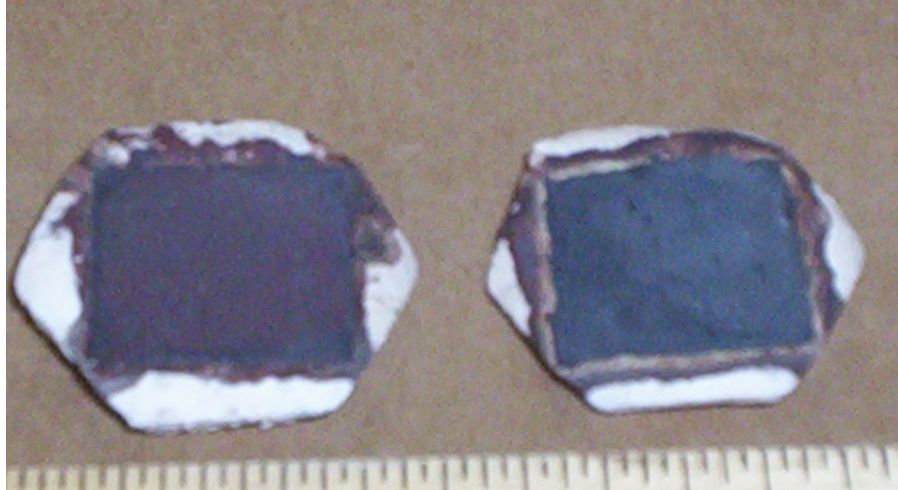


Figure 49: Showing thermal sprayed alumina reinforced Fe₃Al and Fe₃Al 304H partially attacked by this Fe₂O₃, K₂SO₄, Na₂SO₄ but alumina reinforced Fe₃Al showed better property.

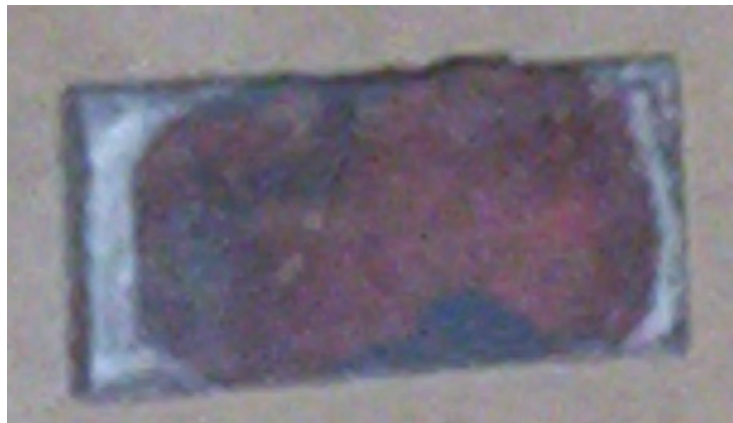


Figure 50: Showing uncoated 304H severely attacked by this Fe₂O₃, K₂SO₄, Na₂SO₄

Table 16 represents the samples appearances after corrosion tests and figure 51 to 55A represent SEM and EDS analysis of corroded uncoated and thermal sprayed coating samples.

Table 16: Corrosion Test Results of Fe₃Al and Alumina Reinforced Fe₃Al Coating on 304H and IN 718 Substrate

Sample	Type of Thermal Spray Coating	Corrosion Attack
Fe ₃ Al on 304 H	Plasma Spray	Partial corrosion on coating but no corrosion on the substrate
Alumina reinforced Fe ₃ Al on 304 H	Plasma Spray	Partial corrosion on coating but no corrosion on the substrate
Fe ₃ Al on 718	Plasma Spray	Partial corrosion on coating but no corrosion on the substrate
Alumina reinforced Fe ₃ Al on 718	Plasma Spray	Partial corrosion on coating but no corrosion on the substrate
Fe ₃ Al on 304 H	HVOF	Partial corrosion on coating but no corrosion on the substrate
Alumina reinforced Fe ₃ Al on 304 H	HVOF	Partial corrosion on coating but no corrosion on the substrate
Fe ₃ Al on 718	HVOF	Partial corrosion on coating but no corrosion on the substrate
Alumina reinforced Fe ₃ Al on 718	HVOF	Partial corrosion on coating but no corrosion on the substrate
Uncoated 718	No Coating	Severe attack, corroded 718
Uncoated 304 H	No Coating	Severe attack, corroded 304 H
Steel	No coating	Severe attack , corroded and crumbled

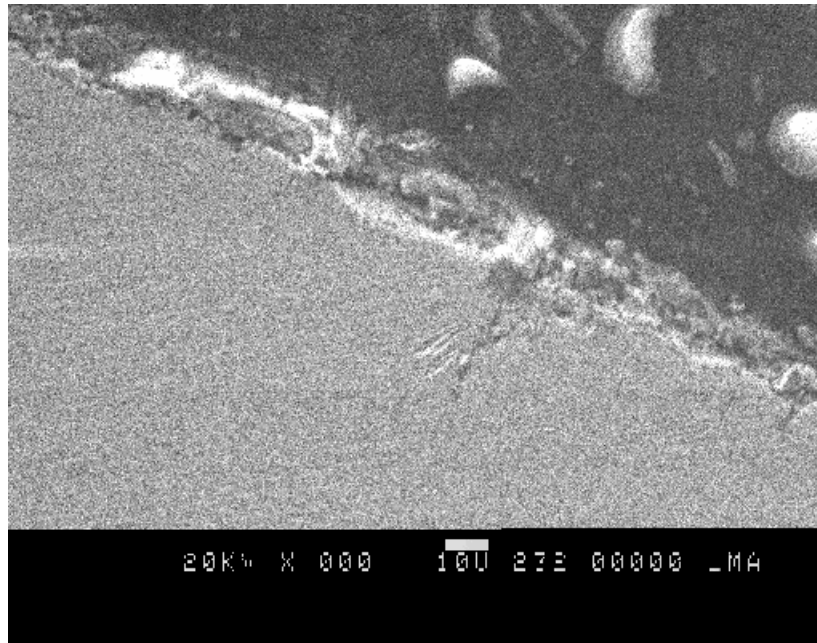


Figure 51: SEM micrograph showing uncoated 304H is corroded and layer is falling off

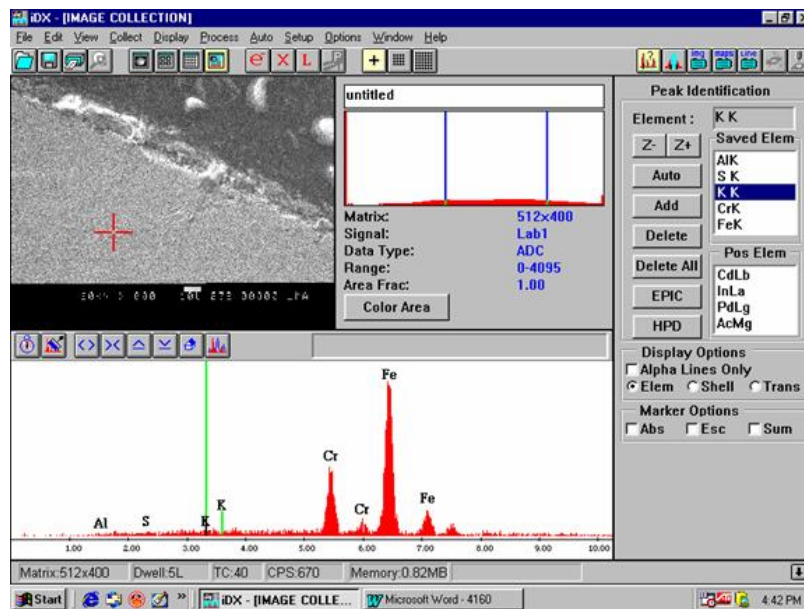


Figure 52: EDS analysis Showing under the corroded layer is comprised of iron and chromium -304H

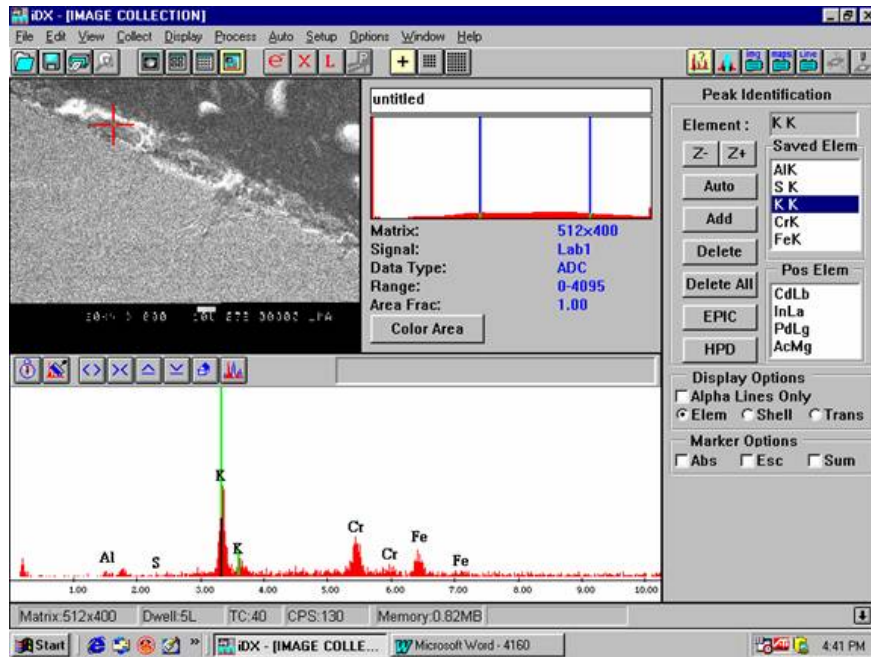


Figure 53 Showing that uncoated 304H surface was attacked by K_2SO_4 and Na_2SO_4

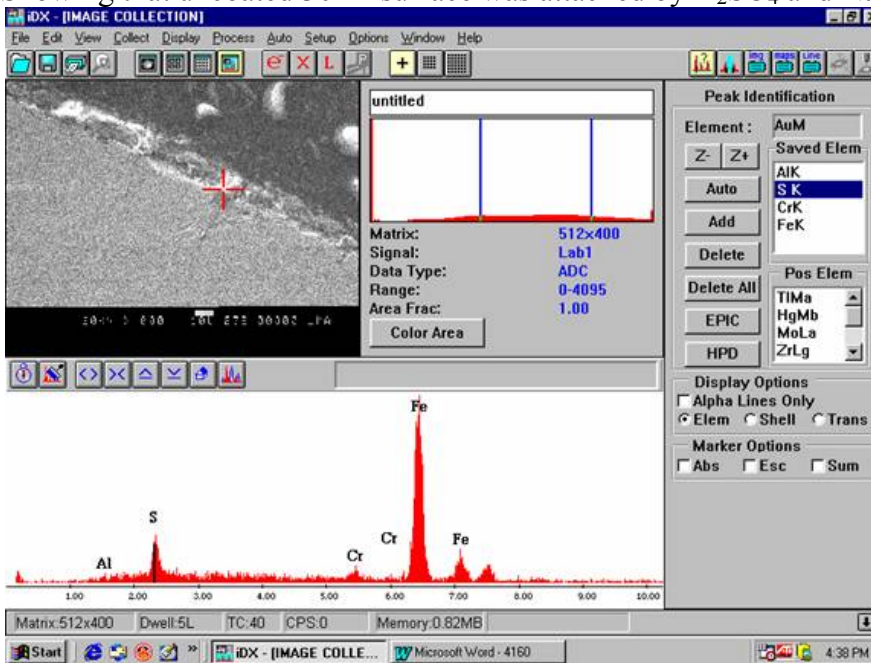


Figure 54: Showing that uncoated 304H surface was attacked by K_2SO_4 and Na_2SO_4

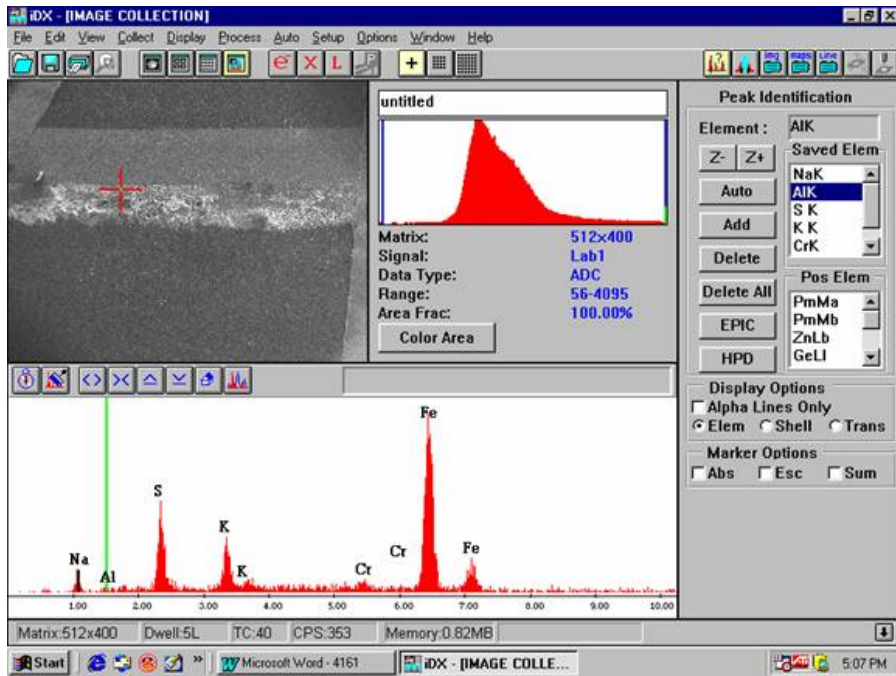


Figure 55: Showing thermal sprayed alumina reinforced Fe_3Al on 304H partially attacked by this Fe_2O_3 , K_2SO_4 , Na_2SO_4 but coating remains intact and underneath layer was not attacked

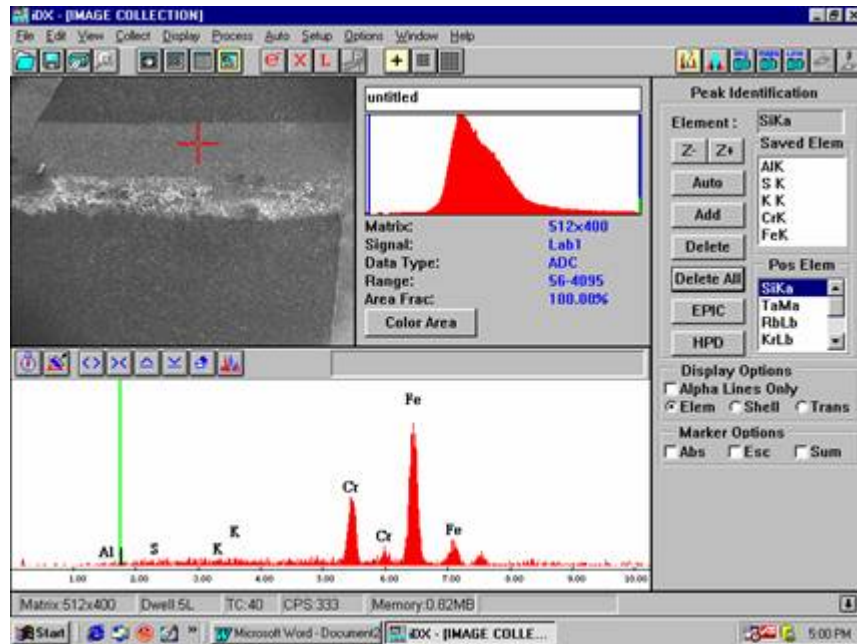


Figure 55A: Showing thermal sprayed alumina reinforced Fe_3Al on 304H partially attacked by this Fe_2O_3 , K_2SO_4 , Na_2SO_4 but coating remains intact and underneath layer was not attacked EDS analysis showing only Fe, Cr

From the SEM microscopy and corrosion test results it was concluded that uncoated steel, IN 718 and 304 H were severely attacked by this alkali slag, But Fe_3Al and alumina reinforced Fe_3Al coating sprayed on IN 718 and 304H were also partially attacked by this slag. But still coatings were adherent to substrate, did not spall off during corrosion test and the coating protected the substrate from the slag reaction. It was also found that corrosion rate of alumina reinforced iron aluminide coating was lower than the iron aluminide coating sprayed on both substrate and corrosion rate of plasma spray coating was higher than the HVOF coating. But once again both coating showed corrosion resistant properties and protected the substrate.

Oxidation Testing: Cyclic Oxidation testing was done on alumina reinforced Fe_3Al Sprayed, Fe_3Al sprayed 304H, IN718 , uncoated 718, 304 H and mild steel in atmosphere of air containing 5% H_2O . Temperature was cycled between $1000^{\circ}C$ and room temperature for 100 hours. The specimens was evaluated for weight change, scale morphology and microscopy. Figure 56 to 57 represent alumina reinforced Fe_3Al Sprayed, Fe_3Al sprayed 304H, IN718 , uncoated 718, 304 H and mild steel Samples after Cyclic Oxidation Tests and oxidation test profile. Figure 58 to Figure 83 represent SEM micrograph of alumina reinforced Fe_3Al Sprayed, Fe_3Al sprayed coating and uncoated 304H substrate after oxidation tests

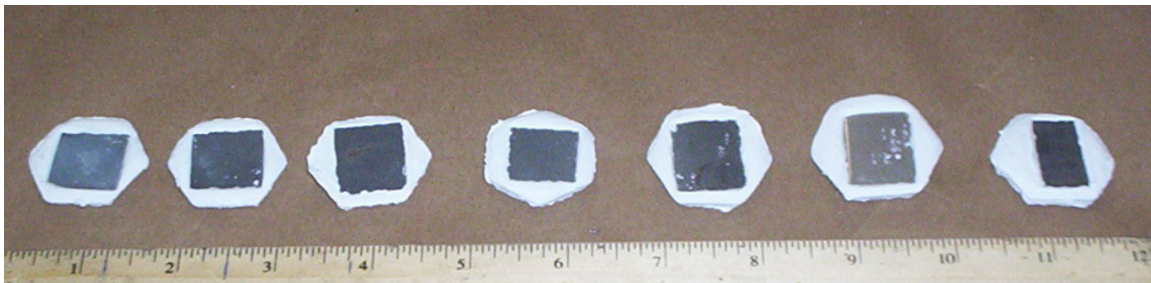
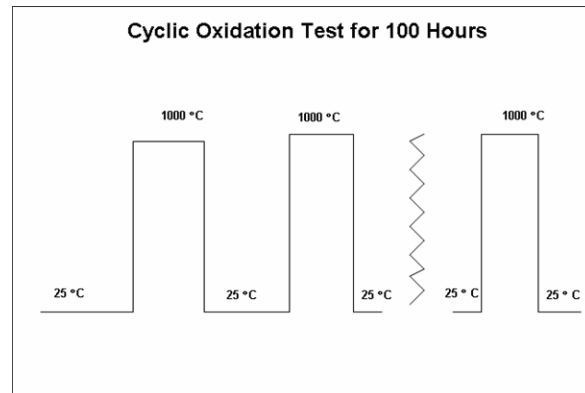


Figure 56: Showing alumina reinforced Fe_3Al Sprayed, Fe_3Al sprayed 304H, IN718 , uncoated 718, 304 H and mild steel Samples after Cyclic Oxidation Tests and oxidation test profile

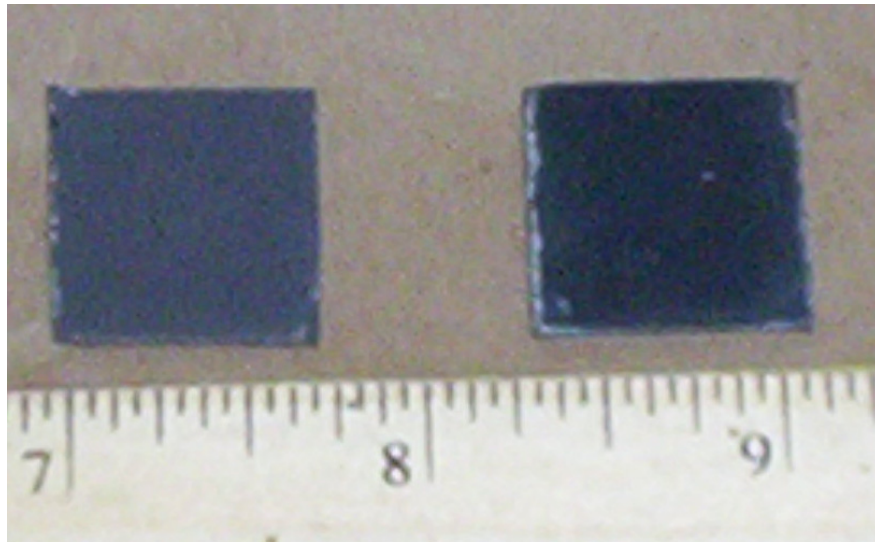


Figure 57: Showing Alumina Reinforced Fe₃Al Sprayed coating is lighter in color than Fe₃Al Sprayed Samples after Cyclic Oxidation Tests. Weight gain is less incase of alumina reinforced thermal sprayed Fe₃Al coating. But coating remained intact after oxidation tests for both Fe₃Al coating and alumina reinforced Fe₃Al coating

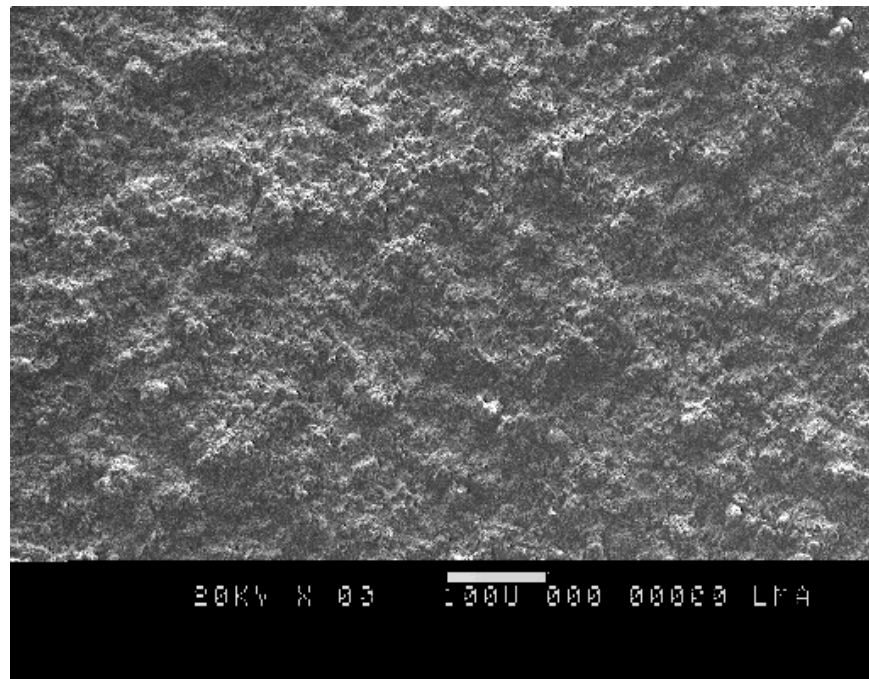


Figure 58: SEM Micrograph showing morphology of as sprayed Fe₃Al coating on 304 H before oxidation test X150

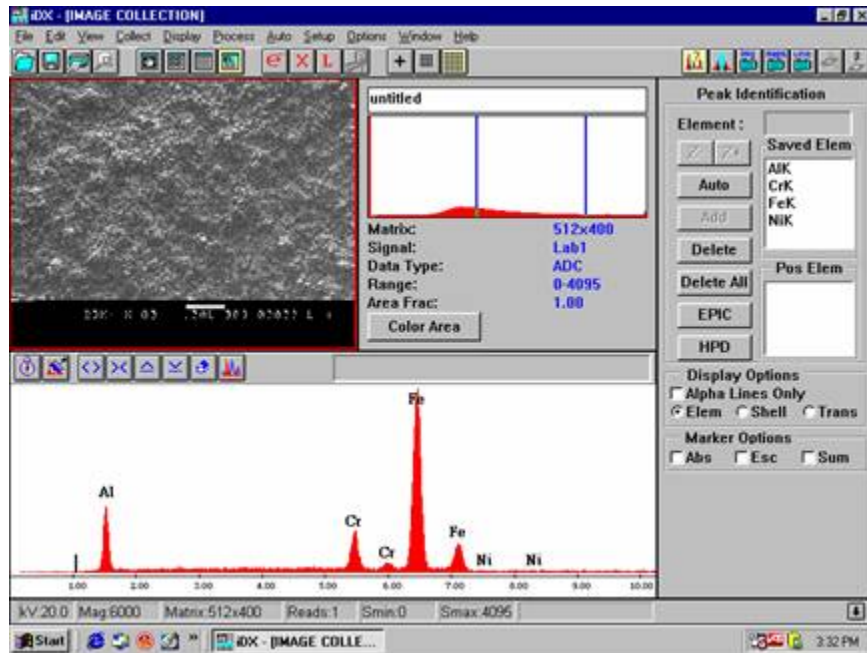


Figure 59: EDS analysis show Fe and Al from Fe_3Al . Cr is from original powders

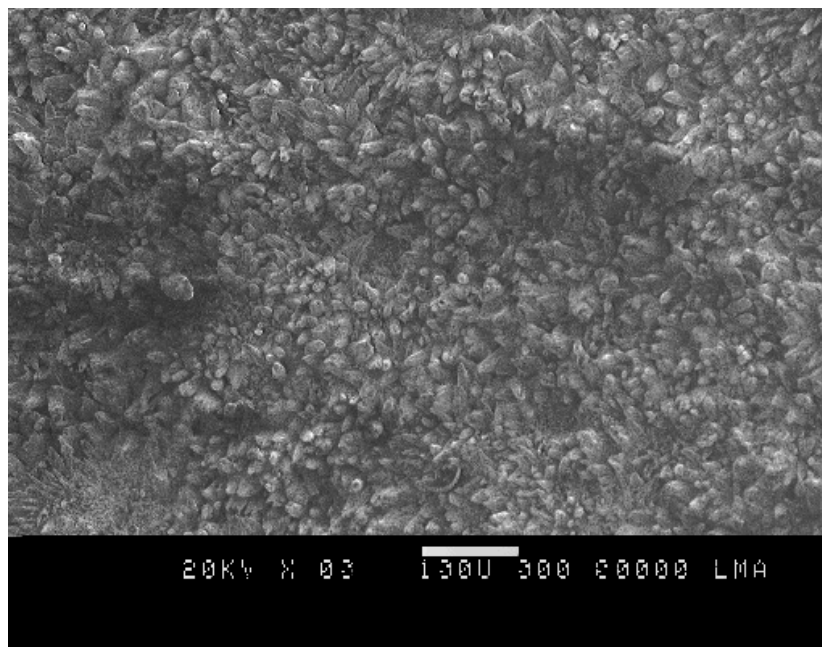


Figure 60: SEM Micrograph of Fe_3Al sprayed on 304 H after the cyclic oxidation test. Figure show some change in morphology but coating showed enough adhesion to protect underneath substrate X150

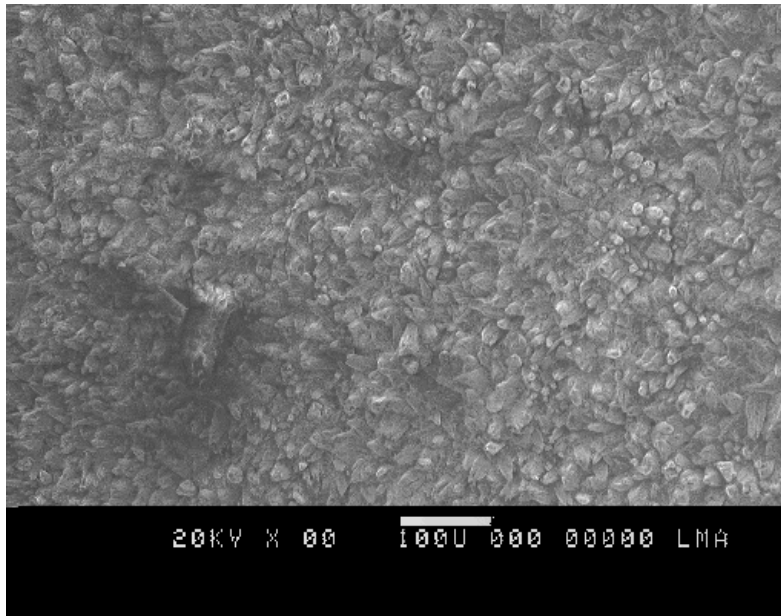


Figure 61: SEM Micrograph of Fe₃Al sprayed on 304 H after the cyclic oxidation test. Figure show some change in morphology but coating showed enough adhesion to protect underneath substrate X150

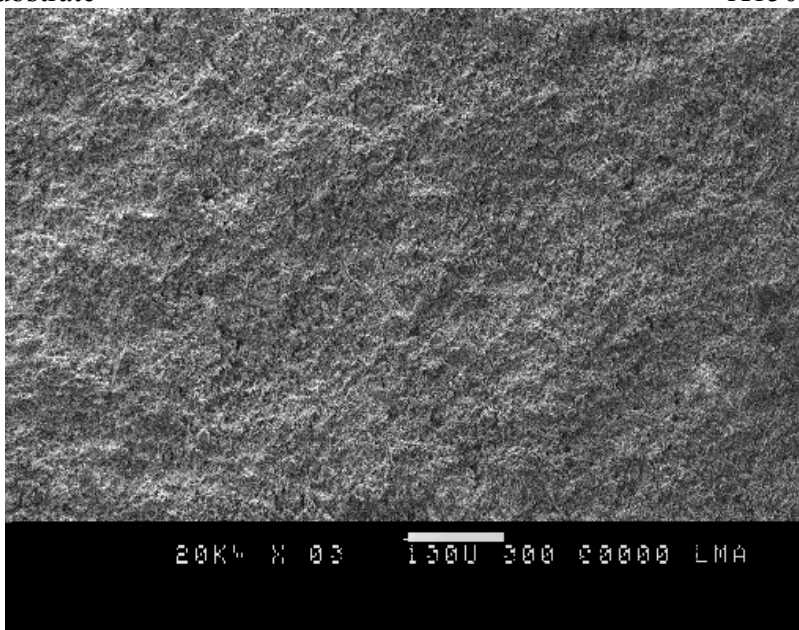


Figure 62: Top oxidized layers were removed underneath oxidized layer showing same morphology like before the test X150

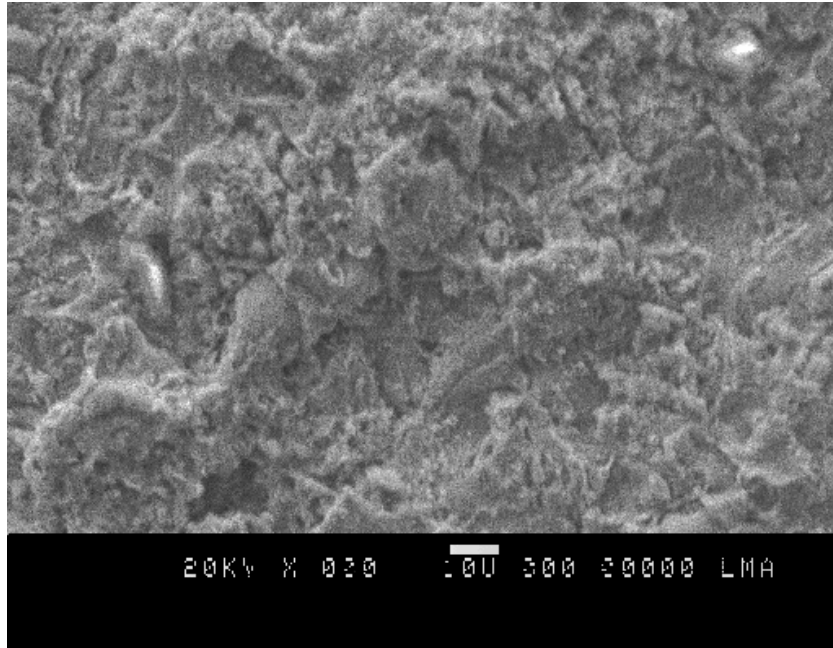


Figure 63: Showing Oxidized layers were removed under the top layer X800

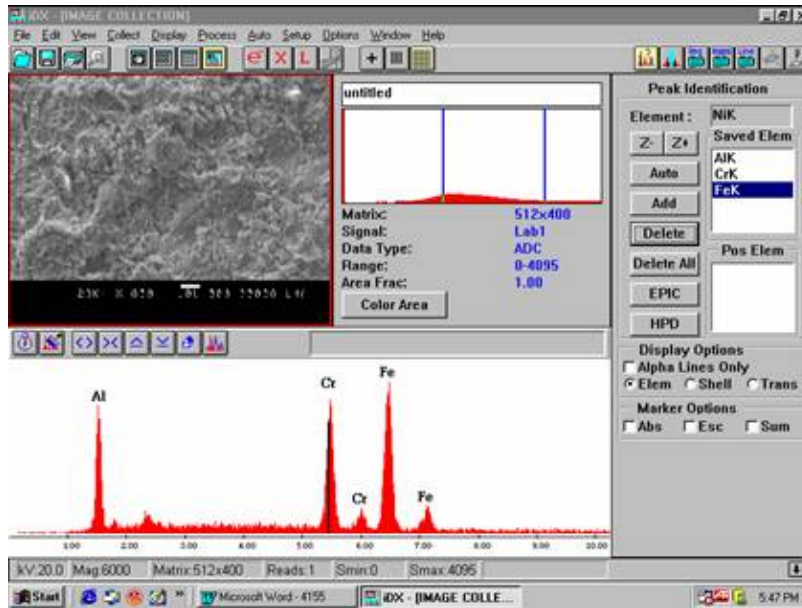


Figure 64: EDS analysis represent Fe₃Al Coating protecting underneath substrate

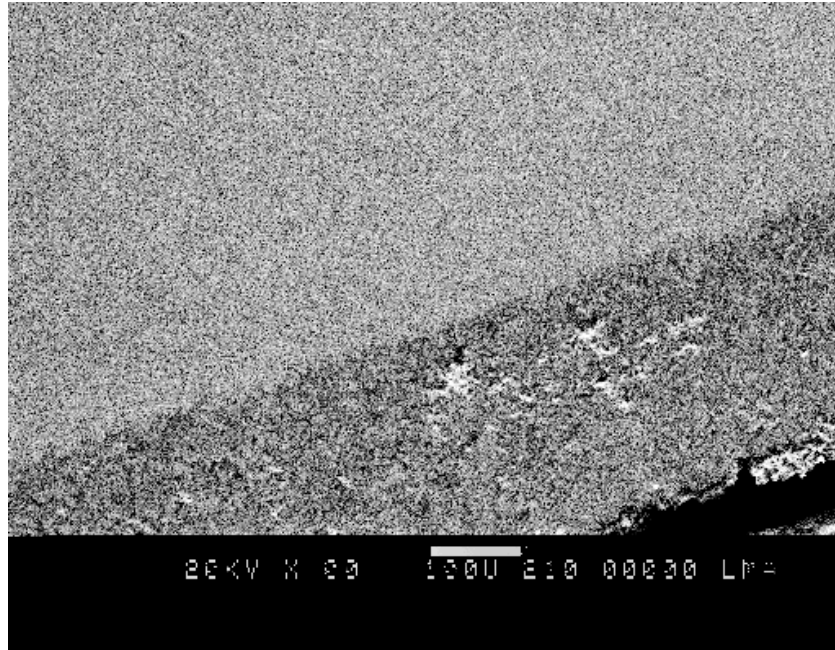


Figure 65: Cross section of the coating represent 10-20 micron thermal Fe₃Al sprayed layers were oxidized after cyclic oxidation test but remaining thermal sprayed layer was not attacked and protected 304H substrate

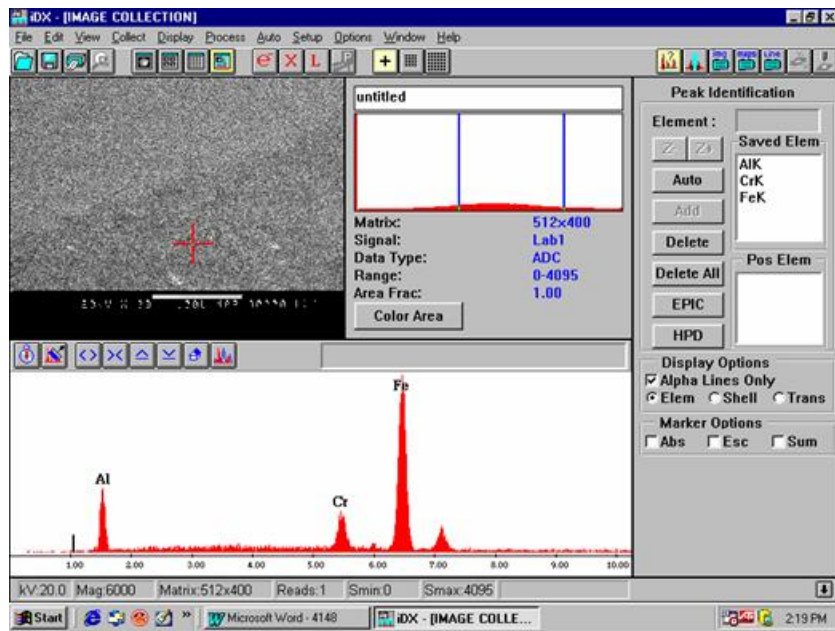


Figure 66: EDS analysis of coating after the oxidation test show Fe, Al, Cr which is similar to before oxidation test

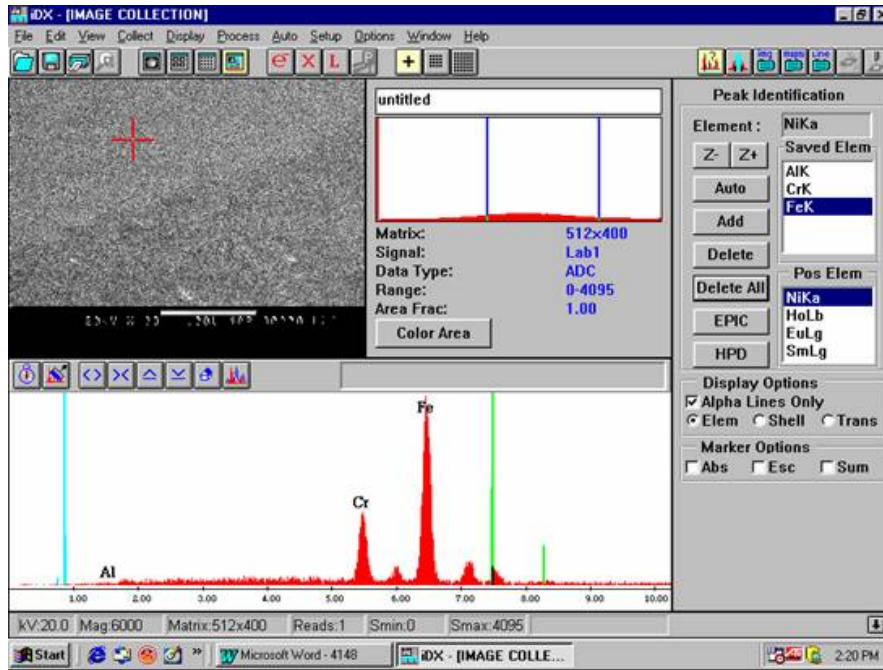


Figure 67: EDS analysis showing only Fe and Cr in the 304 H substrate.

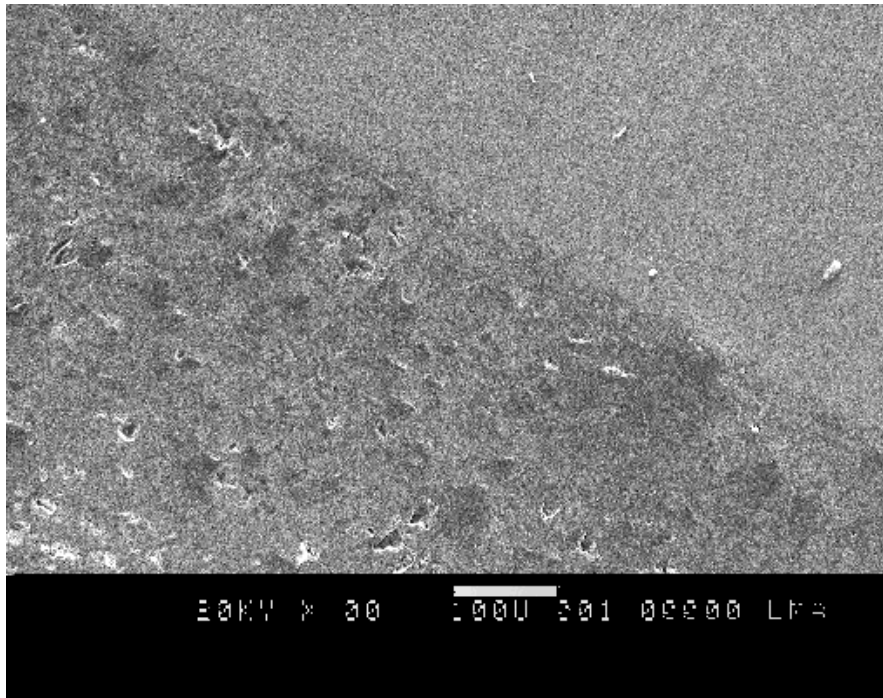


Figure 68: Alumina reinforced iron aluminide coating on 304 H after oxidation test showing nicely bonded with substrate and protecting the substrate from oxidation X150

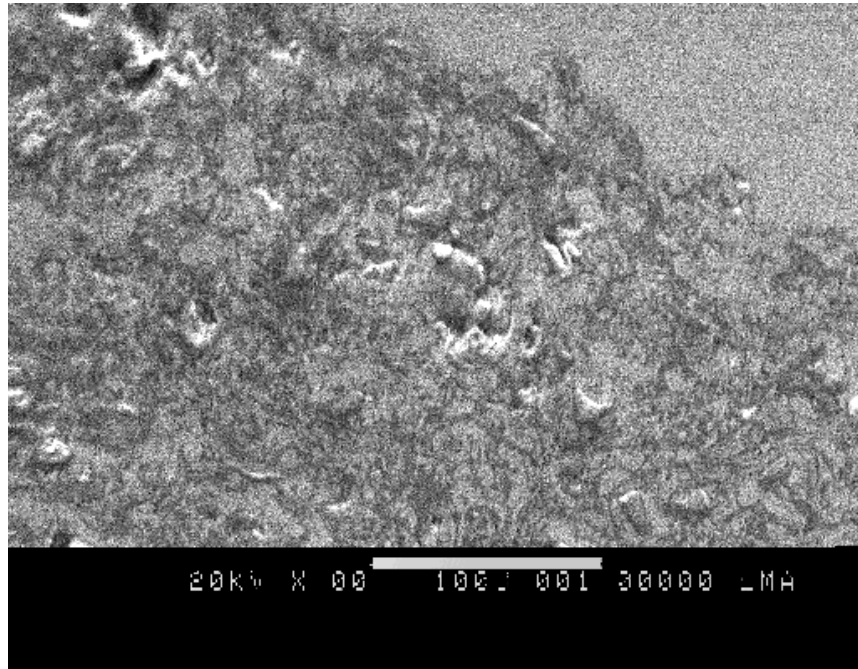


Figure 69: Alumina reinforced iron aluminide coating on 304H after oxidation test X350

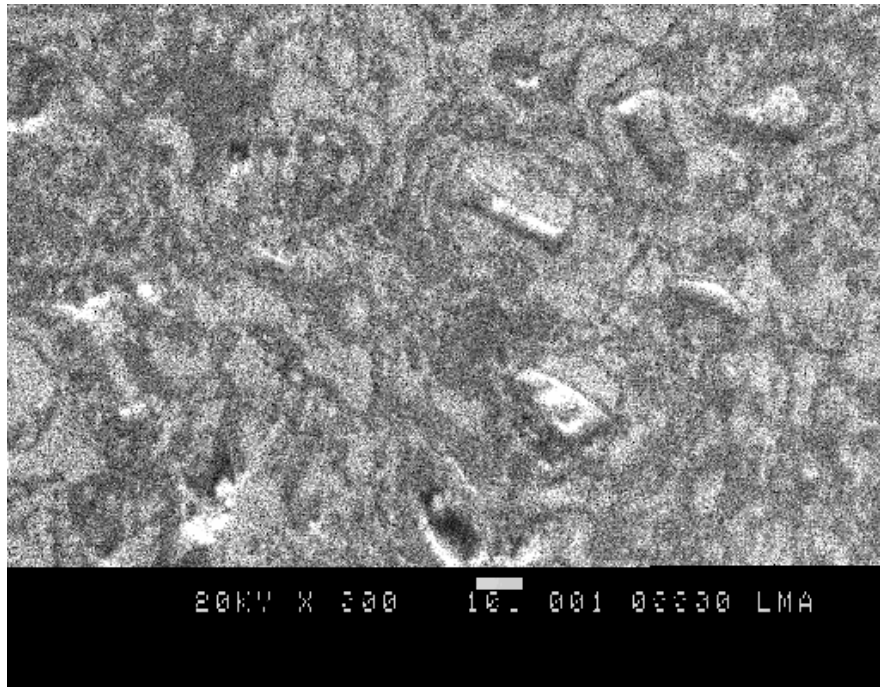


Figure 70: SEM micrograph of Alumina reinforced iron aluminide coating on 304H after cyclic oxidation , Showing alumina particles are still nicely bonded with Fe₃Al matrix for protecting substrate

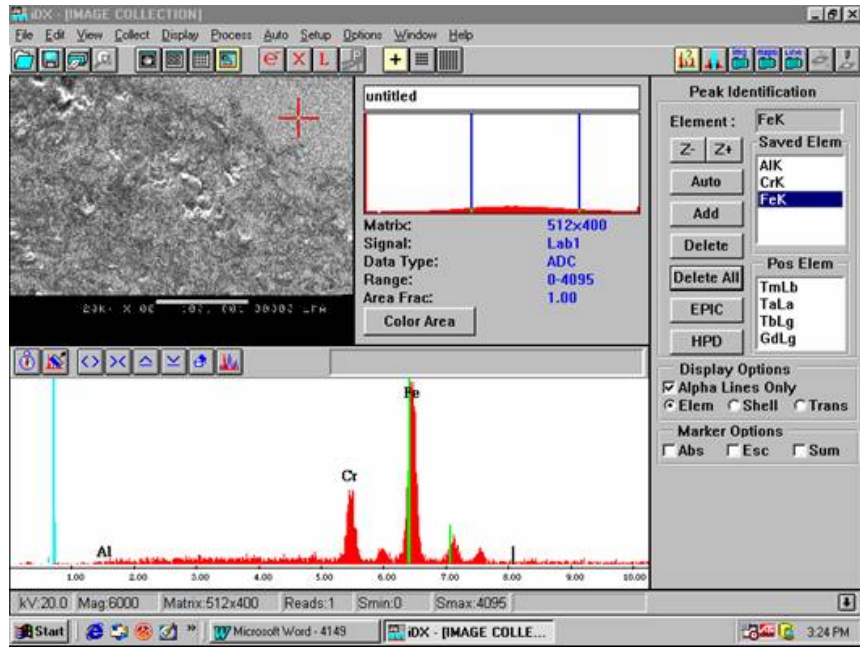


Figure 71:EDS analysis of 304 H substrate

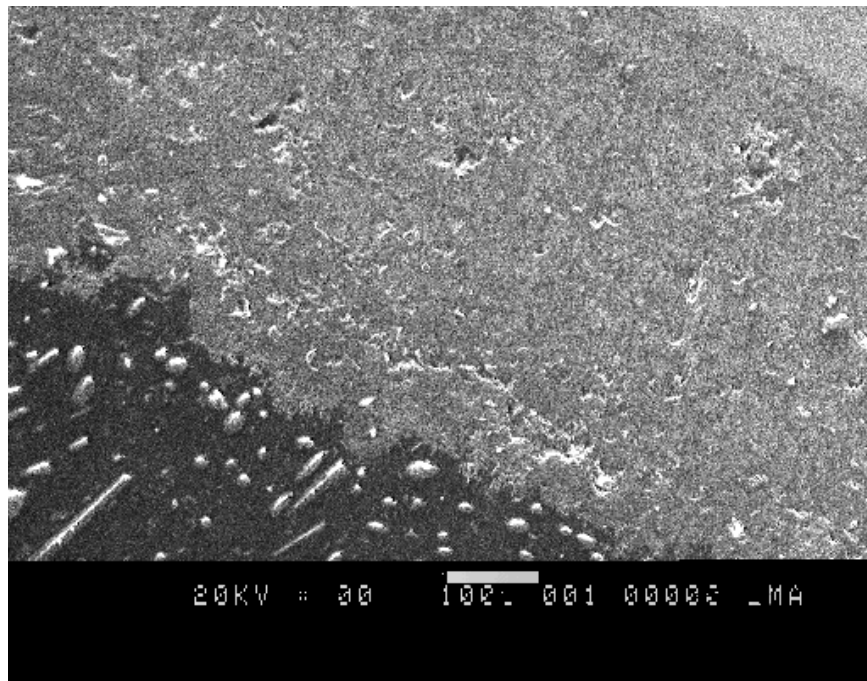


Figure 72: 100 micron oxide layer X140

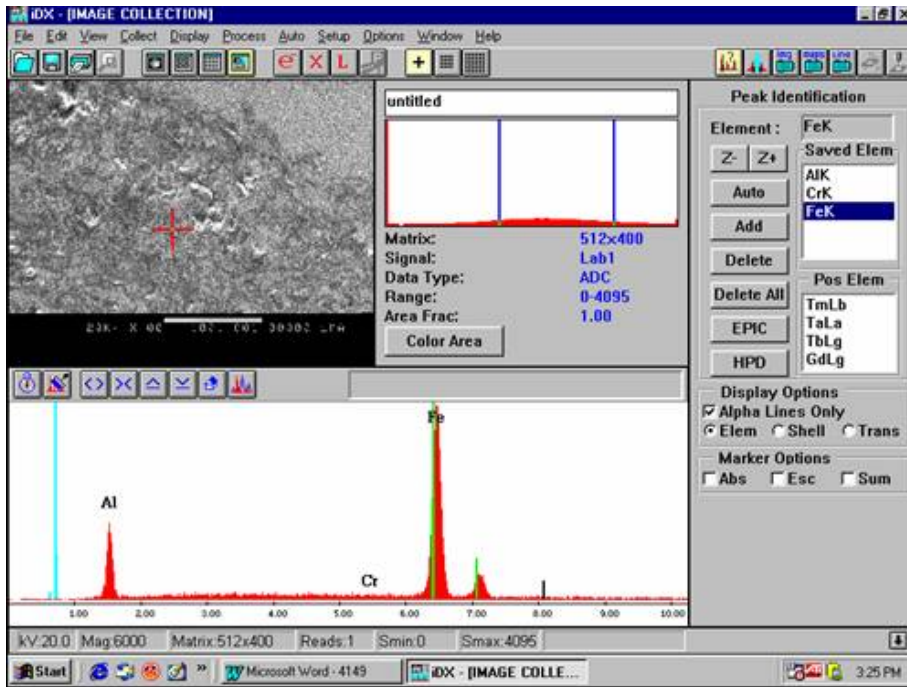


Figure 73: EDS analysis showing alumina reinforced Fe₃Al coating

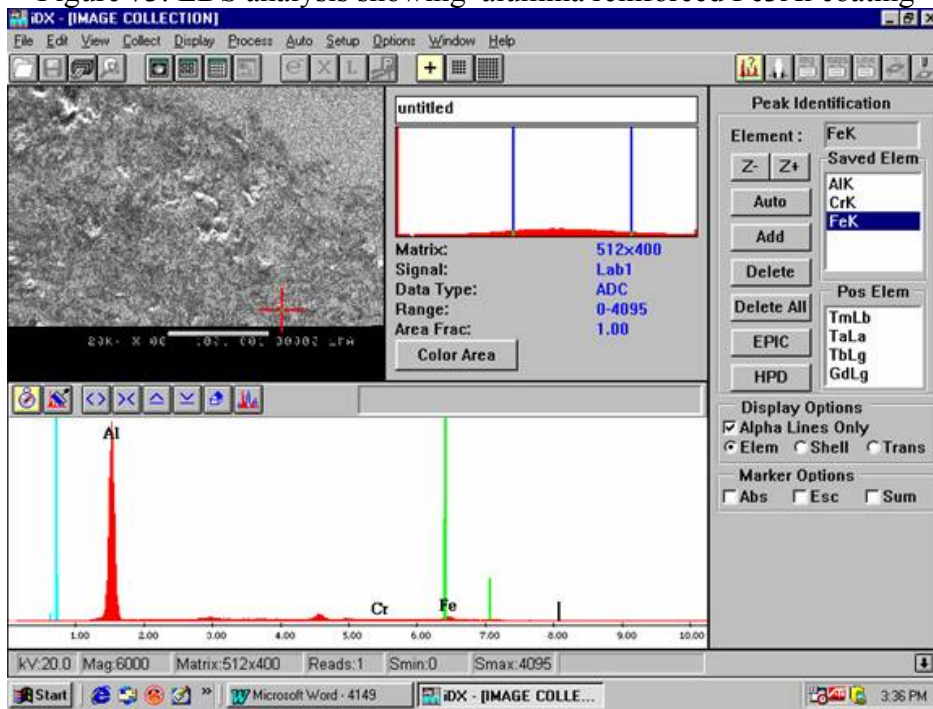


Figure 74: EDS analysis of alumina particles in Fe₃Al- after oxidation

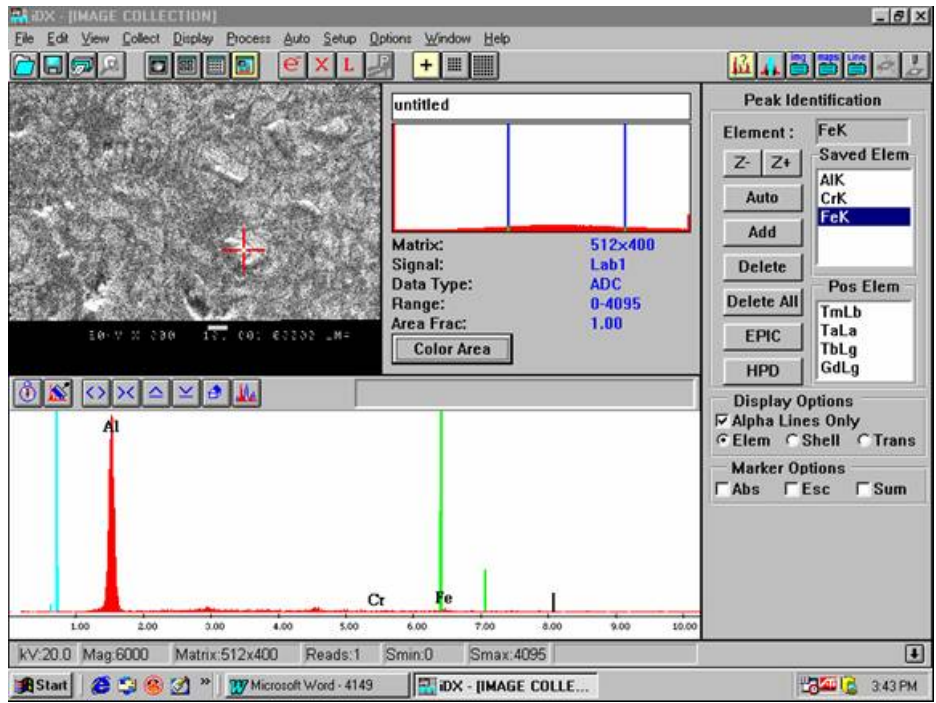


Figure 75: EDS Analysis showing that alumina particles are nicely bonded with iron aluminide matrix even after cyclic oxidation test

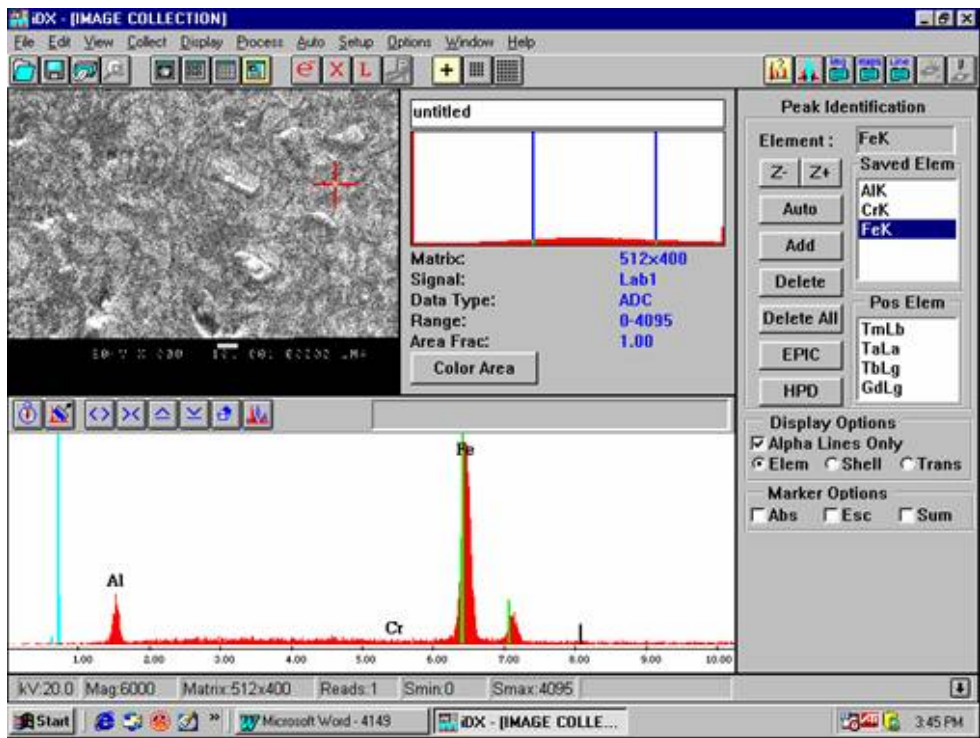


Figure 76: EDS analysis of Fe₃Al matrix area of alumina reinforced Fe₃Al coating

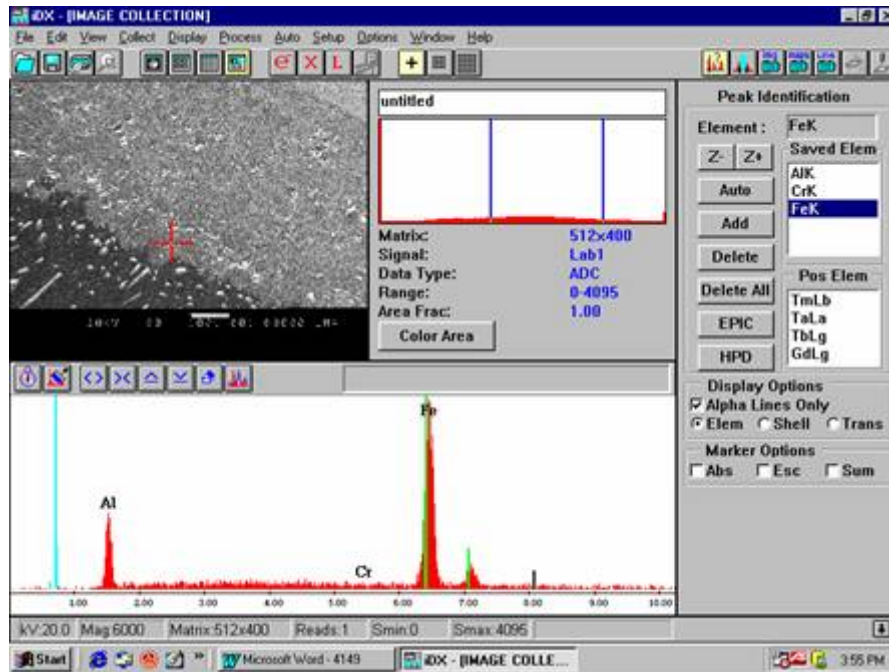


Figure 77: EDS analysis of alumina reinforced iron aluminide coating

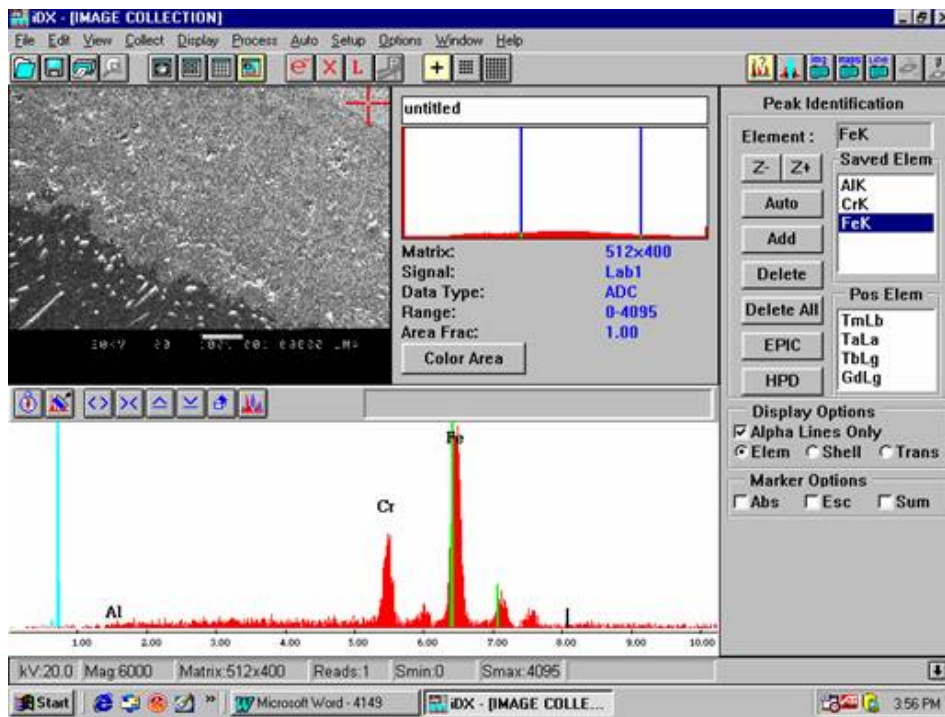


Figure 78: Show coating was not delaminated after cyclic oxidation. Alumina reinforced iron aluminide protected underneath substrate . Here EDS analysis showing the analysis of 304 H substrate

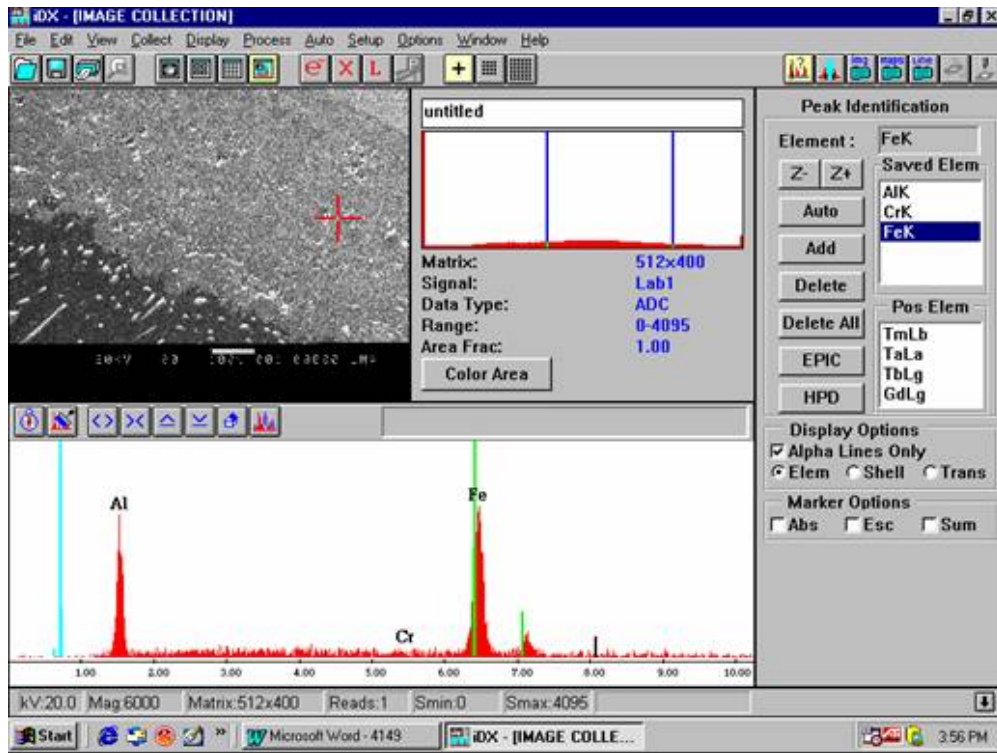


Figure 79: EDS analysis Showing Iron aluminide coating after cyclic oxidation

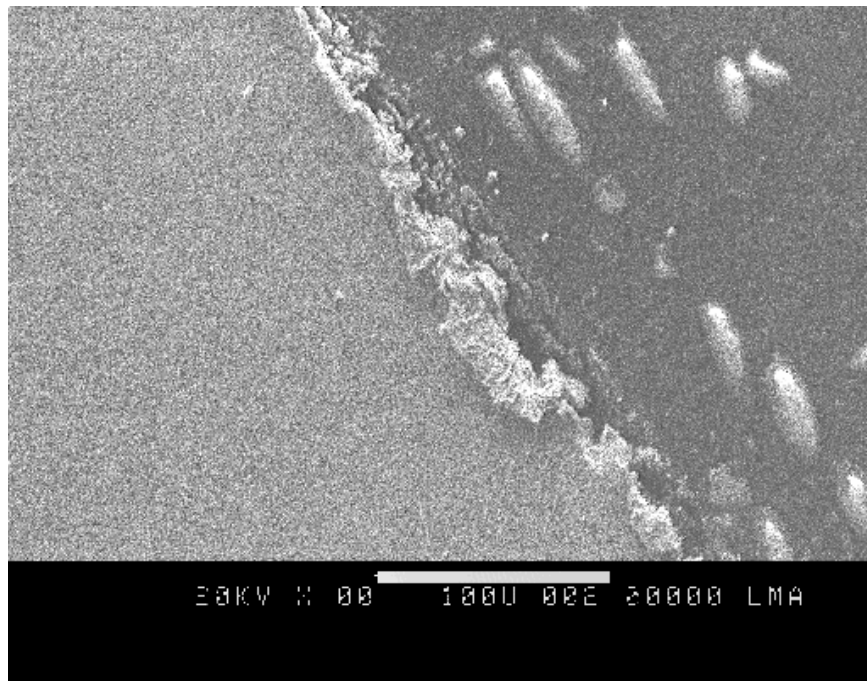


Figure 80: Uncoated 304 H after cyclic oxidation test show oxide layers falling off while polishing X350

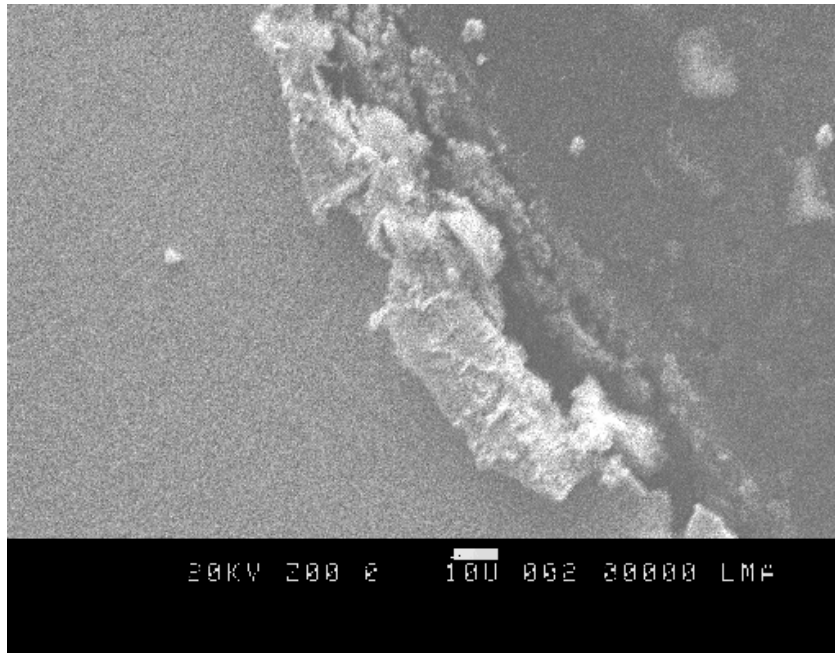


Figure 81 : Showing uncoated 304 H after oxidation – Oxide layer spalling off and substrate was attacked by this cyclic oxidation- show oxidation X700

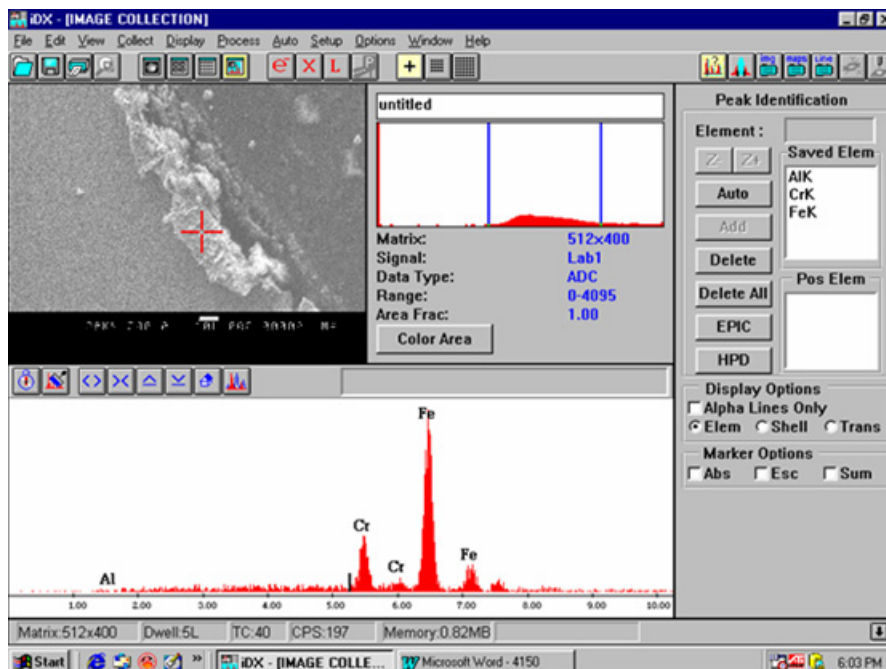


Figure 82: EDS analysis represent the layer is comprised of oxides of iron and chromium

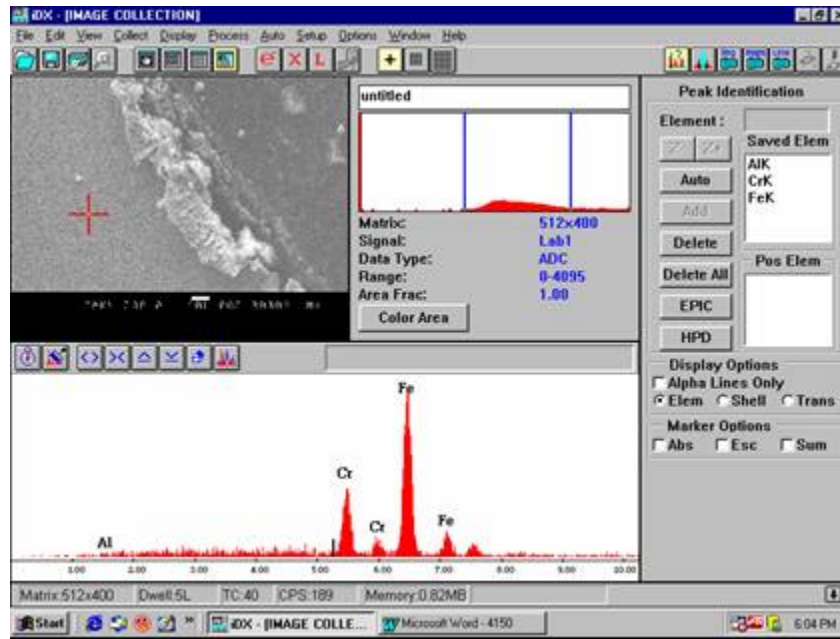


Figure 83: EDS analysis of 304 H substrate after cyclic Oxidation test

Table 17: Oxidation Test Results of Fe₃Al and Alumina Reinforced Fe₃Al Coating on 304H and IN 718 Substrate

Sample	Type of coating	Weight Gain (mg/cm ²)
Fe ₃ Al on 304 H	Plasma Spray	1.03
Alumina reinforced Fe ₃ Al on 304 H	Plasma Spray	0.93
Fe ₃ Al on 718	Plasma Spray	1.01
Alumina reinforced Fe ₃ Al on 718	Plasma Spray	0.92
Fe ₃ Al on 304 H	HVOF	0.989
Alumina reinforced Fe ₃ Al on 304 H	HVOF	0.972
Fe ₃ Al on 718	HVOF	0.985
Alumina reinforced Fe ₃ Al on 718	HVOF	0.968
Uncoated 718	No Coating	1.95
Uncoated 304 H	No Coating	2.15
Medium Carbon Steel	No coating	3.55

From the SEM microscopy and oxidation test results it was concluded that uncoated steel, IN 718 and 304 H were severely attacked by this cyclic oxidation, But Fe₃Al and alumina reinforced Fe₃Al coating sprayed on IN 718 and 304H protected the 304H and IN 718 substrate. Coatings were adherent to substrate, did not spall off during and after oxidation test. It was also found that oxidation rate of alumina reinforced iron aluminide coating was lower than the iron aluminide coating sprayed on both the substrate and oxidation rate of plasma spray coating was slightly higher than the HVOF coating. But once again both coating showed oxidation resistant properties and protected the substrate.

4.0 CONCLUSIONS:

This I&I program developed CVD iron, aluminum, aluminum oxide coated iron powders and iron, aluminum coated alumina powders for fabricating Oxide Dispersion Strengthened Iron aluminide and alumina particle reinforced iron aluminide composites and their thermal spray coating for future power generation equipment.

This program demonstrated ODS iron aluminide and alumina reinforced iron aluminide composites by CIP/sinter and CIP/extrusion route with good mechanical properties. Mechanical properties could be better with further investigation of few more consolidation steps after extrusion. But in this study composite development by CIP/extrusion and their room temperature property improvement was not pursued further in detail. More focus was given on high temperature oxidation, carburization and corrosion resistant thermal sprayed coating and their microstructure development .

Composite's mechanical property results yielded mechanical properties nearly equivalent to MA754, MA758 MA956, and even mechanical strength is superior to MA956 in the case of ODS iron aluminide, with nearly double the properties of current SOTA P/M 410L ferritic steel materials.

This program clearly demonstrated the potential of the Vapor Phase Synthesis of Nanoengineered Powders (VPSNP) process for enabling improved dispersion strengthened materials by CIP/extrusion route and iron aluminide coating by thermal spray (Plasma spray and HVOF) to be produced at reduced costs and improved performance, compared to current iron aluminide or MA956 SOTA materials. In case of thermal spray coating from the results, the potential of thermal sprayed (Plasma Spray and HVOF) Fe₃Al and alumina reinforced Fe₃Al coatings to control oxidation, carburization and corrosion environments such as for the future coal fired power generation component is clear. The following conclusions can be drawn:

- 1) Thermal spray coatings do not catastrophically fail in high oxidizing and corrosion environments
- 2) Aluminum, iron, aluminum oxide, coated iron powders and aluminum, iron, coated alumina is sprayable using High Velocity Oxy-Fuel (HVOF) and Plasma Spray Process with excellent adhesion to 304 H and Inconel 718 series substrates.

- 3) Powdermet's Fe₃Al and alumina reinforced Fe₃Al coating's hardness is comparable to that of hardened 304 H stainless steel.
- 4) Thermally sprayed Fe₃Al and alumina reinforced Fe₃Al exhibit no catastrophic corrosion failure in the presence of Fe₂O₃/K₂SO₄ and Na₂SO₄.
- 5) HVOF and plasma sprayed coatings exhibit no delamination or other catastrophic failure during oxidation, conditions.
- 6) Plasma spray and HVOF iron aluminide coating exhibits low erosion rates against alumina grit erosion test
- 7) A problem encountered with plasma spraying submicron micro-engineered powders is flowability within the plasma and HVOF systems. In particular problem was severe incase of HVOF coating of Fe₃Al. Additional work on granulation for increasing powder flow is required to increase deposition efficiency rates and to optimize deposition parameters further.

In summary, this study clearly demonstrated that thermally sprayed iron aluminide and alumina reinforced iron aluminide coatings can survive under oxidation, carburization and can provide improvements in corrosion condition. It is also expected that CIP/sintered and CIP/extrude/roll route of Fe, Al coated iron and alumina powders will be applicable for future power generation equipment and will be further studied in future for further development. While the results of these initial iron aluminide property measurements are encouraging, but more measurements of iron aluminide properties are needed with the execution of a broader test matrix such as FeAl, particularly for establishing properties for future use. For composite detail characterization of high temperature tensile, creep, fatigue test and for coatings simulated NO_x atmosphere test, more severe steam oxidation test, and hot corrosion test and mechanical properties are needed. The recommended follow-on plan for further optimization and maturation of this technology (composite and coating fabrication) will require the conduct of further optimization of a broader test matrix iron aluminide composite/coating properties, the development of processing for high tensile, ductile ODS iron aluminide, the fabrication of near-net-shaped components, tube fabrication, along with viable methods for their joining to, and integration with the associated boiler structures. Another continuation program should focus on scale up development considerations in CIP/extrude/hot rolling/warm or cold rolling route and thermal spray coating development in detail. The main object of the later follow-on investigation is to move from batch process to a factory-wise continuous-reactor system implementing a candidate technology, namely the CVD powder coating for fabrication of iron aluminide composite fabrication and thermal coating development for future power generation equipment

5.0: RECOMMENDATIONS

So the processing optimization in this I&I program took the pathway proposed this program which the factory-processing-adaptable, scale-up trials using the

CIP/extrusion/rolling process received only limited attention, before it was abandoned due to poor room temperature properties, breaking of samples while machining and the need for room temperature property modifications, and further out-of-scope consolidation development, and financial need. The CIP/sinter process trials had to be sacrificed (at the program outset) for the more critical processing-microstructure-property optimization effort and the more readily available thermal spray method using this powder. Also the foregoing I &I Program executions made it more apparent that alumina reinforced iron aluminide coating would be potentially more viable for use as an integral part of future boiler components for retrofitting existing equipment. With such generic considerations in mind the following specific recommendations can be made in the wake of the current Program completion:

- (1) In view of the encouraging property trade study results obtained so far in this investigation, a viable follow-up plan for further optimization and maturation of this technology will require the conduct of a further Phase- I SBIR Program for further optimization of the iron aluminide properties, and another Phase II focusing on scale up development considerations. The main object of these follow-on investigations is to evolve to improve room temperature ductility factory-wise continuous-reactor process using the CVD coated powders and thus successfully replace the current batch processing methods. The execution of these Phases will be required before proceeding to a Phase III – Technology Transition.
- (2) The processing optimization follow-on plan should also focus not only on further enhancement of the intrinsic properties of the iron aluminide itself, but also to develop the technology needed for fabrication of useful power generation component /joining by this iron aluminide to replace current expensive nickel base alloys . This phase of work should also consider a broader iron aluminide characterization test matrix at the component test level.
- (3) Follow-on work activities should also consider the development and fabrication of near-net-shaped extruded /rolled or forged elements, and the methods of their attachment to, and integration with the associated power generation such as boiler equipment structures.