



## FeAl and FeCrAl as Alternative Coatings for NiAl

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### ABSTRACT

As an economic alternative to NiAl, Fe-based alloys have potential application as materials for the intermediate coatings in the fabrication of plasma-sprayed ceramic coatings, usually  $\text{Al}_2\text{O}_3$  or  $\text{Cr}_2\text{O}_3$ , on metallic substrates. These ceramic coatings are applied to various industrial components to improve wear resistance. The purpose of the intermediate coating is to improve the adhesion between the ceramic coating and the metallic substrate both during the plasma spraying process and during subsequent service. The intermediate coating becomes indispensable when the application requires the ceramic coating to withstand high temperatures. At which, both thermal stress and high temperature corrosion can push the intermediate coating to its limit. Ni-based alloys have proven to be one of the most reliable intermediate coatings in such circumstances where high temperature is involved. However, the rising price of Ni limits its usage in many applications. This paper thus compares the effect of exposure at  $400^\circ\text{C}$  on the adhesion of the Fe-base and Ni-base intermediate coatings. The materials studied are FeAl, FeCrAl and NiAl arc sprayed coatings on stainless steel 304 substrates. All three coatings were top-coated with a plasma-sprayed  $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  coating which is a standard coating used in tribological applications. The work focuses on oxidation testing of the coatings at  $400^\circ\text{C}$ . The oxidation behavior of the three coatings is compared and discussed.

**Keywords:** FeAl, FeCrAl, NiAl, bond coated, oxide

### 1. INTRODUCTION

Thermal sprayed ceramic coatings such as  $\text{Al}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$ , can be used to reduce wear problems in mechanical parts. Ceramic coatings on metallic substrates are subjected to both mechanical and thermal stresses. An intermediate coating is necessary in order to improve the adhesion between the ceramic topcoat and the metal substrate. NiAl is widely used as

the intermediate bondcoat since it also demonstrates excellent oxidation resistance and corrosion resistance at high temperature [1]. However, the rising price of Ni limits its usage in many industrial applications. Fe-based material is a possible alternative for the bondcoat application due to its good adhesion to Fe-based substrates which are encountered in many industrial

components, e.g. steels. FeAl has useful oxidation resistance and high temperature corrosion resistance [2] due to the formation of a protective  $\text{Al}_2\text{O}_3$  layer which is stable at high temperature. Increasing the Al content improves oxidation resistance but makes FeAl alloys more brittle. Other elements such as Cr can be used to improve the oxidation and corrosion resistance of the alloy without the need to increase the Al content [3]. FeCrAl has good oxidation resistance at high temperatures [4-6] and may be considered for use as a bondcoat for high temperature applications.

This paper investigates the microstructure and the adhesion strength of the Fe-based and Ni-based intermediate coatings. In this work the electric arc spraying technique was used to produce three bondcoats which were covered with an  $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  plasma sprayed topcoat. The coated specimens were subjected to tests at 400°C for 6, 24, 48 and 96 hours to study oxidation behavior in relation to the adhesive strengths of the coatings.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Stainless steel grade 304 (72.5 Fe-18.5 Cr-9Ni) coupons of 25.4 mm. diameter and 5 mm. thick were roughened by grit blasting using a 480  $\mu\text{m}$  white alumina grit to obtain rough surfaces of approximately 7  $\mu\text{m}$  Ra. The specimens were then ready for electric arc spraying (EAS) using a TAFA 9,000 system to produce Fe5.5Al, Fe20.9Cr4.8Al and Ni5Al (%wt) bondcoats of approximately 150-200  $\mu\text{m}$  thick. The bondcoat spraying parameters are shown in Table 1. Each of the three bondcoats was top coated with plasma sprayed  $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  of 300-400  $\mu\text{m}$  thickness. The  $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  coating parameters are shown in Table 2.

### 2.2 Oxidation testing

The specimens were separated into 3 groups depending on the type of the bondcoat. Group F is FeAl/ $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$ , group C is FeCrAl/ $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  and group N is NiAl/ $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$ . Samples from each group of specimens

**Table 1.** Bond coating parameters.

Air pressure (bar)	4
Arc load (V)	33
Arc current (A)	250
Distance (mm.)	152

**Table 2.**  $\text{Al}_2\text{O}_3$ -40% $\text{TiO}_2$  coating parameter.

Ar pressure (bar)	6.9
H2 pressure (bar)	3.4
Ar flow (L.P.M)	47
H2 flow (L.P.M)	7
Load (V)	70
Current (A)	500
Powder feed rate (g/min)	27
Distance (mm.)	89

were subjected to the oxidation tests for 6, 24, 48 and 96 hours in an open tube furnace at 400°C with no additional gas flow. The temperature of 400°C is used to represent the localized temperature a coated

component may experience as a result of frictional heating during operation at room temperature. Sample designations are shown in Table 3.

**Table 3.** Specimen identification for oxidation tests.

Specimen group	Substrate	Bondcoat	Topcoat	Oxidation time at 400°C (hour)
F0	stainless steel	FeAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	0 (as-sprayed)
F6	stainless steel	FeAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	6
F24	stainless steel	FeAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	24
F48	stainless steel	FeAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	48
F96	stainless steel	FeAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	96
C0	stainless steel	FeCrAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	0 (as-sprayed)
C6	stainless steel	FeCrAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	6
C24	stainless steel	FeCrAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	24
C48	stainless steel	FeCrAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	48
C96	stainless steel	FeCrAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	96
N0	stainless steel	NiAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	0 (as-sprayed)
N6	stainless steel	NiAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	6
N24	stainless steel	NiAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	24
N48	stainless steel	NiAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	48
N96	stainless steel	NiAl	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	96

### 2.3 Microstructural study

The specimens were cross-sectioned, using a precision saw with low feeding rate (0.08 mm/s) to prevent any damage to the specimen during preparation, and polished to reveal the coating microstructures. A JEOL JSM5401 scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) and RIGAKU TT RAX III X-ray diffractometer were used to characterize the structures and phase compositions of the coatings. The percentage of oxide present in each case was determined using image analysis (Image Pro Plus version5.1) as an average

value from 10 random positions across the SEM images of the coating cross-section.

### 2.4 Adhesive tensile strength

Pull-off testing, according to ASTM C633-79, was used to evaluate the adhesive strength of the coating. Ten specimens per group were tested. The coated flat surface on a cylindrical sample was covered with Klebbi glue and bonded onto another flat surface on an uncoated cylinder. The glue was cured at 200°C for 2 hours to strengthen the bonding. The specimens were then loaded into an Instron 8801 Universal Testing Machine for tensile

testing using 10,000 kg load and 1 mm./minute pulling rate.

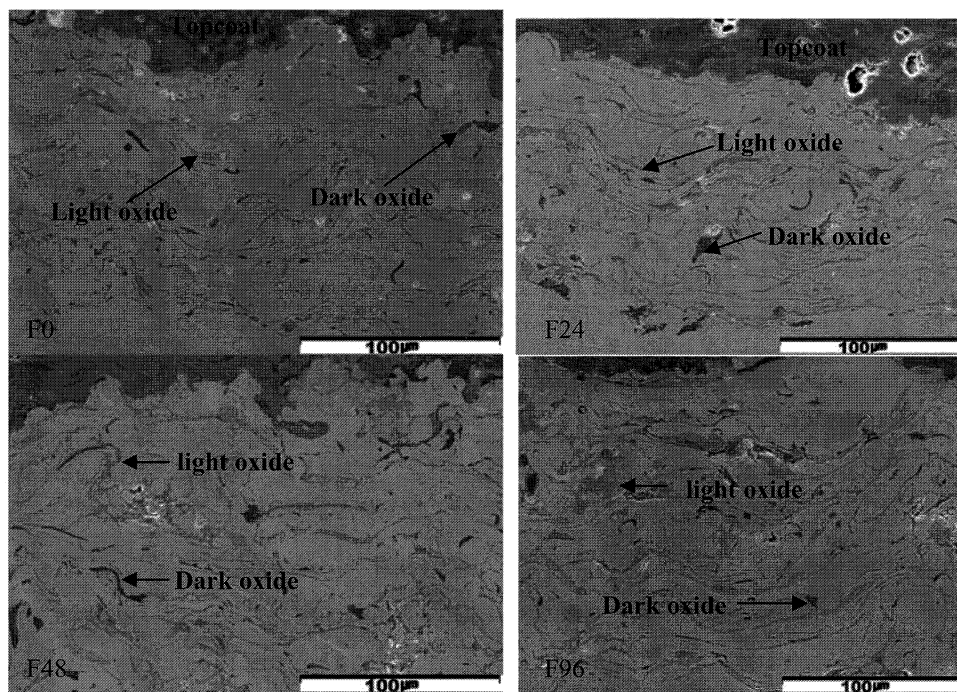
### 3. RESULTS AND DISCUSSION

#### 3.1 Microstructure Characterisation

Figures 1, 2 and 3 show the cross-sectioned micrographs of FeAl, FeCrAl and NiAl specimens respectively, after oxidation at 400°C for various times. There are two distinctive oxides in the microstructures; dark contrast oxide and light contrast oxide. The as-sprayed specimens generally contain the lowest amount of oxide compared to the other specimens. The oxide in this coating was formed during the coating process. EAS coating involves two current carrying, electrically conductive wires fed into a common arc point, creating a temperature of up to the melting point of the wire material at which melting occurs. The

temperature at the wire tips can be as high as 1,500°C [7]. The molten material is continuously atomized and the droplets are accelerated toward the substrate by a compressed air jet. Oxidation occurs while the droplets are in contact with the air jet causing the presence of oxides in the as-sprayed structure. After the oxidation tests at 400°C, the quantities of the oxides are increased with increase in the testing time.

Average percentages of oxide in each bond coat are shown in tables 4, 5 and 6 respectively. The amount of the dark oxide does not increase significantly with the test time in comparison with the light oxide. The light oxide grows significantly with increase in the testing time, resulting in an overall increase in the total amounts of oxide with the testing time, with the exception of the NiAl coating.



**Figure 1.** The cross-sectioned images of FeAl specimens after oxidation tests at 400°C for 0, 24, 48 and 96 hours as shown in F0, F24, F48 and F96 respectively.

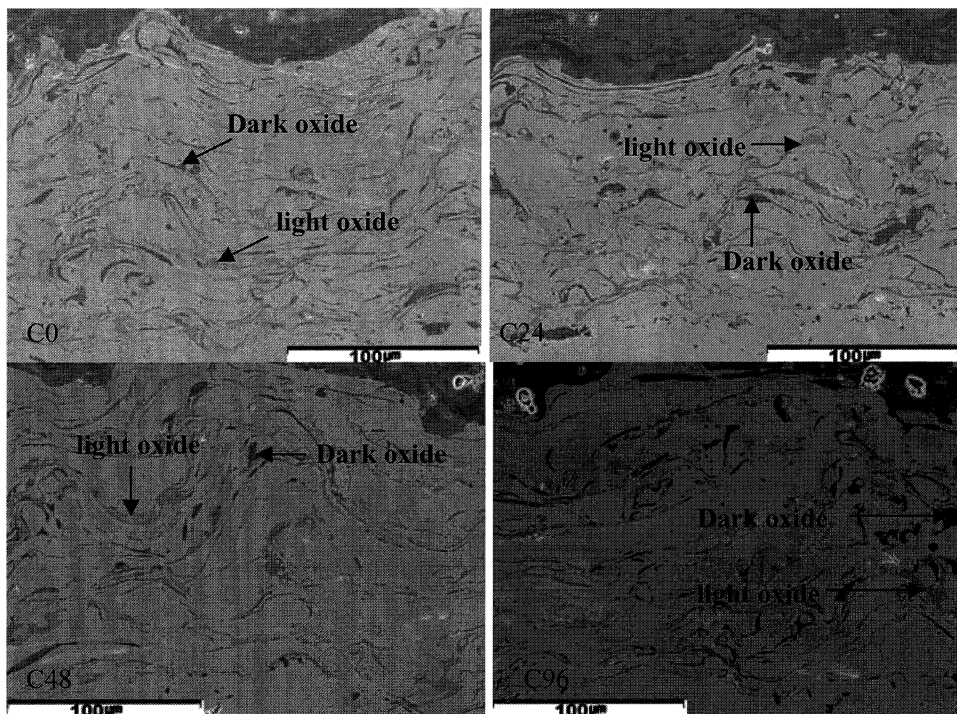


Figure 2. The cross-sectioned images of FeCrAl specimens after oxidation tests at 400°C for 0, 24, 48 and 96 hours as shown in C0, C24, C48 and C96 respectively.

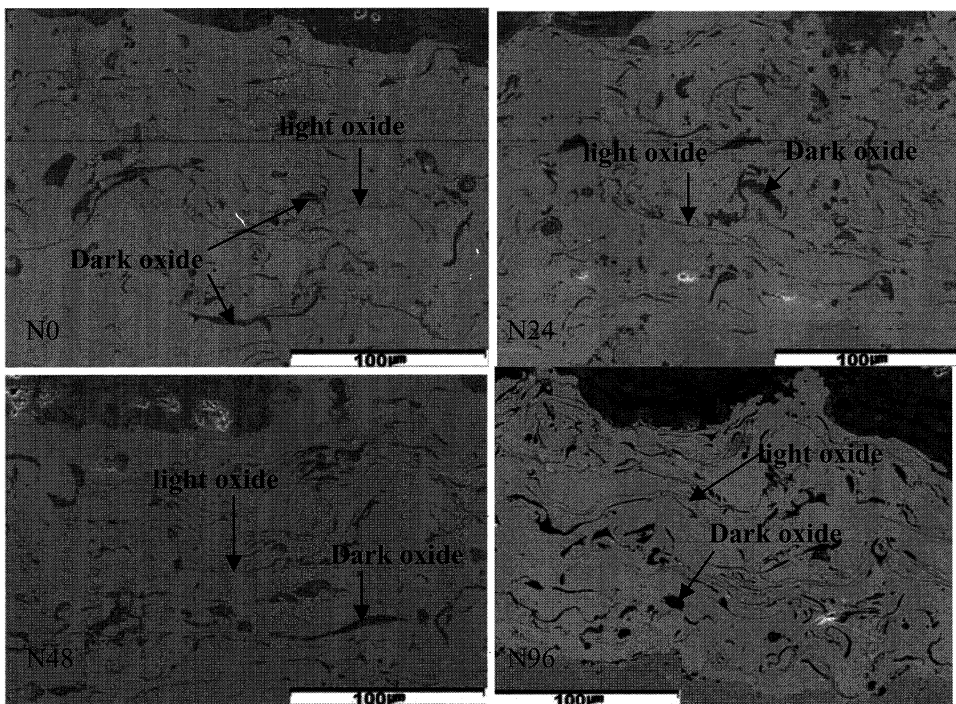


Figure 3. The cross-sectioned images of NiAl specimens after oxidation tests at 400°C for 0, 24, 48 and 96 hours as shown in N0, N24, N48 and N96 respectively.

**Table 4.** Average oxide percentage on FeAl coating.

Specimen	Dark oxide (%area)	Light oxide (%area)	Total oxide (%area)
F0	2.85	7.35	10.20
F6	2.46	13.04	15.50
F24	2.51	13.64	16.15
F48	2.68	16.36	19.04
F96	2.83	19.00	21.83

**Table 5.** Average oxide percentage on FeCrAl coating.

Specimen	Dark oxide (%area)	Light oxide (%area)	Total oxide (%area)
C0	2.65	7.37	10.02
C6	2.79	7.70	10.49
C24	3.70	13.93	17.63
C48	3.25	14.82	18.07
C96	4.28	15.53	19.81

**Table 6.** Average oxide percentage on NiAl coating.

Specimen	Dark oxide (%area)	Light oxide (%area)	Total oxide (%area)
N0	6.19	4.03	10.21
N6	6.92	4.24	11.15
N24	7.65	4.40	12.05
N48	7.33	4.87	12.21
N96	7.76	4.99	12.75

Table 7, 8 and 9 show the chemical compositions of the oxides with the O data omitted due to its inaccuracy. In all three coatings the major component of the dark contrast oxide is Al suggesting that the dark contrast oxide is  $Al_2O_3$ , which is a slow-growing oxide. The amount of the dark oxide does not increase significantly with the test time since the test temperature at  $400^\circ C$  is not high enough for significant formation of  $Al_2O_3$ .

Chemical compositions for the FeAl alloy coating are shown in Table 7. The light oxide is an iron-rich oxide which grows with increase in the testing time, resulting in an overall increase in the total amounts of oxide with the testing time.

Table 8 shows that the light oxide of FeCrAl coating is (Fe, Cr, Al) oxide.

Protective  $Cr_2O_3$  can form at a lower temperature than  $Al_2O_3$ . At  $400^\circ C$ , the formation of  $Cr_2O_3$  requires a lower partial pressure of  $O_2$  than the formation of FeO or  $Fe_3O_4$  according to Gibb's free energy of oxide formation [8]. However,  $400^\circ C$  is still quite low to drive the formation of  $Cr_2O_3$ . Thus a large amount of Fe-based oxide is still formed with some oxide spinel of Fe, Cr, and Al.

Table 9 shows that the light oxide in the NiAl coating is a Ni-rich oxide. The amount of oxide does not increase much during the oxidation test due to its slow growth at  $400^\circ C$ . The rate of increase of the oxide is lower than those of the Fe-base coatings. The lower rate of oxidation imposes lower stress on the coating.

**Table 7.** Chemical composition of oxide on FeAl alloy coating.

	Fe (%wt)	Al (%wt)
Coating based material	95.5	4.5
Dark oxide	32.7	67.3
Light oxide	82.32	17.68

**Table 8.** Chemical composition of the FeCrAl coating.

	Fe (%wt)	Cr (%wt)	Al (%wt)
Coating based material	77.04	20.12	2.84
Dark oxide	16.46	9.40	74.14
Light oxide	66.18	17.39	16.43

**Table 9.** Chemical composition of NiAl coating.

	Ni (%wt)	Al (%wt)
Coating based material	96.67	3.33
Dark oxide	31.88	68.12
Light oxide	100	-

XRD scans were performed on the bondcoats only (the topcoats were polished off prior to the analysis). The results on the as-sprayed bondcoats and the bondcoats after oxidation are shown in figure 4. For FeAl coatings, only the Fe peaks are prominently shown in the scan. This is because the original powder contains only 5.5 wt.% of Al, some Al was lost during spraying due to its low melting point. Thus the Al peaks cannot be seen above the background noise. The small amount of oxide also could not be detected by XRD. FeCrAl coatings exhibit Cr and  $\text{CrFe}_4$  peaks in addition to the Fe peaks. Oxide is also not detected. Ni peaks were found in the NiAl bondcoat, but Ni oxide was not detected in the coating. In all three coatings, there is no significant change that can be observed using the XRD analysis before and after the oxidation tests.

SEM line scanning at the interface of the bondcoat and the substrate after 96 hours of oxidation testing is shown in figure 5. A broken line indicates the interface between the bondcoat and the

substrate. The bondcoat is shown in the area above the broken line and the substrate is shown below the broken line. Fe, Cr and Ni, which are the major elements of the stainless steel 304 substrate, were found below the broken line.

Elemental diffusion is not evident in FeAl and FeCrAl coatings due to the similarity of the chemical composition between the bondcoat and the substrate. Figure 5(c) however indicates limited diffusion of Ni into the substrate.

### 3.2 Adhesive tensile strength of the coating

The C-633 adhesive tensile test measures the weakest bond across the thickness of the coating. Figure 6 shows that the average tensile strength of Klebbi glue is 73 MPa, which is well above the average tensile strengths of the bondcoat/substrate of the samples in this study. Thus, the glue strength does not affect the results. All coatings tested failed at the bondcoat/substrate interface, indicating that the values relate to the

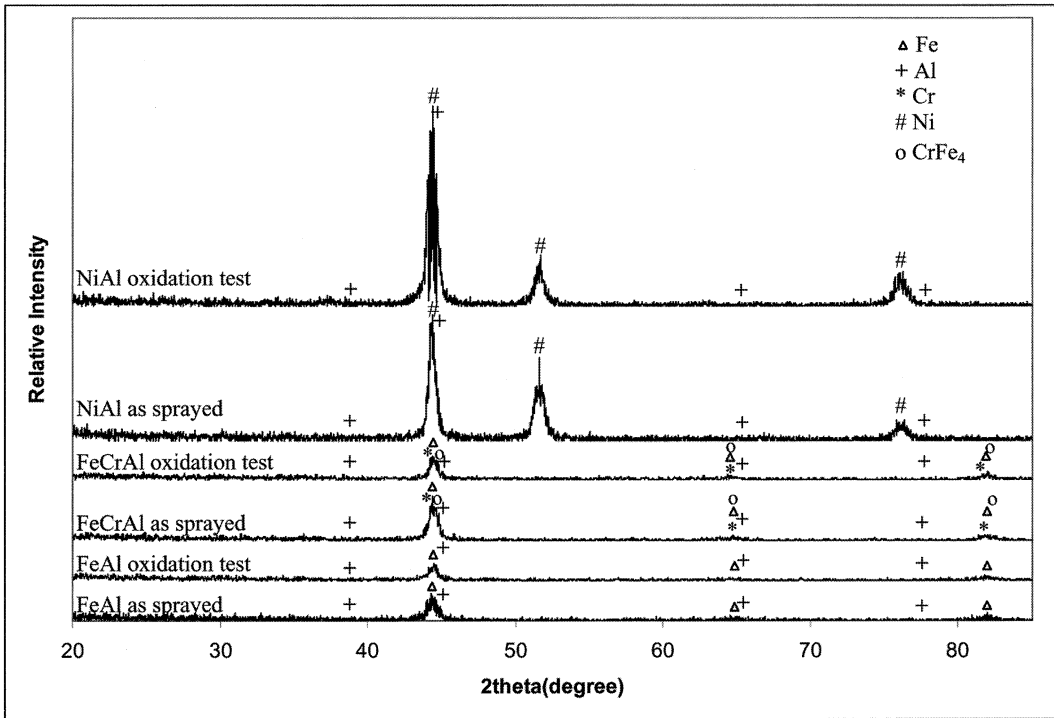


Figure 4. XRD images of the as-sprayed bondcoats and the bondcoats after oxidation tests at 400°C for 96 hours.

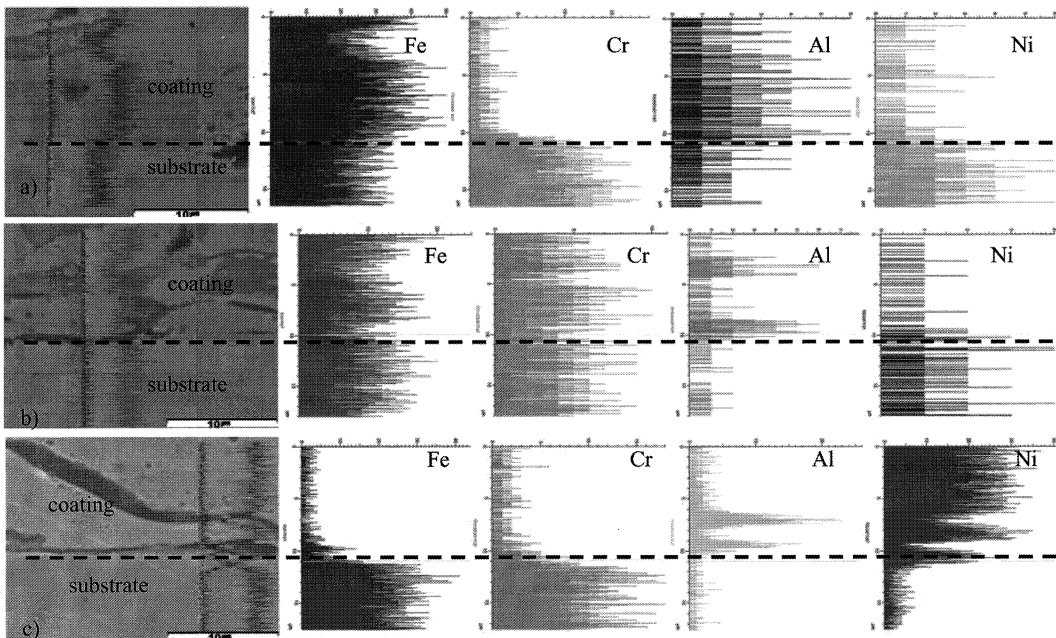
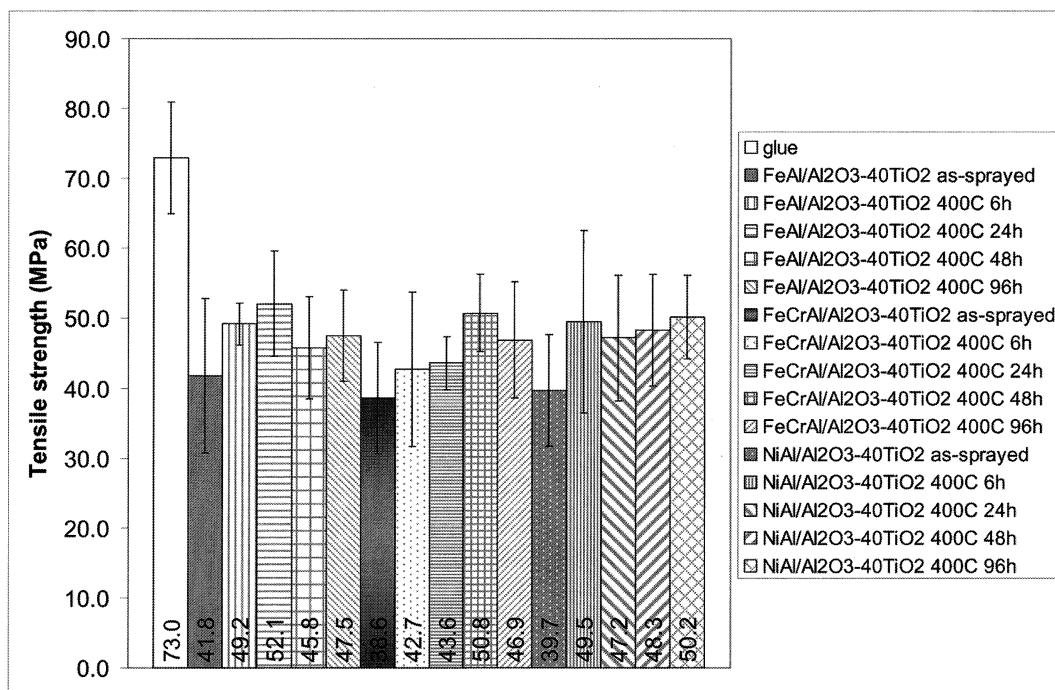


Figure 5. Line scanning at the interface between bondcoat and stainless steel substrate after oxidation tests at 400°C for 96 hours for a) FeAl, b) FeCrAl and c) NiAl coatings.



**Figure 6.** Tensile strength of FeAl, FeCrAl and NiAl bondcoats with Al<sub>2</sub>O<sub>3</sub>-40%TiO<sub>2</sub> topcoat.

bondcoat adhesion strength to the substrate. In the as sprayed state, the adhesion strength of FeAl bondcoat is 41.8 MPa, that of FeCrAl is 38.6 MPa and that of NiAl is 39.7 MPa. After the oxidation tests the tensile strength shows a slight increasing trend with the testing time. However, due to a large fluctuation of the test values, the trend is not significant. The Oxidation tests for 6, 24, 48 and 96 hours result in the average tensile strengths of 49.2, 52.1, 45.8 and 47.5 MPa for FeAl bondcoat, 42.7, 43.6, 50.8 and 46.9 MPa for FeCrAl bondcoat and 49.5, 47.2, 48.3 and 50.2 MPa for NiAl bondcoat, respectively.

The adhesion strengths of the three groups of sample are comparable at each test duration. This work shows that FeAl and FeCrAl can be used as alternative bondcoats to NiAl for service applications below 400°C. However, long term testing should be conducted to study the effect of

the oxide growth on the mechanical properties of the Fe-based coatings.

#### 4. CONCLUSIONS

- Cr in FeCrAl arc sprayed coating can reduce the oxidation rate of the alloy by reducing the formation of Fe-rich oxide.
- The rate of oxidation is lower in NiAl coating in comparison to the Fe-based coatings.
- FeAl and FeCrAl can be used to replace NiAl as a bondcoat for Al<sub>2</sub>O<sub>3</sub>-40%TiO<sub>2</sub> plasma-sprayed coating for applications below 400°C.

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