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A Study of Ni-5wt.% Al Coatings Produced from Different Feedstock Powder

J. Svantesson and J. Wigren

Ten different Ni-5wt.%Al powders—three clad, one sintered, three water atomized, and three gas atomized—have been evaluated and plasma sprayed. The study focused on how manufacturing method, chemical composition, and particle size distribution of the powders affect the quality of the coating. Properties such as microstructure and mechanical behavior, as well as oxidation and corrosion resistance, are discussed. In conclusion, recommendations concerning the selection of powders for different applications are presented.

1. Introduction

NICKEL-5wt.% aluminum powders are used in gas turbine engines for plasma spraying of bond coats in cold to medium-hot sections of engines. Common applications are for abradable coatings in fan and compressor casings, as well as knife edge seals with an alumina top coat for clearance control. The powders used in this investigation fulfill the specification requirements from most of the major aircraft engine manufacturing companies.

2. Experiments

Ten Ni-5wt.%Al powders that are available for plasma spraying were examined (Table 1). Each powder in the various manufacturing technique categories represents a different supplier. In the following discussion, the powders will only be referred to by the number code (1 to 10) given in Table 1. No proprietary tradenames are available.

Morphologies and cross sections significantly differ as a result of the manufacturing methods (Fig. 1). The clad and gas-atomized powders are sphere-like, whereas the water-atomized powders are irregular in morphology. All powders, except for the clad ones, are fully alloyed. The Hall flow and apparent density tests, performed in accordance with ASTM B213[1] and 212,[2] respectively, underline these different characteristics (Table 1). Clad, sintered, and gas-atomized powders exhibit similar behavior due to their rounded appearance, whereas water-atomized powders have significantly higher Hall flow and lower apparent densities due to irregular shapes. A greater Hall flow time indicates that the material does not have good rheological properties.

Phase analysis performed by X-ray diffraction confirmed results previously presented.[3,4] All powder groups contain αNi, which will dissolve up to 5% of Al.[5] The clad powders also contain free Al and the sintered powder Ni_3Al (Table 1).

Chemical analysis was performed on fused glass beads by X-ray spectrometry (Ni, Al). Trace elements were detected by an infrared absorption method (O, C, S), thermal conductivity (N), and photometry (B, Si) (Table 2). The differences in nickel and aluminum contents were not important for the coating properties described in this article.

The clad powders contain a high amount of oxygen, carbon, and sulfur compared to the others, because the binder contributes to the overall composition. Water atomization results in a higher amount of oxygen compared to gas atomization, which is expected because water is oxidizing toward nickel-aluminum alloys. Powder 9 differs from the others because it exhibits a distinctly higher boron content.

Size distribution measurements (Fig. 2) were carried out by a wet Microtrac (Leeds & Northrup) method, except for Powder 1 which required dry analysis due to its water-soluble binder. No correlations between the determined size distributions and coat-

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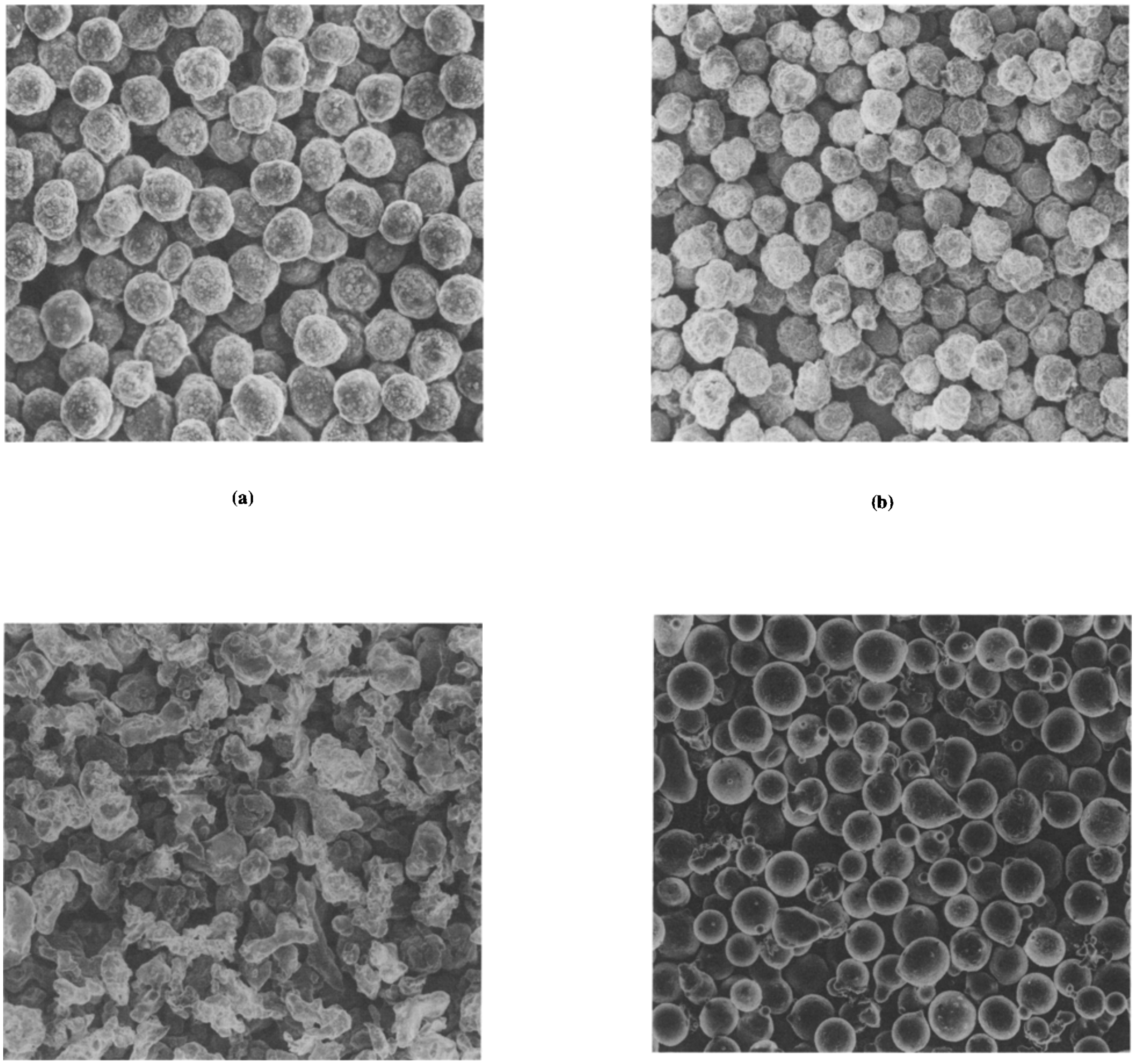
Table 1 Powder Characteristics

Powder No.	Manufacturing technique	Comments	Morphology(a)	Phases αNi	Phases Al	Phases Ni_3Al	Hall flow s	Apparent density, g/cm^3
1	Al clad on Ni	Polyvinylpyrrolidone binder	a					
2-3	Al clad on Ni	Phenolic binder	a	X	X		16.0-17.9	3.7-4.0
4	Sintered	...	b	X		X	17.5	3.9
5-7	Water atomized	...	c	X			26.4-32.4	2.6-2.8
8-10	Gas atomized	Argon	d	X			13.8-15.0	4.3-4.5

(a) See section of Fig. 1 as indicated.



(a) (b)

(c) (d)

Figure 1 Powder appearance. (**a**) Clad. (**b**) Sintered. (**c**) Water atomized. (**d**) Gas atomized.

ing properties were found. A correlation between size distribution and coating adhesion could be expected, but the adhesion seems to be more strongly affected by other powder characteristics, which are discussed below.

All powder systems were sprayed using a Metco EG 88 (ATC) system with a Metco 7MB gun. Round 25-mm buttons and 20- by 100-mm panels of Inconel 718 (nickel-base alloy) were used as the substrate material throughout this investigation.

Spraying parameters were optimized with respect to coating microstructure and adhesion by varying gas flows, spray rates, and spray distances. The gas flows (argon/hydrogen) were selected by testing ranges of 40/7 to 40/16 (with hydrogen being varied) and 30/13 to 60/13 SLPM (with argon being varied) with a total of eight different gas compositions. Additionally, powder flow rates of 25, 50, and 100 g/min were tested. The best adhesion was achieved with gas flows of 40/16 or 30/13 SLPM and a powder flow rate of 25 g/min, but the microstructures were not acceptable due to high amounts of oxides. A final choice of 40/13 SLPM and spray rate of 50 g/min resulted in the most acceptable microstructure, as well as good adhesion.

The spray distance was optimized by spraying at 80, 120, 180, and 250 mm to a thickness of 250 μm. The samples were tested for adhesion according to ASTM C633[6] using FM 1000 epoxy and a gravity bonding technique. Optimal spray distance was found to be 120 mm (Fig. 3). It is notable how little the microstructure was affected by alteration of the spray distance. The final spray parameters are given in Table 3. No significant differences in deposit efficiencies between the ten powders were noted during the spray tests.

3. Results and Discussion

Adhesion test results are averages from three sprayed buttons and are within 3% accuracy. Adhesion was tested on 250- to 280-µm thick coatings (Table 4).[6] The highest adhesion was obtained with Powder 9, possibly due to the high boron content of this powder. Boron is known to influence the properties of bulk materials and some of these effects are described below.

Gessinger[7] describes the effect of boron on lowering the melting point of Astroloy AP1. An increase from 170 to 250 ppm decreases the melting point from 1242 to 1220 °C. Powder 9 contained 2000 ppm compared to Powder 8, which contained 470 ppm, and Powder 6, which contained 290 ppm. The other powders contained less than 10 ppm. A lowered melting point will result in lower coating porosity due to a higher degree of melting of the powder particles.

Boron also improves the ductility and hot working properties of superalloys.[8] Only 15 ppm of boron doubles the rupture life and ductility of Waspaloy. One possibility is that boron increases wettability. It is expected that such materials would exhibit enhanced interlamellar adhesion. Boron has also been suggested as a trace element to prevent oxygen and sulfur from forming brittle films in the grain boundaries of superalloys.[8]

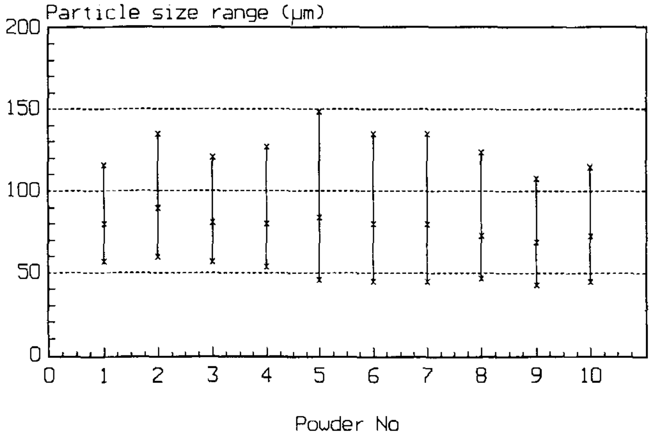


Figure 2 Size distribution of the powders.

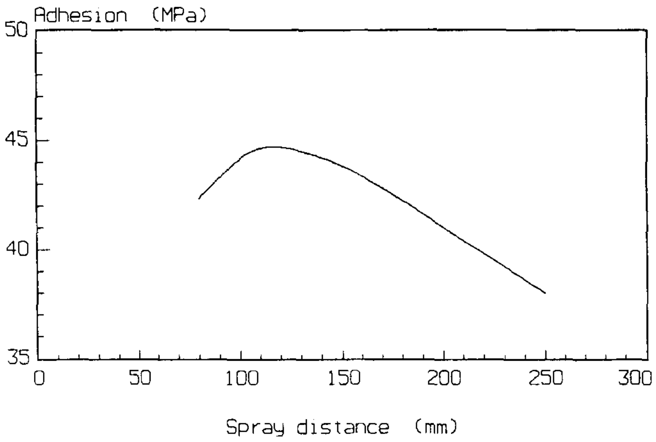


Figure 3 Optimization of spray distance.

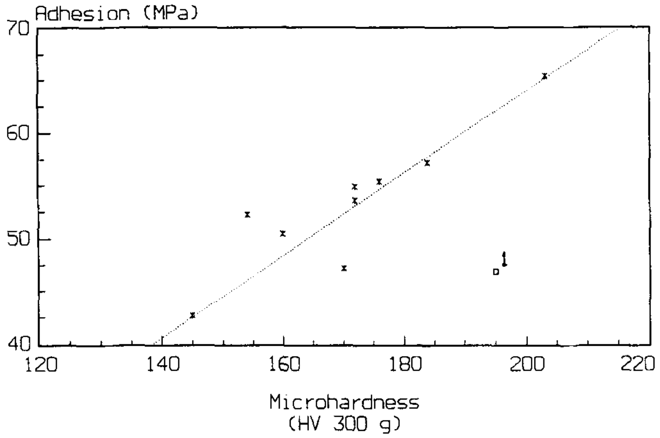
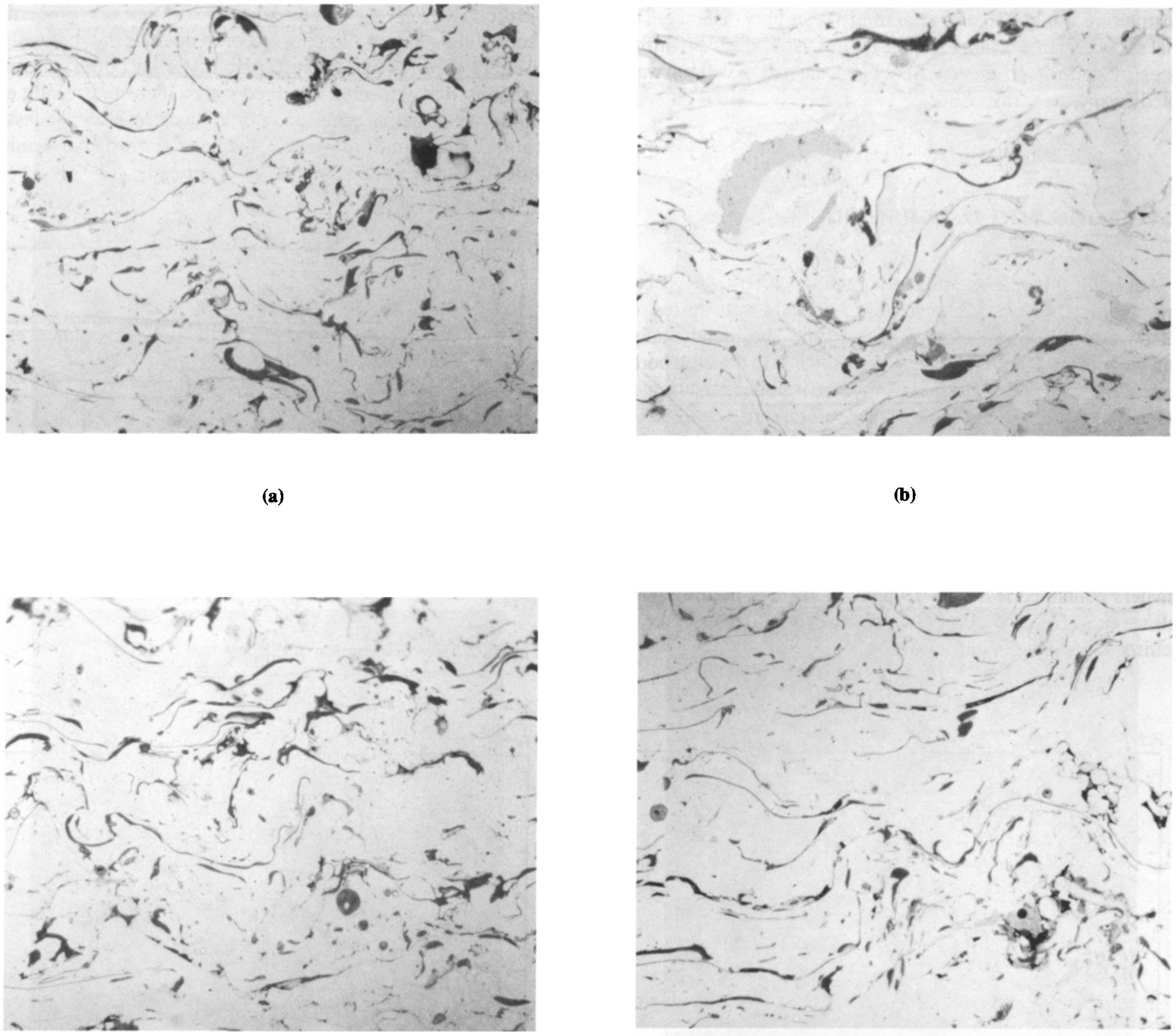


Figure 4 Correlation between adhesion and microhardness. The coating manufactured from the sintered powder is designated 1.

Table 2 Powder Chemical Composition

				Composition, %				
Powder No.	Ni (±0.1)	Al (±0.2)	O (±0.02)	C (±0.02)	S (±0.005)	N (±0.002)	B (±0.005)	Si (±0.005)
1	93.3	4.2	1.48	0.830	0.078	0.190	0.001	0.014
2	94.7	4.0	0.47	0.980	0.024	0.023	0.001	0.009
3	93.3	4.6	0.48	1.150	0.020	0.045	0.001	0.033
4	94.8	4.7	0.07	0.008	0.016	0.004	0.001	0.009
5	94.8	5.2	0.10	0.007	0.001	0.001	0.001	0.029
6	94.2	5.1	0.16	0.036	0.002	0.120	0.029	0.134
7	94.8	4.9	0.19	0.008	0.008	0.089	0.001	0.046
8	94.2	5.1	0.01	0.071	0.001	0.012	0.047	0.107
9	94.3	5.0	0.01	0.030	0.001	0.001	0.200	0.154



(a) (b) (c) (d)

Figure 5 Typical coating microstructures. **(a)** Clad. **(b)** Sintered. **(c)** Water atomized. **(d)** Gas atomized.

Gessinger[7] discussed that addition of boric acid during powder compaction (the CAP process) will combine with metal oxides to give a more active surface and thereby increase the diffusion coefficient for powder consolidation. From the above discussion, it can be expected that there will be a maximum content, above which borides will be detrimental to coatings.

The low adhesion properties of Powder 8 may be attributed to the high silicon content. Silicon is known to be detrimental to hot workability properties.[8] It can be suggested that silicon lowers the bond strength due to the formation of SiC, which increases brittleness. However, a boron addition of up to 0.2% (as seen in Powder 9) will counteract this effect. The high adhesion strength of Powder 6 can probably be attributed to the high silicon content. For water-atomized powders, silicon is intentionally added to take care of the excessive oxygen content, and SiO_2 is formed, which is beneficial for improving adhesion.

Macrohardnesses were measured by HR15N, HR15T, and HR15Y methods, with no significant differences found between coatings manufactured from these powders. The measured hardnesses were 70.9 ± 1.5, 81.7 ± 1.2, and 90.2 ± 0.8, respectively.

There is a correlation between coating microhardness and adhesion, in which higher hardness corresponds to greater adhesion (Table 4 and Fig. 4).

The coating structure varies distinctly between the four categories of powders (Fig. 5). The clad powders show a significant amount of NiO, Al_2O_3, and free Al within an αNi matrix,

Table 3 Final Spray Parameters

Gun	Metco 7MB
Nozzle	GH
Powder port No.	2
Gases	Argon/hydrogen
Flows, SLPM/SLPM	40/13
Current, A	600
Voltage, V	75-80
Carrier gas	Argon
Spray rate, g/min	50
Spray distance, mm	120
Surface speed, m/min	75
Gun movement, mm/rev	6

Table 4 Mechanical Properties of Coatings

Powder No.	Adhesion, MPa	Microhardness, average	HV 300 g range
1	54.8	172	127-196
2	55.3	176	116-206
3	52.2	154	119-195
4	46.8	195	154-232
5	47.1	170	138-199
6	57.1	184	125-250
7	53.5	172	145-205
8	38.4	148	109-169
9	65.3	203	161-233
10	42.6	145	110-185

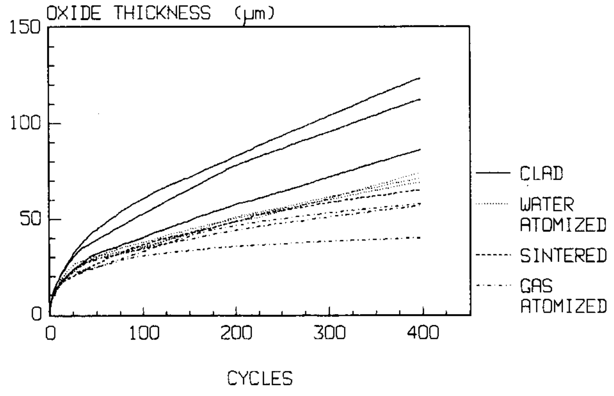


Figure 6 Oxide growth rate at 900 °C.

whereas atomized powders mainly consist of Al_2O_3 in αNi. Sintered powder contains Ni_3Al, NiO, and Al_2O_3 in αNi. Ni_3Al is already present before spraying and is a result of the powder manufacturing technique. The oxides in all coatings are formed during spraying.

Corrosion resistance with respect to chlorides and sulfur may be among the most important properties for this type of coating. Resistance to chloride attack was measured by exposure to a salt spray per ASTM B117[9] for 100 hr. No attack was found either during visual inspection or during cross-sectional examination using light microscopy. Sulfur corrosion resistance was performed in a closed space with saturated SO_2 (liquid) and 95% relative humidity. All coatings were observed to be degraded after 200 hr of exposure.

The clad powder coatings exhibited a white surface, which was found to be aluminum sulfate,[10] probably formed by the free Al present in the as-sprayed coating. The coatings from the sintered and atomized powders resulted in a surface with green spots and stringers consisting of nickel sulfite.[10] These nickel sulfite formations were less voluminous compared to the aluminum sulfate formed on the clad powder coatings.

Coatings from each powder manufacturing technique were heat treated (900 °C for 24 hr) to form an oxide on the coating surface before the corrosion test. No evidence of attack was found, which indicates that the oxides are resistant. However, Ni and Al are the attacked phases in the non-heat-treated coatings.

Oxidation plays an important role in deterioration of Ni-5wt.%Al coatings. Oxide growth rate curves for thermal cycling (20 to 900 °C, 1 hr at temperature) show significant differences in oxidation resistance between coatings made from the ten powders (Fig. 6).

The gas-atomized powders are the best coatings with regard to oxidation resistance, with sintered and water-atomized powders second. The clad powders oxidized most readily. This latter result may be explained due to development of NiO on the clad powders during oxidation; because the aluminum is transformed to Al_2O_3 during spraying. The gas-atomized powders form a film of Al_2O_3 on the surface. This film slows nickel from diffusing to the surface where NiO is formed. The formation of NiO is the oxidation mechanism of the coatings. Water-atomized powders exhibit a higher oxidation rate compared to gas-atomized materials. One explanation could be that, because the water-atomized powders contain more oxygen, they develop some Al_2O_3 during spraying. A consequence would be that less aluminum is left for formation of the Al_2O_3 film during oxidation.

The sintered powder exhibits behavior similar to that of the water-atomized powders. In this case, the formation of Ni_3Al during powder manufacture results in less aluminum being available to form Al_2O_3 during oxidation.

4. Conclusions

Ten Ni-5wt.%Al powders manufactured by four different techniques were evaluated and plasma sprayed. Several correlations between powder characteristics and the as-sprayed coating properties were found. Conclusions from these findings are listed below. For cold environment applications, where good adhesion is required, any powder can be used, except for gas-atomized powders with no or low boron content. For hot environment applications, gas-atomized powders are superior with respect to oxidation resistance. Moderate salt-rich atmospheres do not seem to be harmful to any of these coatings. For sulfuric atmosphere applications, coatings that are heat treated have the best corrosion resistance. If heat treatment is not performed, then coatings from sintered, water-, and gas-atomized powders result in nickel sulfite on the surface, whereas coatings from clad powders form aluminum sulfate.

Acknowledgment

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