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Evaluation of NiAl and NiAl-B Deposited By Vacuum Plasma Spray

J.Z. Chen, H. Herman, and S. Safai

Nickel aluminide, NiAl, has potential for high-temperature structural applications because of its high melting temperature, low density, high elastic modulus, and excellent oxidation resistance. However, inadequate room-temperature ductility and low strength at high temperatures limit the use of NiAl for practical service. An added limitation is the difficulty of forming this intermetallic compound. Plasma spray forming is suggested to produce NiAl in useful shapes and forms. In this study, vacuum plasma spray has been used to produce coatings and free-standing forms of NiAl and NiAl microalloyed with boron (NiAl-B). This research focuses on an investigation of the structures and properties of the as-sprayed deposits. Preliminary analysis reveals that the deposits formed by vacuum plasma spray are very dense and essentially oxide free. The as-received aluminide powders and the sprayed deposits have the same phase structure. Tests also indicate that the mechanical properties achieved by plasma spray are comparable to those achieved by other processing techniques.

1. Introduction

INTERMETALLIC compounds are being investigated extensively for use as high-temperature structural materials. These compounds, and composites based on them, are expected to be in demand for aerospace as well as for other high-performance applications. Much research has focused on Ni_3Al, which has a yield strength that increases with temperature.[1] Of particular interest here is NiAl, which offers four key advantages:[2]

- Its density (5.86 g/cm^3)[3] is approximately two thirds the density of nickel-base superalloys.
- It has high thermal conductivity (four to eight times that of nickel-base superalloys), thus life-limiting "hot spot" temperatures in turbine blades can be reduced.
- It has excellent oxidation resistance. In fact, NiAl has been used as a surface protective coating since the 1950s.
- Its simple, ordered bcc *B*2 crystal structure allows more plastic deformation than many other intermetallic compounds. Furthermore, NiAl is a promising matrix for composite materials such as NiAl reinforced with TiB_2.

On the negative side, inadequate room-temperature ductility and low strength at high temperatures limit its practical service. The high strength and low ductility of NiAl at room temperature also pose great difficulties for fabrication into useful components.

Various processing techniques for fabricating nickel aluminides have been examined. Conventional casting of nickel aluminide is difficult because segregation and very large grain sizes are detrimental to properties. Secondary processing, such as hot extrusion, is essential to refine the grain size through dynamic recrystallization.

Thermal spray forming of near-net shapes can circumvent the need for complex forming processes. Vacuum plasma spray (VPS) combines melting, quenching, consolidation, and self-annealing in a single operation,[4-6] and is capable of producing protective coatings as well as thick, dense, free-standing forms, from which near-net shaped components can be manufactured. The VPS technique is also a rapid solidification process with cooling rates of 10^7 K/s,[5] resulting in fine-grained microstructures, which will lead to improvements in mechanical properties of NiAl. Furthermore, it has been shown that VPS-processed aluminides[7,8] have properties that are superior to such materials formed using competitive techniques. In particular, VPS two-phase nickel aluminides (NiAl-Ni_3Al) display excellent mechanical properties.[9]

This article is a part of an on-going study of VPS spray-formed NiAl. Vacuum plasma spray has been used to produce coatings and free-standing forms of NiAl and NiAl-B, with emphasis on investigating microstructures and properties of the deposits.

Key Words: free-standing forms, intermetallics, microstructure, nickel aluminide

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2. Experimental Procedure

Two powders with chemical compositions corresponding to pure, stoichiometric NiAl and NiAl + 0.044 wt% B were provided by Pratt & Whitney Inc. The particle size was –44 μm.

Table 1 Spray parameters

Gun	PT-F4V
Nozzle (anode) diameter, mm	7
Powder injector, mm	3
Current, A	700
Voltage, V	68
Argon flow rate, SLPM	50
Hydrogen flow rate, SLPM	9
Carrier gas (Ar) flow rate, SLPM	2
Chamber pressure, mbar	60
Spray distance, cm	30
Powder feed rate, g/min	30-40

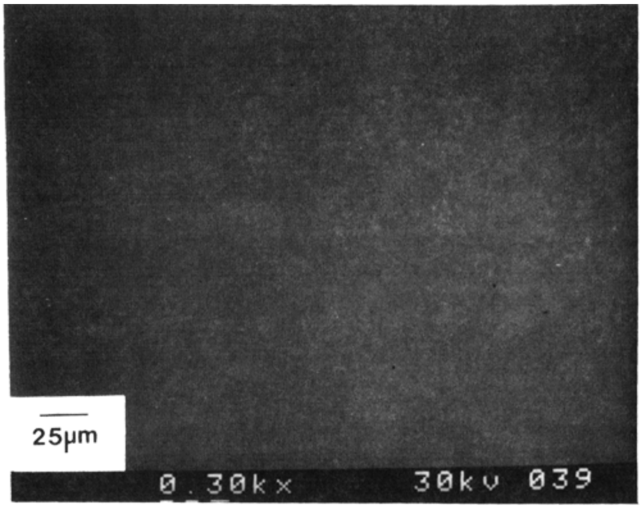


Fig. 1 SEM cross section showing dense, oxide-free as-sprayed NiAl deposits typical of coatings and free-standing forms.

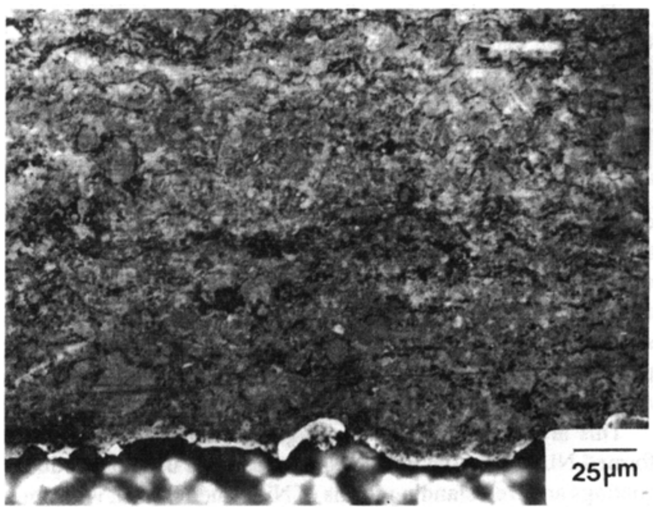
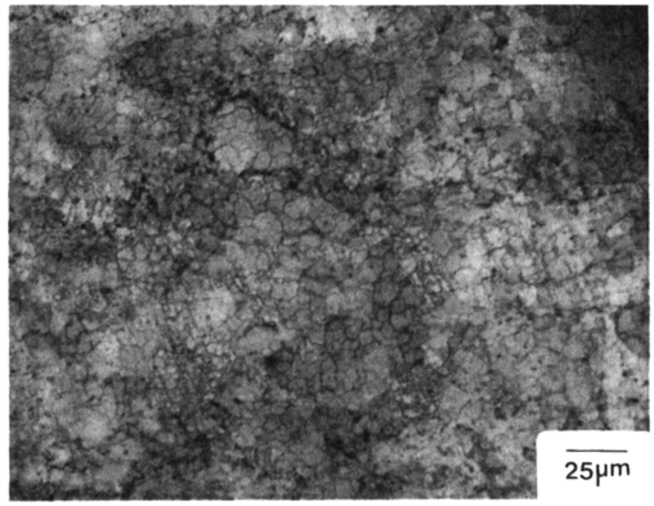
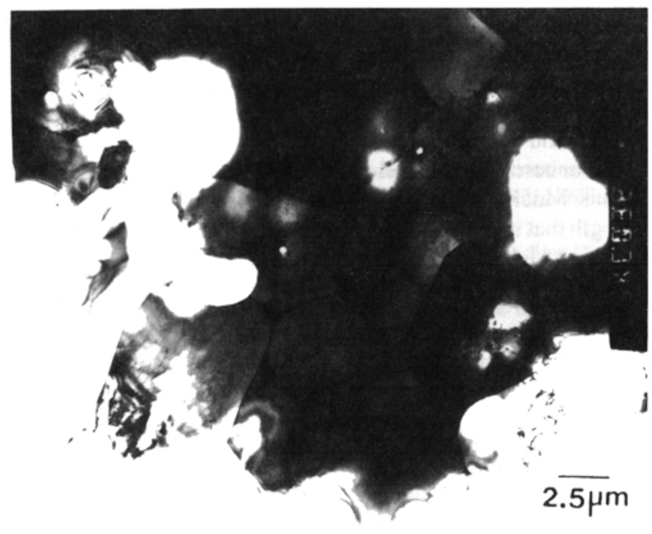


Fig. 2 Optical micrograph of cross section of the as-sprayed NiAl coating after etching. The grain size was too small to be observed.



(a)



(b)

Fig. 3 (a) Optical micrograph of cross section of the as-sprayed NiAl free-standing form after etching. The grain growth size was revealed. (b) TEM micrograph of the as-sprayed NiAl free-standing form.

Vacuum plasma spraying was performed on a Plasma-Technik VPS unit using a PT-F4V plasma gun with internal powder injection, under the conditions listed in Table 1. The feedstock powders were sprayed onto mild steel substrates to form deposits with a thickness of about 0.3 mm for coatings and 2.5 mm for free-standing forms, which were separated from the substrates after spraying.

X-ray diffractometry with CuKα radiation was performed to identify phases. Optical metallography, scanning electron microscopy (SEM), and transmission electron microscopy (TEM) analyses were carried out. The densities of free-standing deposits were measured by the water displacement technique. Vickers microhardness measurements (VH) were conducted on polished cross sections using a 500-g load for 15 s. Preliminary cavitation/erosion tests of coatings were performed in distilled water at approximately 20 °C, using an ultrasonic vibratory device composed of a piezoelectric transducer and an exponentially shaped horn, operating at a frequency of 20 kHz with an amplitude of 40 μm. The distance between the specimen and horn tip was 0.64 mm.

3. Results and Discussion

X-ray diffraction performed on both the powders and the as-sprayed deposits showed only the NiAl phase. This indicates that VPS-formed NiAl deposits retain the phase structure of the starting powder. This absence of a phase change associated with the VPS forming of NiAl is considered to be a positive feature of VPS processing.

The SEM micrographs of cross sections of the as-sprayed coatings and the free-standing forms were all featureless and in-

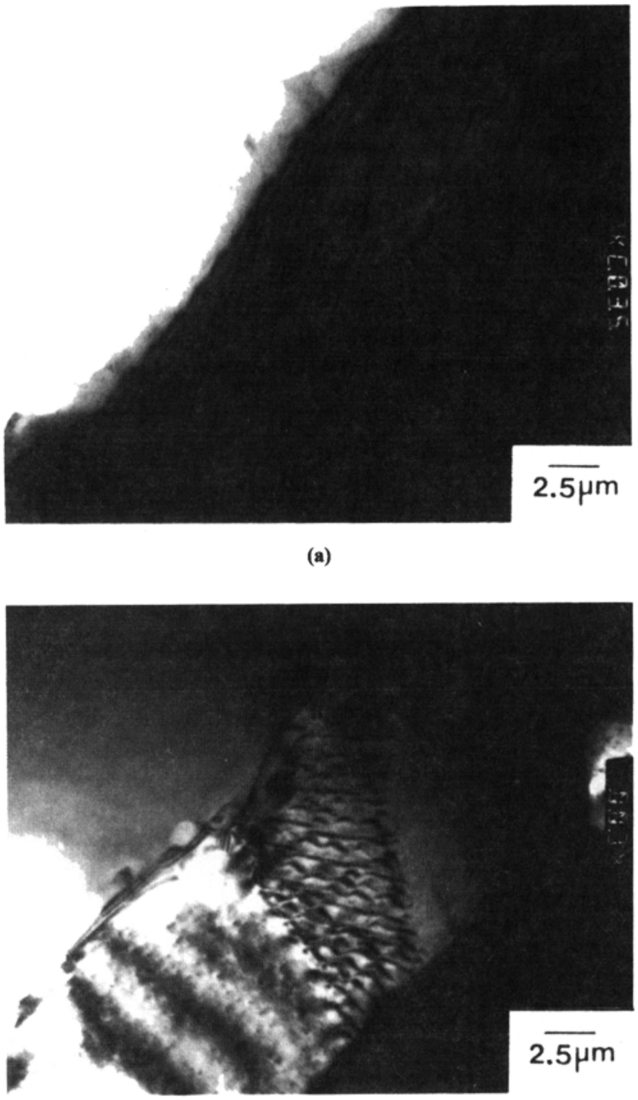
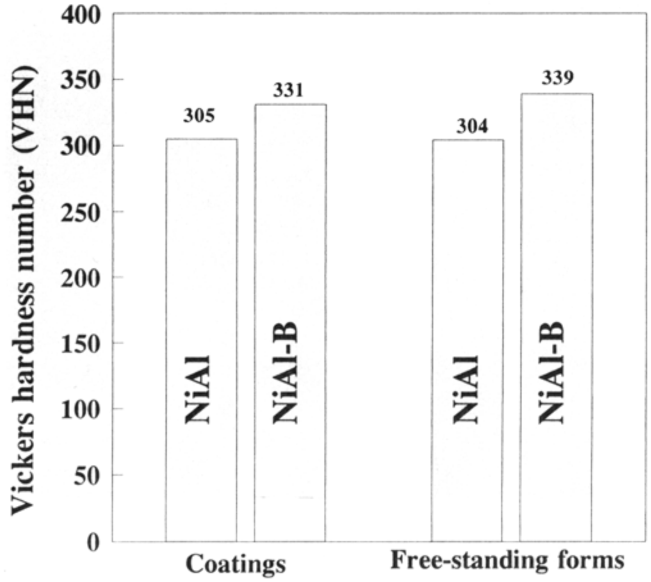


Fig. 4 TEM micrographs of the as-sprayed NiAl free-standing form. (a) Low-angle grain boundary dislocations. (b) Dislocation networks.



dicative of dense, oxide-free, pore-free deposits (Fig. 1). The densities of the free-standing forms were 5.82 ± 0.01 g/cm^3 for NiAl and 5.74 ± 0.02 g/cm^3 for NiAl-B, i.e., 99.3 ± 0.02% and 98.0 ± 0.04% of theoretical density, respectively, based on water displacement measurements.

Although the essentially featureless SEM micrographs, as shown in Fig. 1, were almost identical for all deposits, further investigations disclosed that each specimen differed in other aspects. The microstructures of the as-sprayed deposits were also revealed after etching the specimens with CrO_3 + HCl as well as investigated with TEM. The grain size of the as-sprayed *thin* coating, which is generally submicrometer,[5] could not be resolved by optical microscopy (Fig. 2). However, a grain structure was observed in the as-sprayed *free-standing form* (Fig. 3a), and distinct grains on the order of several micrometers were distinguished by TEM (Fig. 3b). The free-standing deposit, which was spray-formed for a much longer duration compared to the thin coating, discloses the self-annealing effects of VPS due to the high temperature of the process. Self-annealing of the VPS process also leads to stress-relief, and therefore, thick deposits can be produced. Moreover, TEM examination showed low-angle grain boundary dislocations, another indication of the self-annealing effect of VPS, as well as dislocation networks (Fig. 4a and b).

The microhardness measurements of the coatings and the free-standing forms of the as-sprayed NiAl and NiAl-B deposits are given in Fig. 5. Those values are comparable to the hardness of the materials produced by other methods.[10,11] Although microhardness generally increases with decreasing grain size, the current results show that the VH values are more dependent on chemical composition than on grain size. As mentioned above, the grain sizes of the coatings and the free-standing forms show significant differences, but VH values of the coatings and the free-standing forms for NiAl or NiAl-B are almost identical. When pure NiAl is compared with NiAl microalloyed with boron, the VH values of the latter deposits (the coating or the free-standing form) are about 10% higher than those of NiAl.

The addition of boron, which dramatically increases low-temperature ductility in Ni_3Al, has been investigated by George and Liu.[12] They found that the ultimate tensile strength of NiAl increases by about 50% with 0.030 wt% boron microalloying. The strength increase is sensitive to the amount of boron addition. The strengths of VPS NiAl and NiAl + B (0.044 wt%) deposits will be measured later in this program.

To date, few studies of cavitation/erosion have been carried out on ordered intermetallic NiAl, although cavitation/erosion of other intermetallics, such as Ni_3Al and two-phase material (Ni_3Al-NiAl) have been investigated.[13-15] The alloy is more erosion resistant in the ordered state than in the disordered state.[13] Johnson et al. observed that alloys based on the intermetallic Ni_3Al provide superior resistance to cavitation/erosion compared to many commercial erosion-resistant alloys.[14]

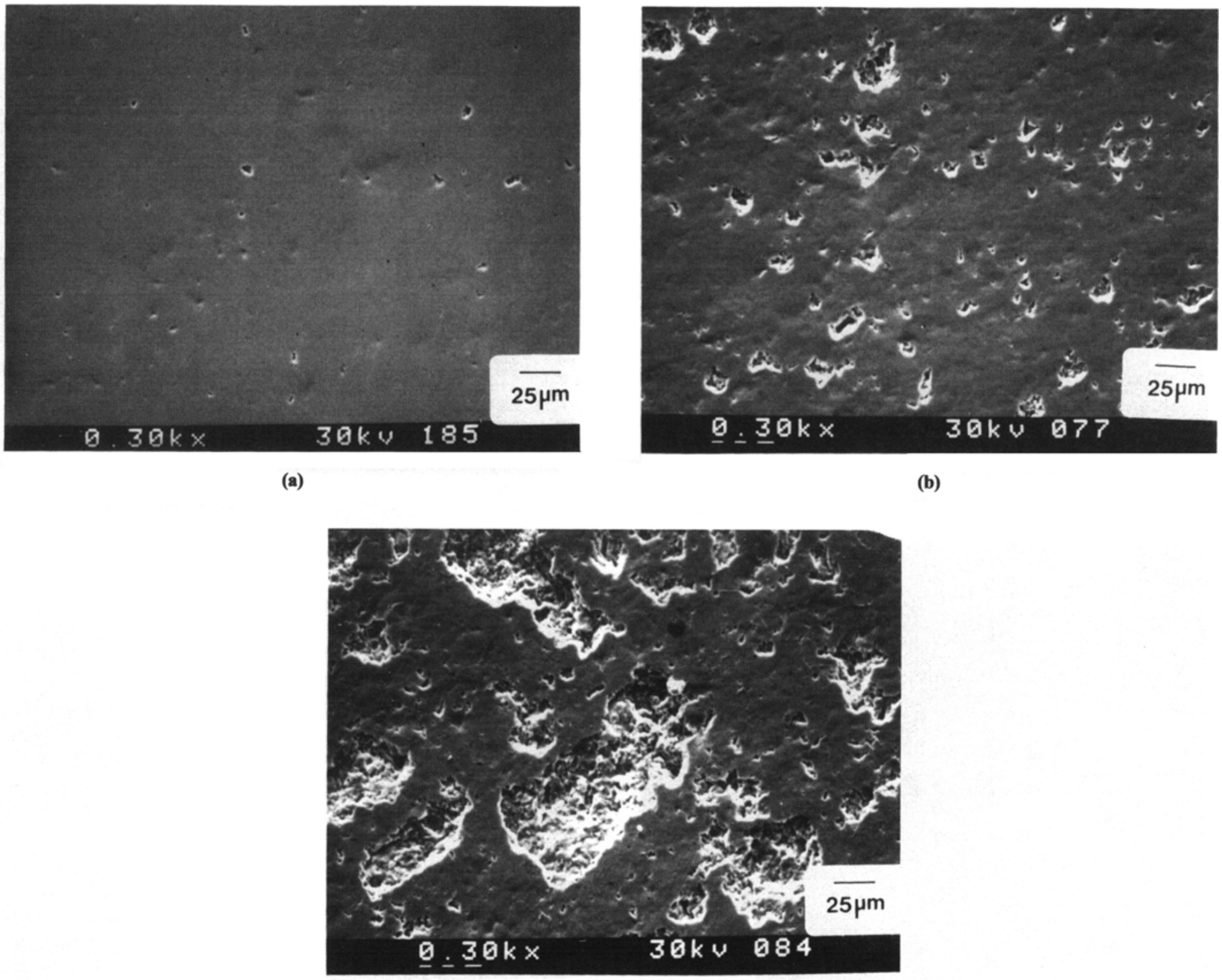


Fig. 6 SEM micrographs of the eroded surfaces of the as-sprayed NiAl coating for various test times. (a) 2 min. (b) 40 min. (c) 120 min.

Alloys based on the intermetallic NiAl, which has a high ordering free energy, may be expected to have good erosion resistance. The eroded surfaces observed by SEM are presented in Fig. 6 after various test times. It is found that the surface after 2 min is plastically deformed at several locations. The pits appear as pores or as shallow indentations, as shown in Fig. 6(a). As exposure increases to 40 min, the number of pits increases and the pits become larger. Wavy surface undulations also occur (Fig. 6b). After 120 min, the surface is further damaged; large craters are formed, but as shown in Fig. 6(c), the entire surface is not completely damaged. Further experiments demonstrate that, based on the evolution of surface damage during cavitation/erosion, VPS deposits of NiAl and NiAl-B display similar damage features. Preliminary study shows that the weight loss rate of NiAl and NiAl-B is in the range of ~1 mg/h, which is much lower than that of type 304 stainless steel.[14] Further work is being carried out on quantitative weight loss measurements of these materials.

4. Conclusion

Dense, oxide-free and pore-free coatings as well as free-standing forms of NiAl and NiAl-B were deposited by the vacuum plasma spray process. The deposits retained the same phase structure as the starting powders. The as-sprayed free-standing deposits exhibit a large grain size due to self-annealing during vacuum plasma spray processing. The microhardness of the deposits is comparable to that produced by other processing methods. The hardness of NiAl-B is about 10% higher than that of NiAl. The coatings of NiAl and NiAl-B appear to have similar cavitation/erosion features.

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