JTTEE5 6:335-344
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Reactive Spraying of Nickel-Aluminide Coatings

S.C. Deevi, V.K. Sikka, C.J. Swindeman, and R.D. Seals

Reactive spraying of nickel aluminides was accomplished via reaction synthesis techniques in which nickel and aluminum powders were fed through a direct-current plasma torch onto carbon steel substrates. The as-sprayed coatings obtained by reactive spraying were characterized by x-ray diffraction and microscopic techniques. Reactive spraying of nickel and aluminum resulted in coatings consisting of Ni, Al, Ni_3Al, $NiAl_3$, Ni_5Al_3, NiAl, and Al_2O_3, depending on the experimental conditions. Nickel aluminide phases observed in plasma spray depositions were compared with the phases obtained by combustion synthesis techniques, and the formation of phases in reactive spraying was attributed to the exothermic reaction between splats of aluminum and nickel. Primary and secondary reactions leading to the formation of nickel aluminides were also examined. The splat thickness and the reaction layer suppressed the formation of desired equilibrium phases such as Ni_3Al and NiAl. As-sprayed coatings were annealed to enhance the diffusional reactions between the product phases and aluminum and nickel. Coatings obtained by reactive spraying of elemental powders were compared with as-sprayed and annealed coatings obtained with a bond coat material in which nickel was deposited onto aluminum particles.

Keywords combustion synthesis, nickel aluminides, plasma spraying, reactive spraying

1. Introduction

Intermetallic compounds based on Ni_3Al and NiAl possess high melting points, simple ordered crystal structures, low densities (10 to 25% lower than superalloys), and good high-temperature mechanical properties as compared to many of the commercial alloys and superalloys. They possess sufficiently high concentrations of aluminum in the range of 13 to 32 wt% to form continuous, fully adherent alumina layers on the surface when exposed to air or oxygen atmospheres. The alumina layers provide excellent oxidation and carburization resistance up to 1100 °C or higher. Nickel aluminide, based on Ni_3Al, exhibits excellent wear resistance at temperatures higher than 600 °C, with wear resistance increasing with temperature by two or three orders of magnitude. Also, the cavitation erosion resistance of Ni_3Al is superior to many currently used materials (Ref 1-4). These attributes make intermetallics based on Ni_3Al and NiAl suitable for a variety of high-temperature coating applications. The coatings can potentially provide the qualities and attributes of MCrAlY coatings based on chromium (where M = Ni, Co, and Fe).

Some of these attributes have been realized in thermal-sprayed Ni_3Al and NiAl coatings (Ref 5-8). Recent advances in alloy design involving small additions of boron to an off-stoichiometric Ni_3Al resulted in a tensile ductility of 40 to 50% (Ref 3). However, NiAl still exhibits very low ductility at room temperature and lacks high-temperature strength for monolithic applications. On the other hand, thermal spray deposition of Ni_3Al and NiAl coatings can protect the substrate materials against oxidation, corrosion, erosion, and wear in specific environments. Thermal spray deposition techniques are also ideal for net- and near-net-shape forming processes (Ref 9-12) and can overcome the processing problems associated with low room-temperature ductility and inadequate high-temperature strength for structural applications. Herman et al. (Ref 7-10, 12) investigated thermal spray deposition of nickel aluminide coatings and characterized the microstructures and coating quality based on prealloyed and mechanofused powders.

The formation of Ni_3Al and NiAl is accompanied by a significant release of energy, and the magnitude of the heat released is dependent on the Al/Ni ratio. The energy associated with the reaction between nickel and aluminum has been used to combine the synthesis and densification of Ni_3Al and NiAl by powder metallurgical routes (Ref 13-21). Table 1 provides melting points, weight percent of aluminum, densities, and heats of reaction (Ref 22). The heat of reaction can raise the temperatures of nickel and aluminum close to their melting points. It is thus reasonable to expect that, under certain conditions, in situ coatings of Ni_3Al, NiAl, and mixed phases of nickel aluminides can be obtained by feeding nickel and aluminum powders through a plasma spray gun. Intense heat is transferred to the particles during passage of the powders through the plasma, and the heat transfer can uniquely promote the formation of intermetallic coatings on the substrate. This approach, based

Table 1 Weight percent of aluminum, densities, heats of formation, and melting points of nickel aluminides

Intermetallic	Al, wt%	Density, g/cm³	Heat of formation ($\Delta Hf298$), kcal/mol	Melting point, °C
Ni_3Al	13.28	7.293	−36.6 ± 1.2	1395
NiAl	31.49	5.910	−28.3 ± 1.2	1639
Ni_2Al_3	40.81	4 787	−67.5 ± 4.0	1133
$NiAl_3$	57.96	3.957	−36.0 ± 2 0	854

Source: Ref 22

S.C. Deevi, Research Center, Philip Morris, Richmond, VA 23234, USA; **V.K. Sikka,** Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA; **C.J. Swindeman** and **R.D. Seals,** Thermal Spray Technology Center, Oak Ridge Centers for Manufacturing Technology, Oak Ridge, TN 37831, USA.

on the use of at least one elemental powder or unalloyed material, is hereafter termed "reactive spraying" and is a unique method of obtaining intermetallic coatings and their composites in situ. In some cases, preheating the substrate or annealing the coating may be required to homogenize and stabilize the phase structures of coatings.

This paper summarizes some observations on the reactive spraying of nickel aluminides with an emphasis on the characterization of phases by x-ray diffraction (XRD), optical microscopy, scanning electron microscopy (SEM), and Vickers microhardness measurements. Coatings were obtained in this study by varying the feed rates and ratios of Ni/Al, and a total of at least 30 samples were obtained by reactive spraying.

2. Experimental Methodology

Plasma-sprayable powders of nickel and aluminum were prepared and sprayed as follows: (1) nickel and aluminum powders were blended and then fed to the plasma gun; (2) powders of nickel and aluminum were fed separately by adjusting the volumetric flow to attain the desired chemical composition (i.e., dual fed); and (3) powders of nickel and aluminum were mechanically alloyed to different lengths of time in an attritor under an argon cover and were fed through the plasma. Coatings of various thicknesses were deposited on carbon steel substrates using different power levels, feed rates, velocities (subsonic or mach I), plasma temperatures (by varying the Ar/He ratio), and

Table 2 Spray parameters

Sample no.	Target Ni/Al ratio, wt%	Method of mixing	Power, kW	Velocity	Substrate temperature, °C	Average coating thickness, mm
121494-15	68:32	Blended	30	Subsonic	...	0.25
121494-21	68:32	Blended	33	Mach I	...	0.30
121594-3	87:13	Blended	35	Mach I	...	0.275
32395-25	68:32	Blended	40	Subsonic	...	0.175
32395-30	87:13	Blended	40	Subsonic	...	0.225
40395-10	68:32	Blended	40	Subsonic	490-670	0.25
40795-6	2:1	Dual fed	48	Subsonic	540-680	0.625
40795-8	3:1	Dual fed	48	Subsonic	540-670	0.80
40795-9	95:5	As supplied	37	Subsonic	100	0.15

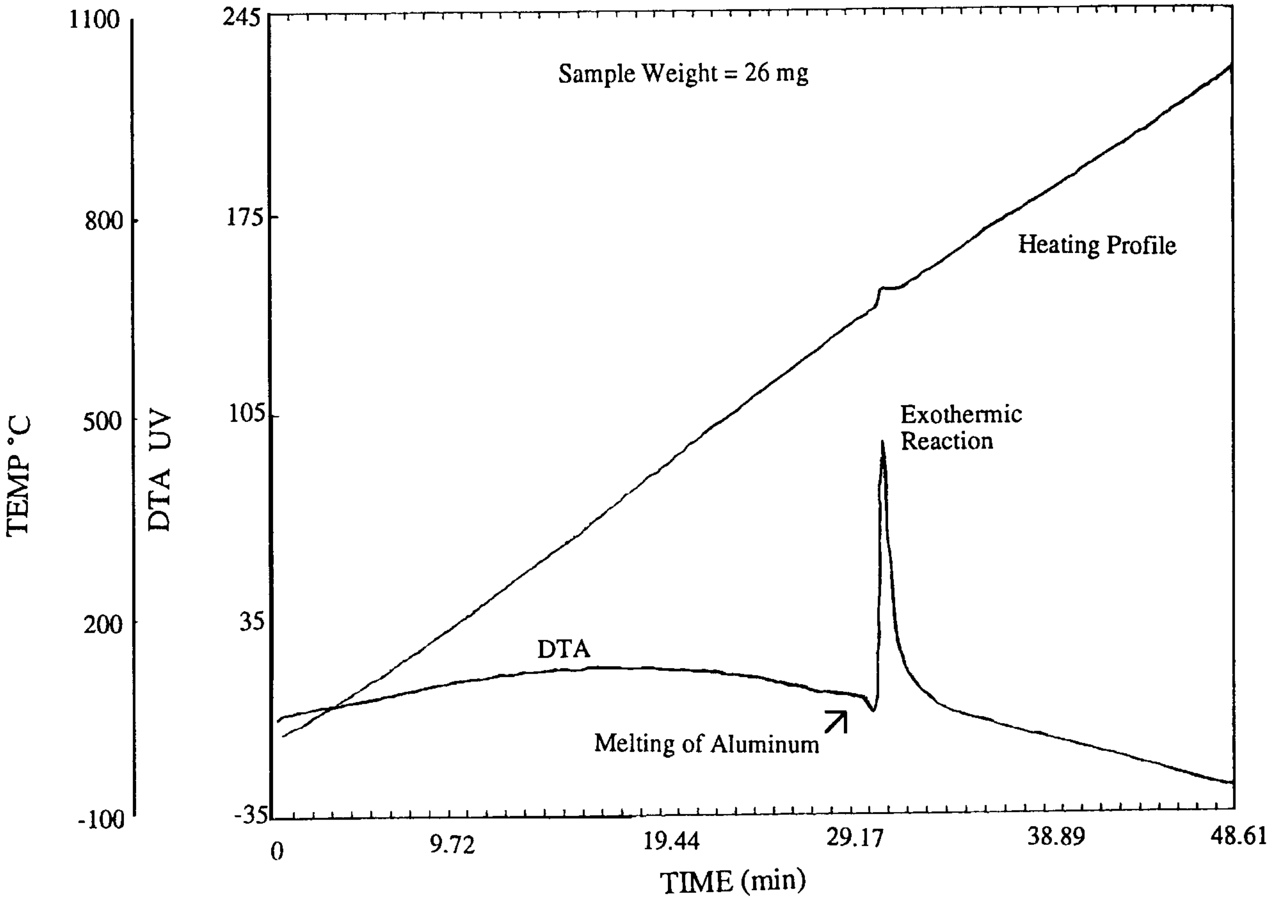


Fig. 1 DTA of nickel and aluminum (1:1) powdered mixture in argon atmosphere at 10 °C/min

substrate preheat temperatures to carry out reactive spraying of nickel-based intermetallics. Table 2 lists spray parameters and average coating thicknesses of the coatings obtained under different conditions. Coatings were annealed in argon by heating the substrates at 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h, followed by furnace shutdown.

Phase identification by XRD was carried out with CuKα radiation, and a step-scanning procedure was employed to obtain the diffraction patterns. Phases were identified with JCPDS (Joint Committee on Powder Diffraction Standards) peak files, and lattice parameters were calculated to determine the solubility of aluminum in nickel. Optical metallography, SEM, and Vickers microhardness measurements were carried out on polished cross sections after etching.

3. Results

To illustrate the exothermic reaction between nickel and aluminum, a mixture of nickel and aluminum in a 1:1 atomic ratio was heated in argon at 10 °C/min in a differential thermal analysis (DTA) unit. As seen in Fig. 1, a strong exothermic reaction followed melting of aluminum, with the reaction occurring in a very narrow range of temperature. Since the reaction occurred above the melting point of aluminum, the reaction can be termed as a liquid (Al)-solid (Ni) diffusional reaction. Therefore, exothermic reactions leading to nickel aluminides are expected with plasma spraying of nickel and aluminum powders, provided that aluminum is heated close to its melting point and that the diffusional distances between aluminum and nickel are small. If these conditions are met, nickel aluminides will be formed exothermically in the coatings.

Reactive spraying of a mixture of nickel and aluminum in a 1:1 ratio at 40 kW resulted in several phases (sample no. 32395-32). The XRD pattern on the coating indicated the presence of nickel and aluminum along with the nickel aluminide phases Ni_2Al_3, Ni_5Al_3, and $NiAl_3$ (Fig. 2). The microstructure of the coating and the interface between the coating and the substrate can be seen in Fig. 3(a). A dark phase present around the splats may possess nickel aluminide phases (Fig. 3b). This sample was annealed in argon at 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h. Annealing allowed further diffusional reactions to take place, and the observed phases were Ni and Ni_3Al with some Ni_5Al_3 phase. The relative intensities of the peaks and the phases shown in Fig. 2 and 4 are different, and NiAl phase was not observed even after annealing.

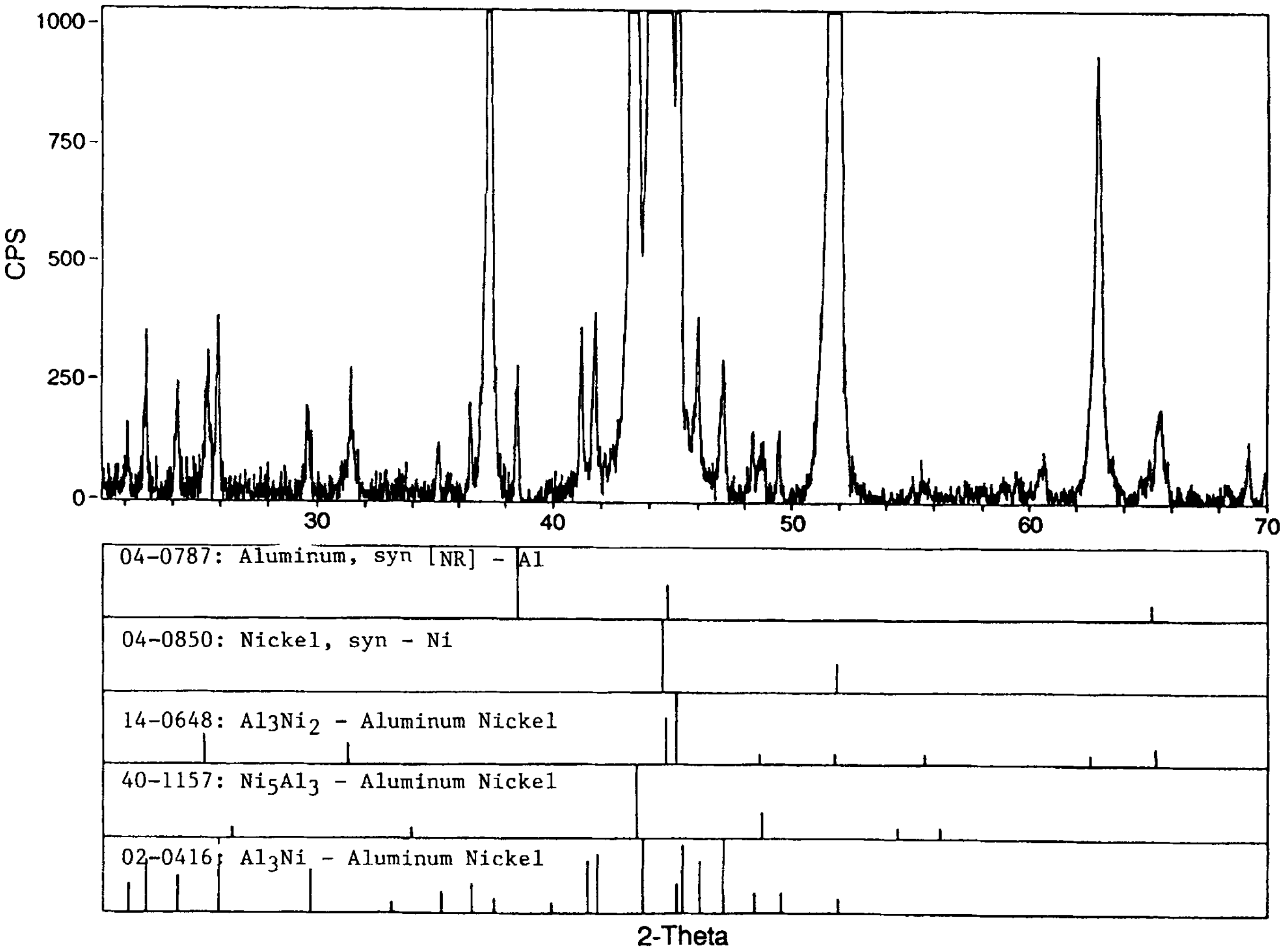
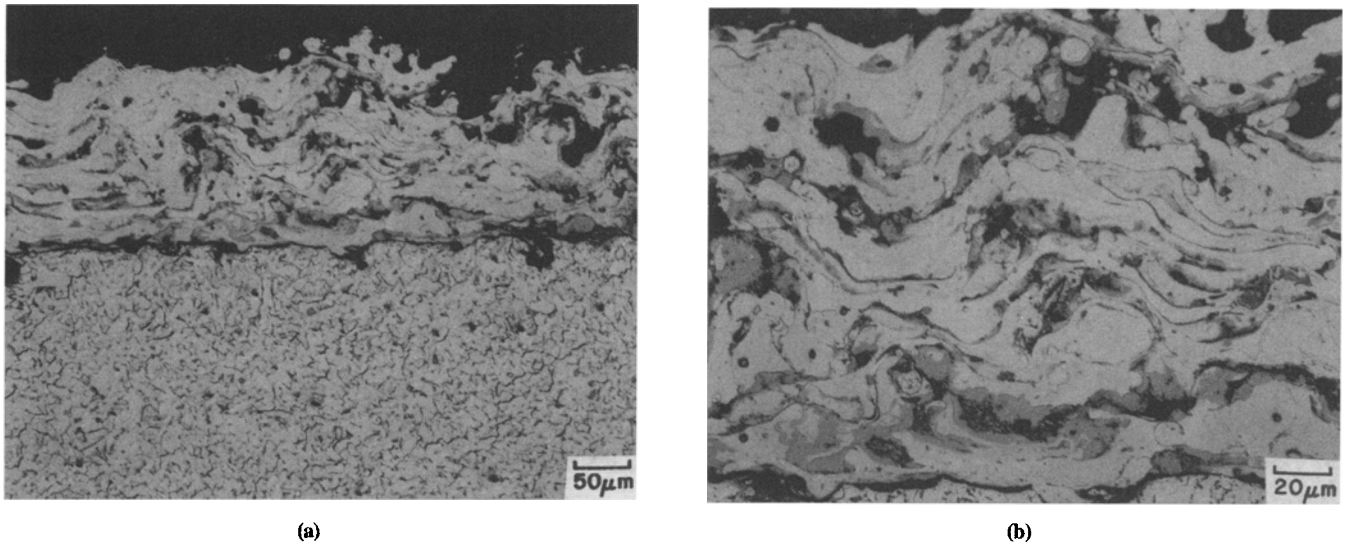


Fig. 2 XRD pattern obtained after plasma spraying of nickel and aluminum (1:1) powders at 40 kW



(a)

(b)

Fig. 3 Microstructure of plasma-sprayed coating with nickel and aluminum (1:1)

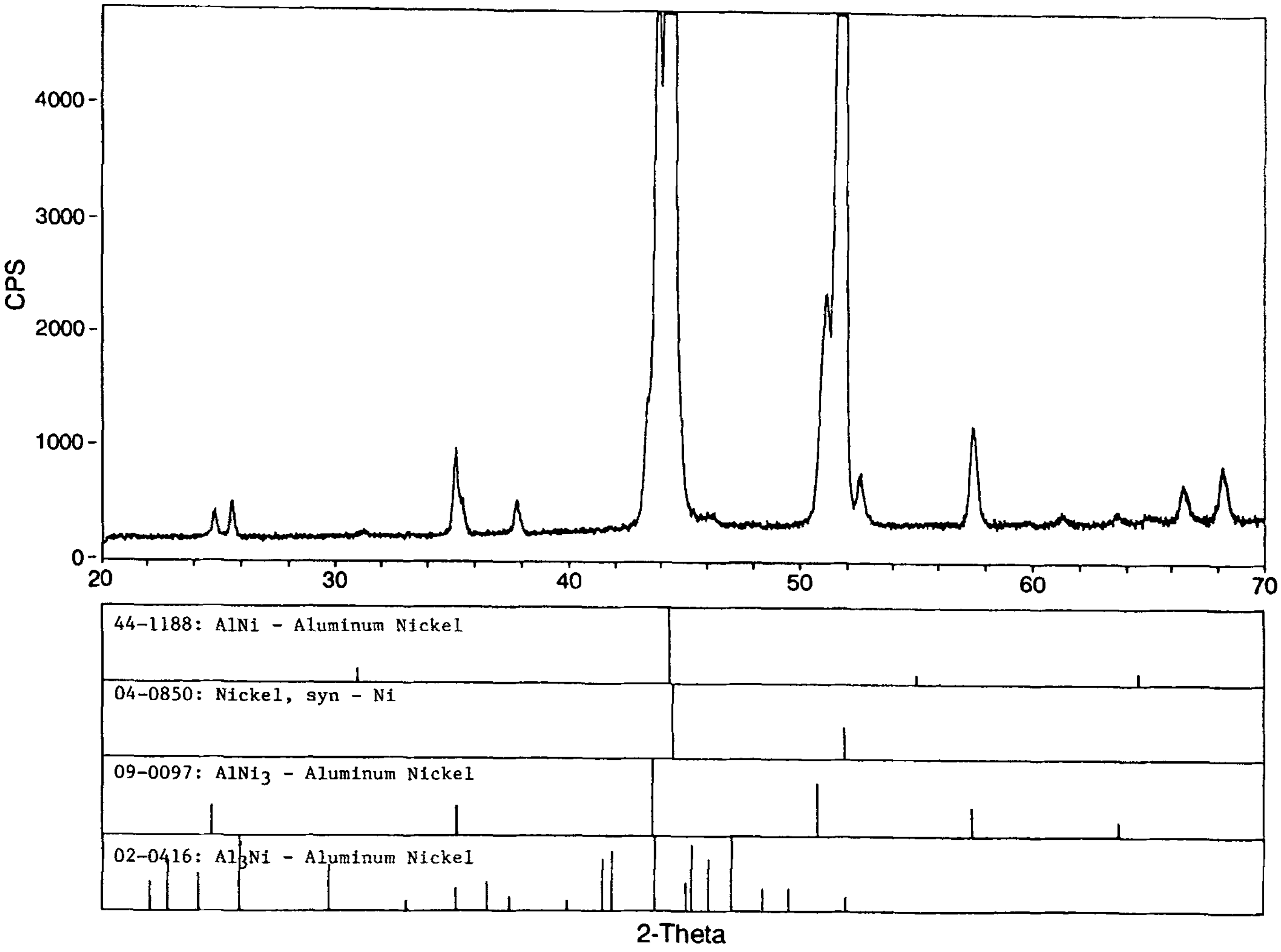


Fig. 4 XRD pattern of the coating obtained after heating at 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h

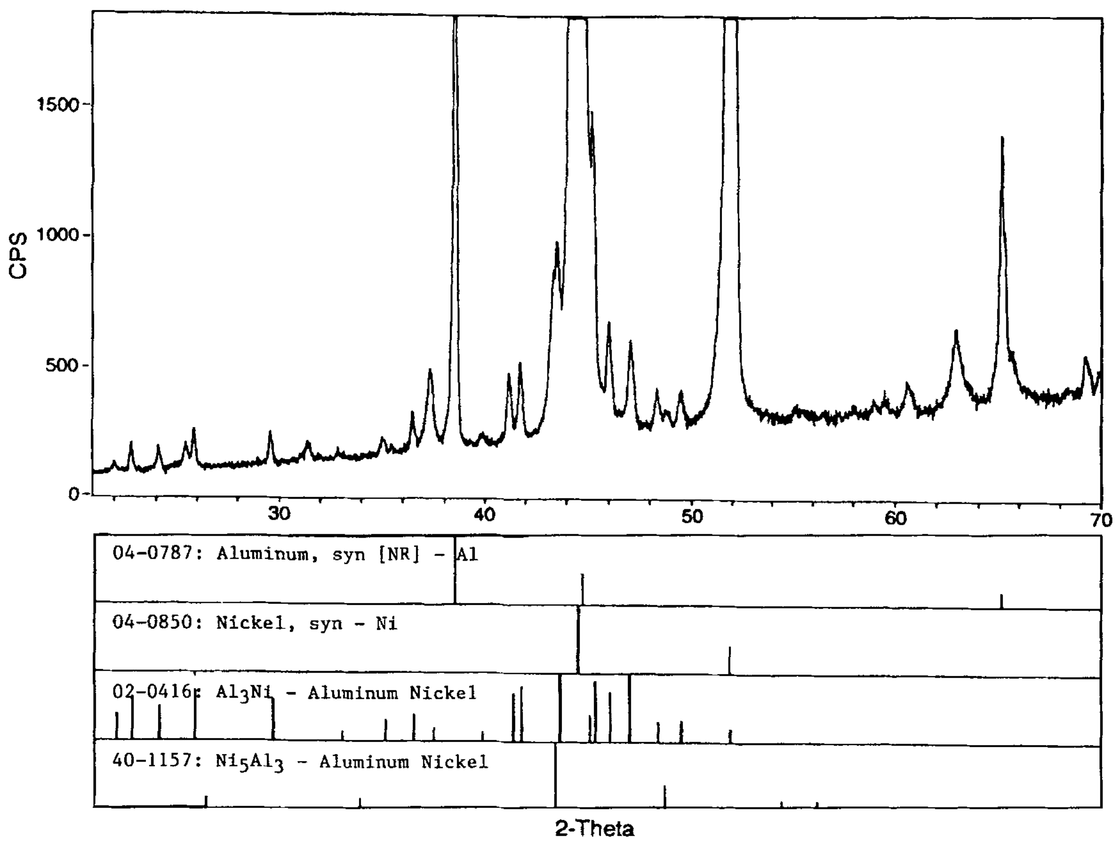


Fig. 5 XRD pattern obtained after plasma spraying a mixture of nickel and aluminum through a single feeder

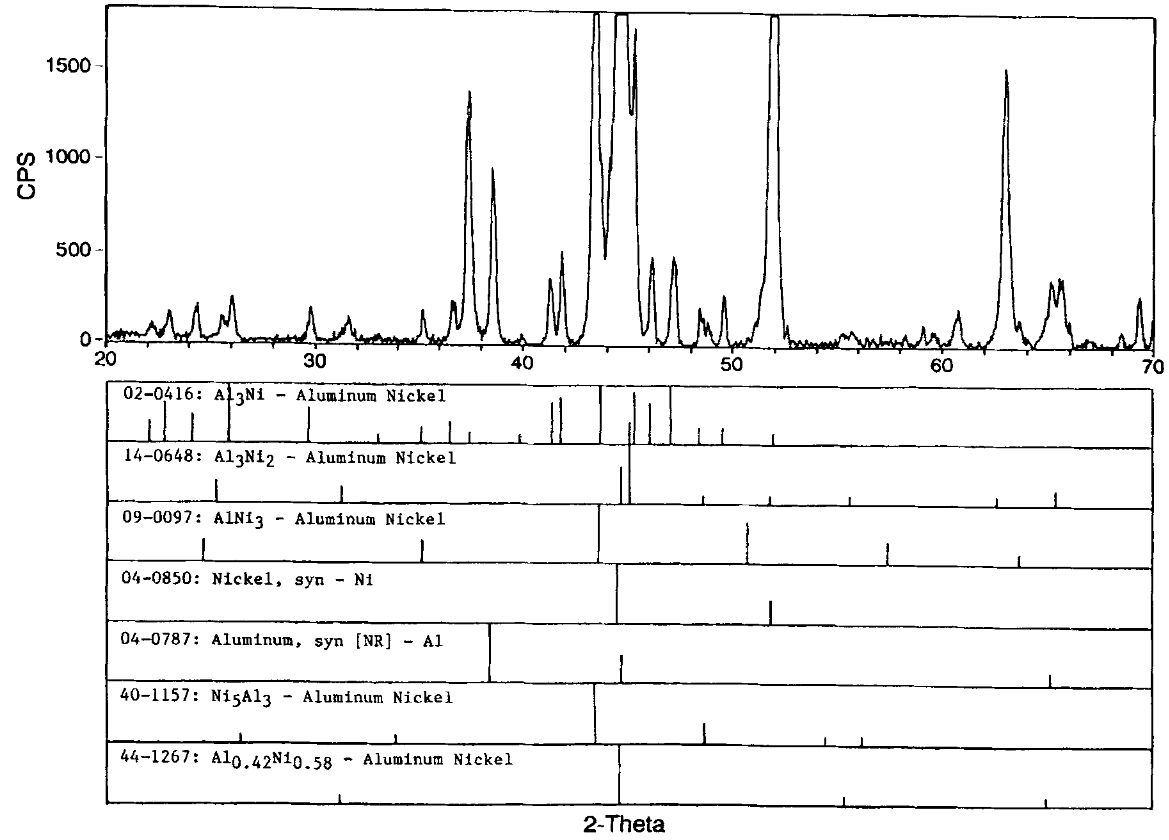
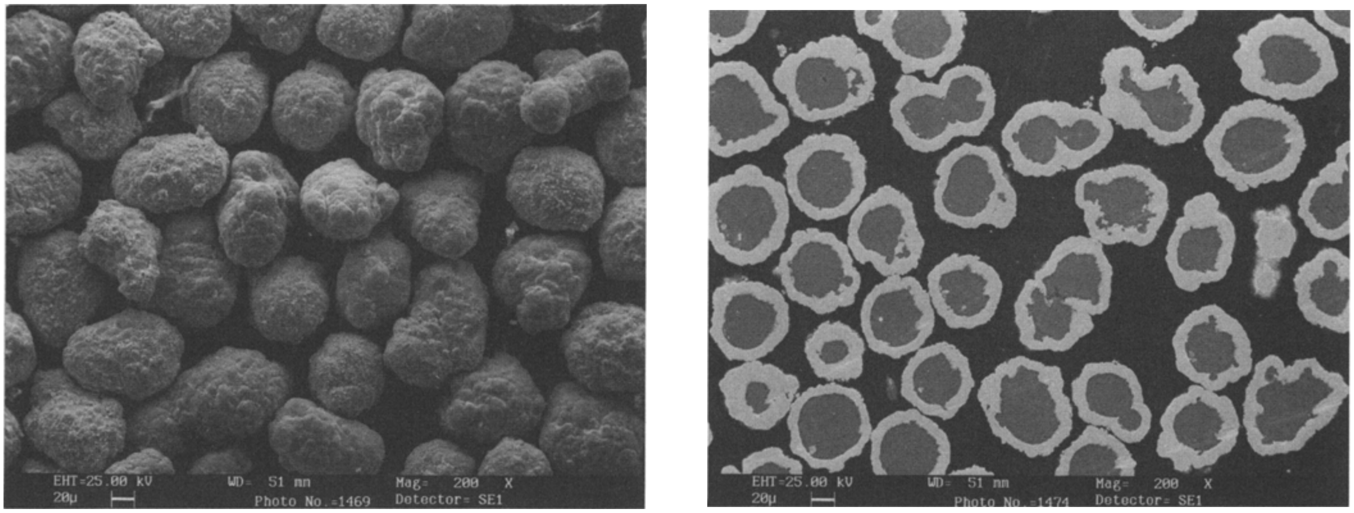


Fig. 6 XRD pattern of plasma-sprayed coating with nickel and aluminum (1:1)



(a) (b)

Fig. 7 Morphology of (a) bond coat Ni/Al powder and (b) polished cross sections of the Ni/Al particles. X-ray dot mapping indicated that the inner core is aluminum and the outer core is nickel.

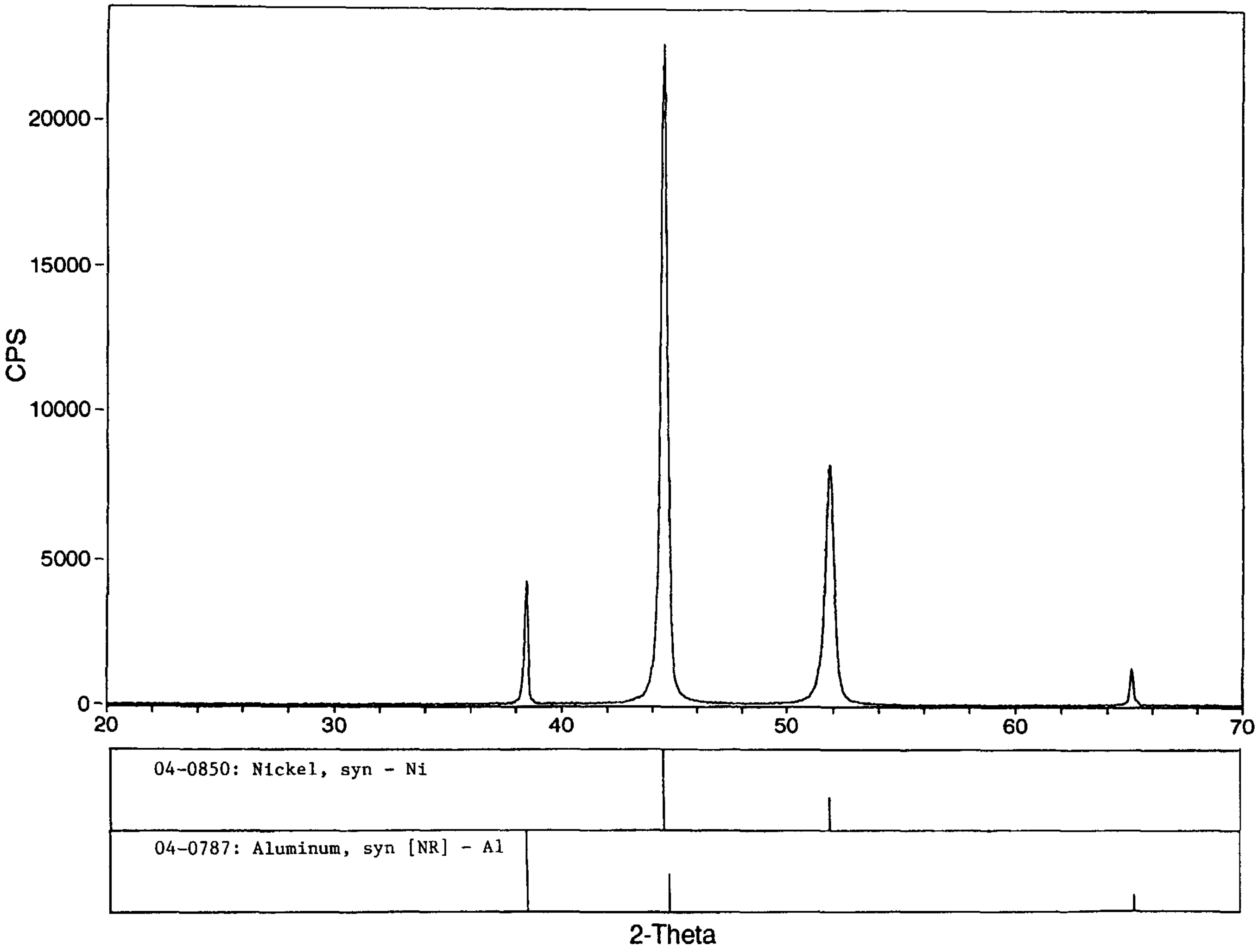


Fig. 8 XRD pattern of as-received bond coat Ni/Al powder shown in Fig 7

A coating obtained with the same ratio of nickel and aluminum, but at a velocity of mach I (sample no. 32395-32), shows the presence of Ni_5Al_3 phase along with Ni, Al, and $NiAl_3$ phases in the as-sprayed coating. The x-ray pattern of the as-sprayed coating after annealing in argon (at 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h) indicated $NiAl_3$ as the primary

Table 3 Observed phases in selected plasma-sprayed coatings obtained on carbon steel substrates

Sample no.	Target Ni/Al ratio, wt %	Method of mixing	Power, kW	Velocity	Preheat or postspray annealing	Average coating thickness, mm	Observed phases(a)
32395-32	87:13	Blended	40	Subsonic	...	0.225	Ni(vs), Al_3Ni(s), Al(w), Al_3Ni_2(vw)
32395-32	87:13	Blended	40	Subsonic	Postspray 1 h, 600 °C; 1 h, 800 °C; 1 h, 1100 °C; furnace cool	0.225	Ni(vs), $AlNi_3$(s), Al_3Ni(w), Al(vw)
32395-4	87:13	Blended	44	Mach I	...	0.200	Ni(vs), Al(s), Al_3Ni(w), Ni_5Al_3(vw)
32395-4	86:14	Blended	44	Mach I	Postspray 1 h, 600 °C; 1 h, 800 °C; 1 h, 1100 °C; furnace cool	0.200	$NiAl_3$(vs), Al(s)
41895-12	1:1	Dual fed	45	Subsonic	...	0.775	Ni(vs), Al_3Ni(s), Al(w), Ni_5Al_3(vw), $Al_{0.42}Ni_{0.58}$(t)
40795-9	95:5	Encapsulated powder	37	Subsonic	Preheat	0.150	Ni(vs), Al(s), Al_3Ni_2(t)
40795-9	95:5	Encapsulated powder	37	Subsonic	100 °C preheat; postspray 1 h, 600 °C; 1 h, 800 °C; 1 h, 1100 °C	0.150	Ni(vs), $AlNi_3$(s), Al(w)

(a) vs, very strong; s, strong; w, weak; vw, very weak; t, trace

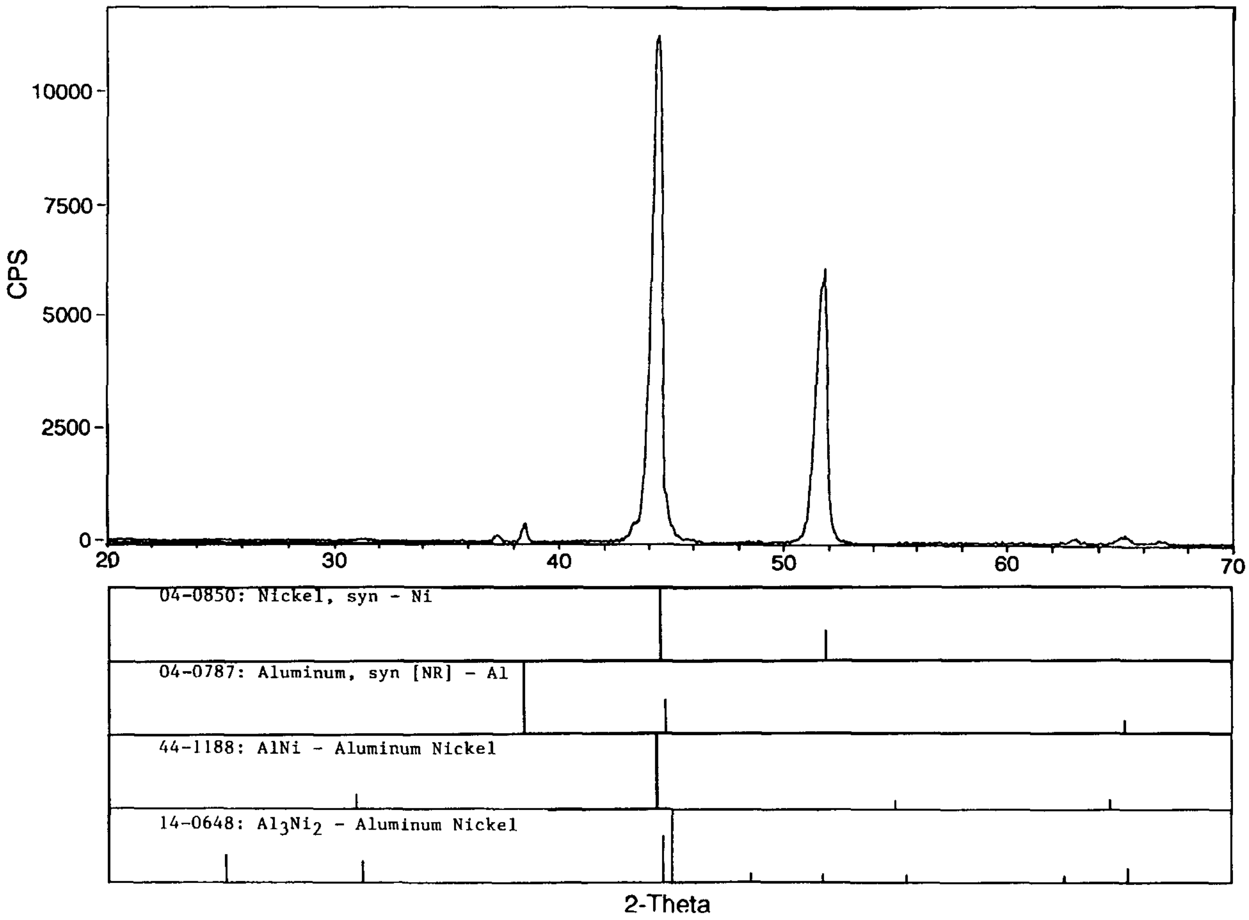


Fig. 9 XRD pattern of as-sprayed plasma-sprayed coating using bond coat Ni/Al powder material shown in Fig. 7

phase, and the intensity of the Al peak decreased dramatically (Fig. 5).

Analysis of a coating obtained from a mixture of nickel and aluminum produced by feeding nickel and aluminum to the plasma gun in a ratio corresponding to 1:1 (atomic) at a subsonic velocity indicated the presence of $NiAl_3$, Ni_2Al_3, and a trace of Al_2O_3 phases along with Ni and Al (Fig. 6). Annealing resulted in $Ni_{1.1}Al_{0.9}$, Ni_3Al, Al_2O_3, and Ni. Annealing of the as-sprayed coating resulted in a small Al peak, suggesting that a major portion of the aluminum reacted with nickel to produce the aluminides.

These experimental observations suggest that nickel aluminide phases can be obtained by reactive spraying of nickel and aluminum powders, and the phase formation depends on the thermal spray parameters and on the ratio of Ni/Al. Table 3 provides a selected list of experimental conditions and the intermetallic phases observed from XRD patterns. To compare the phases obtained from elemental powders with that of a bond coat material of nickel and aluminum in the ratio of 80:20 by volume (95:5 by weight, manufactured by Sherritt Gordon, Fort Saskatchewan, Alberta, Canada), the bond coat powder was plasma sprayed onto carbon steel substrates. This powder consists of an inner core of aluminum surrounded by nickel on the outer periphery. Figure 7(a) shows the morphology of the powder, and Fig. 7(b) shows the polished cross sections of several particles. Except in the case of a few isolated particles, nickel surrounds the aluminum core uniformly. The XRD pattern shown in Fig. 8 reveals that the bond coat powder consists of pure aluminum and pure nickel, with no intermetallic phases. The XRD pattern of the as-deposited coating indicated the presence of pure aluminum and pure nickel, along with a trace amount of Ni_2Al_3, but the relative ratio of Al/Ni decreased from 0.188 in the as-received powders to 0.0366 in the sprayed coating (Fig. 9). Annealing the coating to 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h resulted in the formation of Ni_3Al phase (Fig. 10), and the intensities of Al reflections decreased significantly when compared to the plasma-sprayed coating (compare Fig. 10 with Fig. 9).

4. Discussion

Exothermic reactions associated with the formation of nickel aluminides have been used successfully to obtain Ni_3Al and

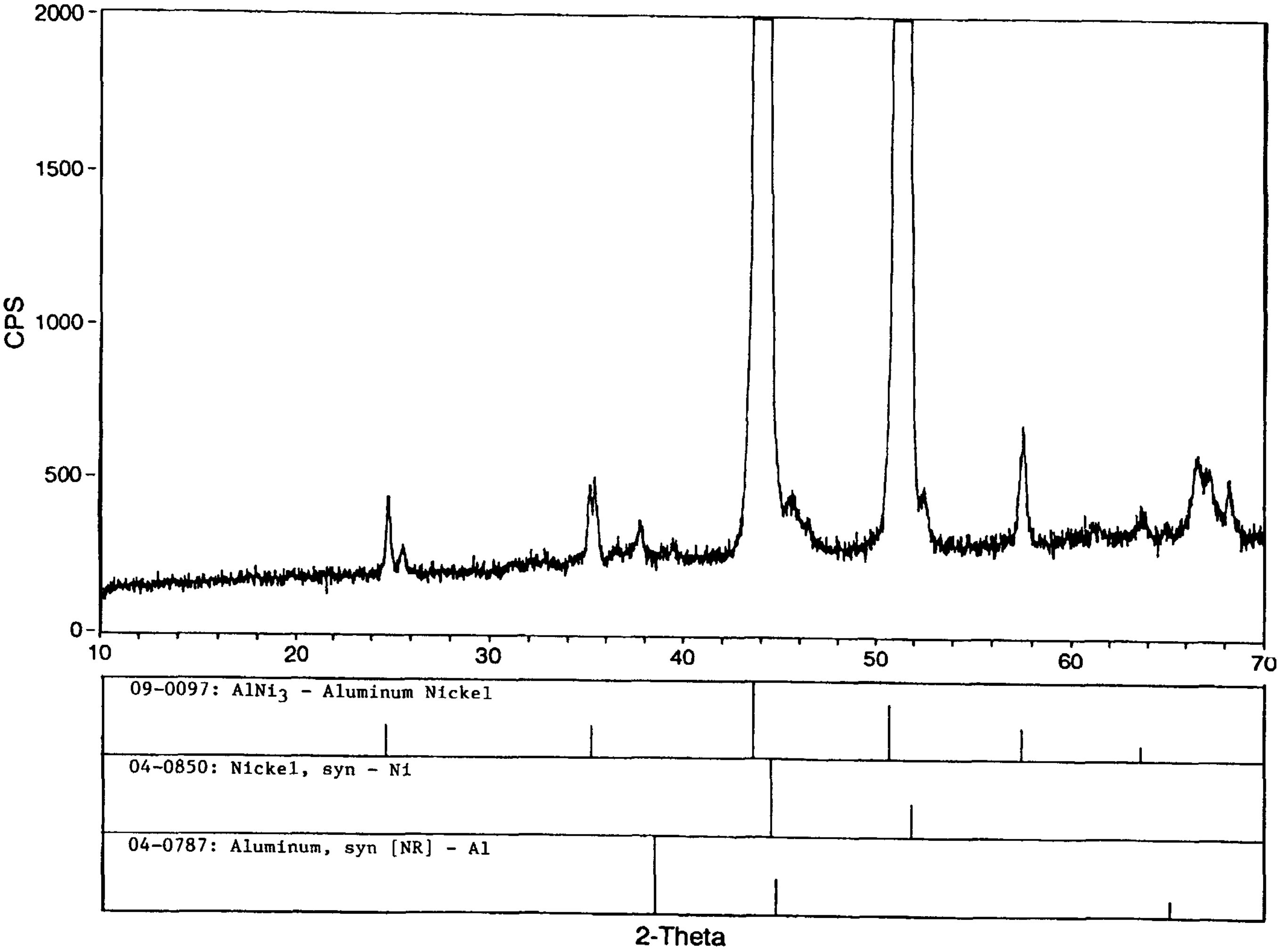


Fig. 10 XRD pattern of as-sprayed coating (obtained from bond coat Ni/Al powder) after heating at 600 °C for 1 h, 800 °C for 1 h, and 1100 °C for 1 h

NiAl by the combustion synthesis (also known as reaction synthesis) technique. In this technique, the ignition of elemental powders (in the ratios corresponding to Ni_3Al or NiAl) led to propagation of a combustion wave resulting in either Ni_3Al or NiAl phases. Particle sizes of nickel and aluminum played a dominant role in the formation of the nickel aluminides, and mixed phases were present when the particle size of aluminum was increased by keeping the particle size of nickel constant at 6 μm (Ref 17). Increasing the particle size of aluminum lowered the combustion velocity and also decreased the combustion temperature due to the partial melting and reaction of aluminum as the wave propagated through the sample.

The heating rates observed in the reaction synthesis of NiAl are at least 100,000 K/min; therefore, melting of aluminum and exothermic diffusional reactions must take place within the residence time available during the propagation of the front. In contrast, the powders are fed through an intense heat source either at subsonic or sonic velocities in plasma spraying, and such high velocities result in significantly lower residence times for the initiation and occurrence of exothermic diffusional reactions between aluminum and nickel in reactive spraying. Even though powders of approximately 15 μm in size can be fed through a plasma gun, larger powder particle sizes are typically used in plasma spraying due to their ready availability. Larger particle sizes of nickel and aluminum, in the range of 30 to 60 μm, ensure consistent and uniform flow through the plasma gun. Even though the particle sizes are larger, and residence times are shorter in plasma spraying, the temperature of the plasma is sufficient to bring the particles close to their melting points. Therefore, one would expect the formation of nickel aluminide phases exothermically. Lower residence times limit interfacial contact; thus, the exothermic diffusional reactions may take place only after the molten splats of aluminum and nickel are deposited on the substrate.

The phases observed in the thermal explosion mode of combustion synthesis (Ref 23) of nickel aluminides compare to those observed in the present investigation. For example, Philpot et al. (Ref 23) noted the presence of Al, $NiAl_3$, Ni_2Al_3, and Ni prior to the final exotherm, and Ni remained even after heating the powder mixture to 900 °C or above. Interestingly, aluminide phases with high aluminum content disappeared only on heating, and Ni_3Al became a secondary phase while the unreacted nickel still remained in the heated sample. Herman et al. (Ref 7, 24) also observed several nickel aluminide phases even when blended powders were vacuum plasma sprayed, and NiAl phase was observed only after annealing the coating at 1100 °C for 4 h. These authors also observed secondary phases after vacuum plasma spraying of mechanofused samples. In combustion synthesis and in reaction synthesis techniques, melting of aluminum and its subsequent reaction with solid nickel results in the formation of nickel aluminide phases. In reactive spraying, the reactions may take place between molten splats of aluminum and nickel after they are deposited on a substrate.

Based on these findings, it is possible to write the following reactions for the exothermic formation of nickel aluminides during reactive spraying of nickel and aluminum powders:

$$3Al_{(l)} + Ni_{(l)} \rightarrow NiAl_{3(s)} \quad \text{(Eq 1)}$$

$$3Al_{(l)} + 2Ni_{(l)} \rightarrow Ni_2Al_{3(s)} \quad \text{(Eq 2)}$$

$$3Al_{(l)} + 5Ni_{(l)} \rightarrow Ni_5Al_{3(s)} \quad \text{(Eq 3)}$$

$$Al_{(l)} + 3Ni_{(l)} \rightarrow Ni_3Al_{(s)} \quad \text{(Eq 4)}$$

$$Al_{(l)} + Ni_{(l)} \rightarrow NiAl_{(s)} \quad \text{(Eq 5)}$$

Intermetallic phases may also form due to secondary reactions between an aluminide phase and nickel as shown below:

$$NiAl_{3(s)} + Ni_{(s)} \rightarrow Ni_2Al_{3(s)} \quad \text{(Eq 6)}$$

$$Ni_2Al_{3(s)} + Ni_{(s)} \rightarrow 3NiAl_{(s)} \quad \text{(Eq 7)}$$

$$Ni_2Al_{3(s)} + 3Ni_{(s)} \rightarrow Ni_5Al_{3(s)} \quad \text{(Eq 8)}$$

$$NiAl_{(s)} + 2Ni_{(s)} \rightarrow Ni_3Al_{(s)} \quad \text{(Eq 9)}$$

As Table 1 and Fig. 11 show, the heat of reaction associated with the formation of these intermetallic phases varies depend-

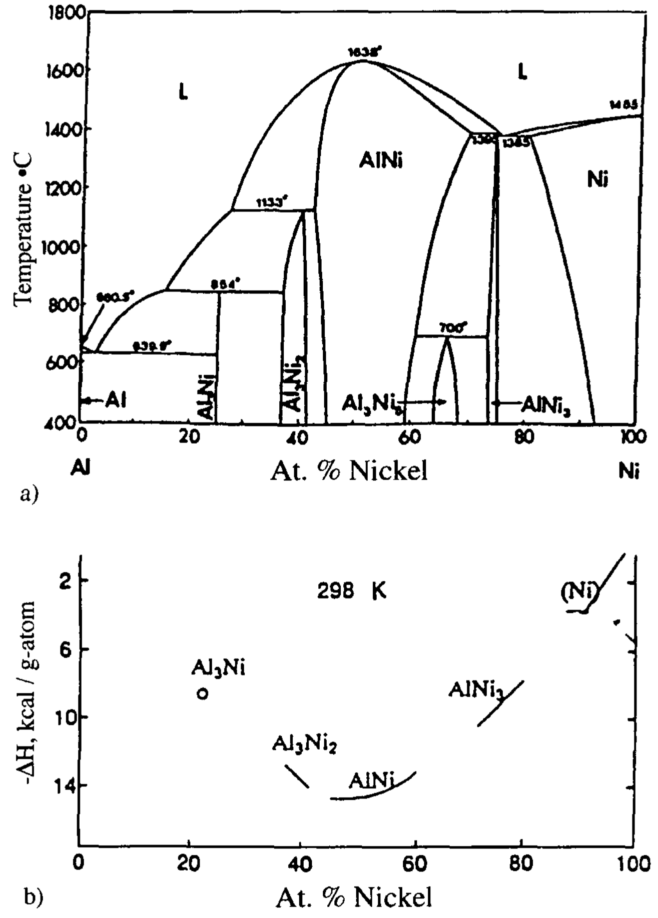


Fig. 11 (a) Phase diagram of Ni-Al. (b) Heats of formation associated with nickel aluminide phases

ing on the atomic percentage of nickel in the intermetallic phase. The maximum heat is released during the formation of NiAl phase (Ref 22). Formation of multiple intermetallic phases suggests that the reactions are diffusional controlled, and the formation of the initial product layer controls the diffusion of aluminum through the product layer. Diffusion of aluminum is crucial in the formation of aluminides, and it has been well established that aluminum is the dominant diffusing species in the aluminides. One possible explanation for the formation of aluminum-rich nickel aluminides may be that the splats of nickel and aluminum react exothermically and solidify extremely quickly (due to the high cooling rates) after deposition on the substrate. The high rates of solidification coupled with the formation of a product layer prevent any further reaction.

Our results suggest that the exothermic formation is completed after the molten particles are deposited as splats on the substrate, and the major part of the reaction may be occurring on the substrate. This is consistent with the observations by Chen and Herman (Ref 24). The initial formation of several aluminum-rich nickel aluminide phases suggests that it is difficult to closely control the composition of nickel and aluminum to tailor the desired coating. The phase formation can be controlled by adjusting the flow rates of the powders and the power levels, and by increasing the residence times. Also, the lack of a significant Ni_3Al phase, even in an as-sprayed coating from a bond coat material where nickel and aluminum are in contract with each other, suggests that the conditions must be carefully controlled to obtain intermetallic phases by reactive spraying.

The lack of nickel and aluminum in the proper ratio (at the interfaces of splats) prevents the formation of a desired nickel aluminide even after annealing. The splat thickness, length, and width are much larger than the particle diameters of the powders used in combustion synthesis experiments. In reactive spraying (unlike combustion synthesis), loss of aluminum may occur, and aluminum loss may also prevent the formation of a desired phase. Therefore, particle sizes and experimental conditions must be controlled to facilitate the interdiffusion of nickel and aluminum in the gun. Two different types of plasma plumes were noticed during spraying, and merging the plumes may lead to the initiation of the exothermic reaction in the plume. Reduction of particle sizes and splat thicknesses may favor formation of Ni_3Al and NiAl coatings by reactive spraying; therefore, further work will be carried out with a high-velocity oxyfuel spraying with fine powders of nickel and aluminum.

Acknowledgments

This work was carried out while S.C. Deevi was on a sabbatical at the Oak Ridge National Laboratory under the Philip Morris Fellowship Program. The authors thank R.L White, R.T. Ratcliff, and C.E. Hatfield for their help during the experimental work, and K. Spence for editing.

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