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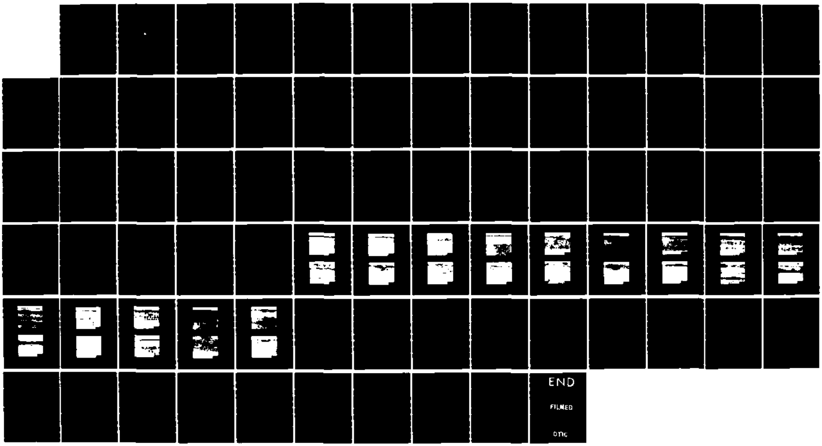

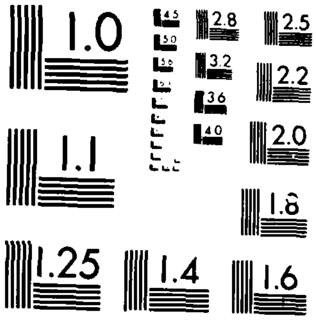
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NAVAL POSTGRADUATE SCHOOL

Monterey, California

THESIS

EFFECT OF CHROMIUM ADDITION TO THE LOW TEMPERATURE HOT CORROSION RESISTANCE OF PLATINUM MODIFIED ALUMINIDE COATINGS

by

Mark W. Dust
December 1985

Thesis Advisor: D.H. Boone

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Effect of Chromium Addition to the Low Temperature Hot Corrosion Resistance of Platinum Modified Aluminide Coatings

by

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ABSTRACT

Marine gas turbines face many adverse conditions such as reduced fuel quality and a salt environment which present the sulfur, chloride, and sulfates required to initiate and propagate hot corrosion. A particularly severe type of hot corrosion is low temperature hot corrosion (LTHC) encountered at the low temperatures (600-750°C) used for low power destroyer operations. Platinum-aluminides have demonstrated great success as protective coatings which delay the onset of high temperature hot corrosion attack (800-1000°C). Chromium is known to provide good LTHC resistance. The effect of chromium addition to platinum-aluminide coatings was investigated using two different nickel-base superalloys, IN-738 (16% Cr) and IN-100 (10% Cr).

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I. INTRODUCTION

A. MARINE GAS TURBINES AND HOT CORROSION

Although gas turbine engines have been in common use since the early 1950's, it has been only in the past fifteen years that the United States Navy has begun selecting gas turbines as propulsion plants for new surface combatants. Gas turbines offer many advantages as a marine propulsion engine: 1) high performance, 2) compact installation, 3) rapid start from cold iron, 4) high reliability, 5) simple maintenance, and 6) minimum smoke [Ref. 1]. However, marine gas turbines face many conditions such as harsher environments and decreased fuel quality which were not encountered in previous use. The effect of these adverse conditions were investigated in 1969 when the United States Navy selected the LM2500 gas turbine for the SPRUANCE class destroyers and started operational testing on the GTS CALLAGHAN.

It was found that the first and second stage high pressure turbine blades and vanes, which are usually the limiting components of gas turbine engines, had drastically reduced lifetimes when operated at temperatures above 800°C. In industrial use, the same components lasted up to five times longer. Further testing also indicated that when the engines were operated at a lower power consistent with usual destroyer operations, the turbine blade lifetimes were reduced even more at metal temperatures from 600-730°C. The decreased lifetimes were found to be caused by hot corrosion which is an aggressive attack on the substrate resulting from the combination of normal oxidation, high operating temperatures, and the presence of contaminants such as sulfur, sulfates, and chlorides from ingested fuel and air. [Ref. 2]

Three methods may be used to increase hot corrosion resistance. The first is to prevent the presence of contaminating substances by using high quality fuels and improved filtration systems. It would be extremely expensive to provide fuel of sufficient quality to give any significant resistance. It would also be very impractical to commit naval ships to high grade fuels which may not be readily available in emergency situations.

The second option is to improve the hot corrosion resistance of the turbine blade material itself. Superalloys, the material used for turbine blades, encompass a large group of metals which have the ability to maintain strength and resist deformation under extreme heat. Superalloys can have nickel, cobalt, or iron as the principal constituent. Nickel-based high temperature alloys are generally used for the pivotal first and second stage turbine blades and vanes.

Initial nickel-base superalloys had high chromium contents. To increase their strength at high temperatures, the aluminum and titanium contents were increased with a concomitant decrease in chromium content. However, as the chromium content decreased and the operating temperatures rose, hot corrosion resistance decreased. At this time no suitable combination has been found which gives superalloys the necessary high hot corrosion resistance and required strength at high operating temperatures, although research programs continue in this area. [Ref. 3]

The third option to improve hot corrosion resistance is to use protective coating systems. The trend has continued for superalloys to become increasingly temperature-capable. The corresponding decrease in hot corrosion resistance has resulted in coatings being given the most attention as a viable method to retard hot corrosion. [Ref. 4]

B. LOW AND HIGH TEMPERATURE HOT CORROSION

Unfortunately, protective coatings do not encounter just one type of degradation mechanism when it comes to hot corrosion resistance. High temperature hot corrosion (HTHC) occurs at temperatures in the 800-1000°C range. HTHC is also referred to as Type I hot corrosion because it was the first type of hot corrosion that was encountered. Coatings which provide HTHC resistance have been available for many years. Low temperature hot corrosion (LTHC) occurs in the

600-750°C range and is also referred to as Type II hot corrosion. As noted earlier, testing on GTS CALLAGHAN indicated corrosion rates that were much greater for the lower range of operating temperatures. This was contrary to expectations at the time which were that the corrosion rates would be negligible at these lower temperatures. The operating temperatures of marine gas turbines necessitated the development of protective coatings which would give LTHC resistance without sacrificing required resistance at higher temperatures. [Refs. 5,6]

The development of protective coatings necessitates an understanding of the mechanisms of hot corrosion in superalloys. Both types of hot corrosion usually entail a two stage process. The first stage, initiation, is essentially identical in both types. This stage does not require the presence of the contaminating substances of sulfates and sulfur associated with hot corrosion. It proceeds in a manner similar to simple oxidation degradation, although at a faster rate.

Initially, the elements in the thin surface layer are oxidized. Chromium and aluminum diffuse to form an internal oxide layer underneath the external scale. The composition of these layers depends on the composition of the superalloy itself. The internal oxide layer with chromium or aluminum forms a protective barrier which is replenished by further diffusion from the substrate interior. The initiation stage ends when the chromium and aluminum have been sufficiently depleted so that the barrier is penetrated. The rate at which the initiation stage proceeds is dependent on such factors as alloy composition, alloy surface conditions, gas environment, and cracking of the oxide scale.

Then the second stage, propagation, begins at a rate much faster than initiation. The major objective of hot corrosion protection systems is to delay the onset of the propagation stage. Several propagation modes may occur depending on the effect of contaminating deposits at the surface of superalloys. These modes may be separated into two general categories, those involving a component from the deposit and those involving salt fluxing reactions.

When marine gas turbines are operated, sodium sulfate, sodium chloride, and other contaminants from the combustion gases, low quality fuels, and salt air are deposited on the turbine blades and vanes. The sulfur and chlorine from these deposits form non-protective surface scales which greatly enhance the hot corrosion rate. Sulfur induced degradation, also called sulfidation, was one of the first hot corrosion mechanisms to be encountered. Chlorine induced degradation promotes increased hot corrosion rates by causing the protective oxides to form as particles and not as layers. This makes them more susceptible to cracking and spalling.

The salt fluxing reactions can be basic or acidic. Basic fluxing involves the reaction of the protective oxide scale with oxide ions generated by dissociation of sodium sulfate in the deposit. For basic fluxing to maintain its corrosive attack, the sodium sulfate must be continually renewed. Basic fluxing is not generally considered to be as devastating as acidic fluxing.

Where basic fluxing involves the reaction of oxide ions generated by the deposit with the protective oxide layer, acidic fluxing involves the donation of oxide ions to the deposit from the protective oxide layer. There are two types of acidic fluxing: alloy induced and gas phase induced. Alloy induced acidic fluxing occurs when the refractory elements, molybdenum, tungsten, and vanadium from the superalloy form oxides in the sodium sulfate deposit. The refractory element oxides cause the deposit to become acidic and allows the accelerated hot corrosion attack to become self-sustaining without the necessity for additional sodium sulfate. Gas phase induced acidic fluxing occurs when the presence of an acidic component of the gas (sulfur trioxide) results in a deficiency of oxide ions in the sodium sulfate deposit. The protective oxide layer breaks down because it is contributing required oxide ions to the deposit. This type of acidic fluxing requires a constant supply of sulfur trioxide. [Refs. 7,8]

All or some of these mechanisms may be present in the hot corrosion of a specific superalloy under certain conditions. However, sulfur and chlorine induced degradation, basic fluxing, and alloy induced acidic fluxing are normally significant only at temperatures above 850°C, the HTHC region. Gas phase induced acidic fluxing is dependent upon the presence of sulfur trioxide. The higher the temperature, the lower the sulfur trioxide pressure. Therefore, gas phase induced acidic fluxing is generally associated with lower temperatures, 650-750°C, and is considered to be the principal mechanism for LTHC. A summary of the hot corrosions mechanisms can be found in Table I [Ref. 9]

There are other differences in LTHC and HTHC besides their mechanisms. The appearance and rate of attack differ as well. HTHC attack gives the metal surface a rough, mottled appearance from the presence of sulfide extrusions. LTHC is characterized by a pitting attack [Ref. 10]. HTHC attack occurs at rates which are much less than those occurring for LTHC attack. Figure B.1, [Ref. 11], demonstrates the relative rates of the two types of attack.

C. PROTECTIVE COATINGS

The variety of mechanisms and temperature ranges for hot corrosion presents severe problems for the development of hot corrosion resistant coatings. The difficulties do not end here. In addition to hot corrosion resistance, coatings must have sufficient ductility to prevent cracking, a compatible thermal expansion with the superalloy substrate, low interdiffusion rates between coating and substrate, practical methods for application, and provide a significant increase in substrate lifetime economically. Two major types of coatings, diffusion aluminides and overlays, have been found to fulfill these requirements. [Ref. 12]

Diffusion aluminide coatings were the first coatings developed for hot corrosion resistance. Aluminum is applied to the surface of the superalloy by a variety of methods, dip aluminizing, forced flow gas phase aluminizing, static gas phase aluminizing, or most commonly, pack cementation. A layer of NiAl is formed on the surface after interdiffusion takes place. The NiAl forms protective aluminum oxide upon oxidation. Although diffusion aluminide coatings provide some hot corrosion resistance, they did not do well under severe hot corrosion conditions and may degraded the surface mechanical properties of the superalloy itself. [Refs. 13,14]

Overlay coatings were developed to overcome these problems. Extensive interdiffusion does not take place so that the structure and composition of overlay coatings can be varied independently of the substrate. Increased hot corrosion resistance and higher ductility to avoid cracking were obtained without the degradation of substrate mechanical properties. Unfortunately, overlay coatings have not solved all problems. Their complex application techniques and relatively high cost have prevented them from becoming the universal coating. Diffusion aluminide coatings, particularly those modified by selective additional elements, have received renewed interest because they are easier to apply and much more economical. [Ref. 15]

Diffusion aluminide coatings, whether modified by element additions or not, are generally classified as having an "inward" or "outward" coating structure. Inward structures are formed by conducting the aluminizing treatment in high activity, aluminum-rich packs at low temperatures (about 700-950°C). The aluminum diffuses with result being a high aluminum gradient in the Ni-Al coating. Outward structures are formed by conducting the aluminizing treatment in low activity, aluminum-poor packs at high temperatures (about 1000-1100°C). Nickel diffuses outward from the substrate with the result being a low aluminum gradient in the Ni-Al coating. In both cases, the aluminizing treatment is followed by a diffusion treatment (about 1050-1200°C). [Ref. 16]

Many elements have been used to modify diffusion aluminide coatings. The most beneficial effects have been gained from the addition of platinum or chromium. Generally the modified aluminide coating is made by a two step deposition process. First, a layer of the modifying element (platinum or chromium) is added to the substrate and diffused. Then the aluminum is added by one of the processes listed earlier. The coating microstructure is controlled by varying the amount of deposition and diffusion times and temperatures.

Platinum was first added to aluminide coatings with the idea that platinum would act as a barrier to aluminum diffusion into the substrate. This would keep more aluminum at the surface to replenish the aluminum oxide layer which resists hot corrosion and oxidation. It was found that platinum modified aluminide coatings did have a greater high temperature hot corrosion resistance than simple aluminide coatings. This increased resistance was not due to platinum acting as a barrier to aluminum diffusion, however, because compositional profiles of the platinum-aluminide coating indicated that the platinum was concentrated at the coating surface [Ref. 17]. Further research has demonstrated that platinum improves HTHC resistance possibly in part by increasing the adherence of aluminum oxide to the coating surface, although the exact mechanism is still not known [Ref. 18]. The HTHC resistance of platinum-aluminides does not mean that they are resistant to LTHC as well. Platinum addition was found to specifically inhibit the basic fluxing mechanism of HTHC, but it did not help to inhibit the gas phase induced acidic fluxing mechanism of LTHC. A platinum modified aluminide coating was found to be resistant to LTHC acidic fluxing only if a "critical platinum-aluminum phase (possibly $PtAl_2$) is continuous at the surface" [Ref. 19]. In general, the addition of platinum to diffusion aluminide coatings greatly improves HTHC resistance, but does not significantly affect LTHC resistance.

Chromium was one of the first modifying elements added to aluminide coatings because its benefits to hot corrosion resistance have long been recognized. The beginnings of coating development can be traced to the point where a decrease in chromium content of superalloys was made to obtain an increase in superalloy high temperature capability, but resulted in an increase in hot corrosion rates as well. Chromium provides LTHC resistance because it forms chromia [chromium oxide] as a protective scale. Chromia does not provide practical HTHC resistance because it volatilizes to chromium trioxide at temperatures above 800°C. Still, chromium does contribute to HTHC resistance by decreasing the amount of aluminum required to form aluminum oxide in nickel-aluminum systems. [Ref. 20]

Attempts have been made to combine the beneficial effects of chromium and platinum by incorporating both of them in diffusion aluminide coatings in an attempt to balance the resistance to both LTHC and HTHC degradation.

Two of these "chromium modified platinum-aluminide" coatings were placed on two different substrates, low chromium content IN-100 (10%) and high chromium content IN-738 (16%). The microstructure of these coatings, along with several base line chromium modified aluminides and platinum modified aluminides, were analyzed. An accelerated LTHC test was performed on the coatings to determine the relationship of microstructure to LTHC resistance.

II. EXPERIMENTAL PROCEDURE

A. BACKGROUND

Many methods can be used to conduct hot corrosion attack studies. Because turbine blade lifetimes are roughly 5000 hours and longer in normal gas turbine use, most of these studies utilize accelerated tests. The closer the test approximates actual hot corrosion conditions in an operating gas turbine, the more complex, expensive, and time consuming the test becomes.

Pressurized burner rigs and simple burner rigs are two common methods which are used. A pressurized burner rig is the best and also the most complex method for simulating hot corrosion conditions. It simulates these conditions and accelerates testing time by controlling the pressure, velocities, composition, and temperature of the hot corrosion gas environment. The use of simple burner rigs, which are unable to control the gas pressure and velocities, greatly reduces the cost of equipment. Higher contaminant levels are used to accelerate the testing time. [Ref. 21]

A third method of hot corrosion testing which involves less complex equipment and lower cost is the use of a laboratory furnace. In this test, attempts are made to duplicate the actual corrosive conditions which occur at the surface of the airfoil in the engines and not the circumstances which led to these conditions being present. Samples are covered with a thin film of contaminating salt and inserted in the furnace. An air/sulfur dioxide gas mixture flows through the furnace which is set at the temperature of interest. LTHC and HTHC testing are conducted in a similar manner except that the weight of the salt film and furnace temperature are different. This method of hot corrosion testing accelerates attack because the application of the salt film greatly reduces the time required for the initiation stage of hot corrosion. The laboratory furnace is able to produce LTHC attack in about 60 hours with results in the form of degradation morphology and relative ranking which compare favorably to those acquired by pressurized and simple burner rigs. [Ref. 22]

B. HOT CORROSION TESTING

The specimens in this study consisted of seven different coatings, each applied on two different superalloy substrates. The fourteen specimens are listed in Table II along with a brief description of the coating manufacturing process. The two superalloy compositions are listed in Table III.

The specimens were received as pins with a diameter of about 0.6 cm. and were cut to a length of about 1.5 cm. The surface area of each pin was determined and then the pin was heated in an oven at 170°C for fifteen minutes to evaporate any moisture. The specimens were reheated at 170°C for about ten minutes to facilitate the application of an even film of salt. A salt solution with a concentration of 63.1 g. sodium sulfate/ 39.1 g. magnesium sulfate in one liter of water was dropped on the specimen. The specimen was heated again to evaporate the water and reweighed. The salt treatment was repeated until there was roughly 1.5 mg. of salt per square cm. of specimen surface.

After all specimens were properly salted, they were placed in the laboratory furnace at a temperature of 700°C. An air (2000 ml./min.): sulfur dioxide (5 ml./min.) mixture flowed through the furnace. The specimens were removed after 20 hours, resalted, and placed back into the furnace. Three cycles of 20 hours each were completed for a total hot corrosion test time of 60 hours.

Both the tested specimens and as-received specimens were cut, mounted, and polished in accordance with standard metallographic procedures. The hot corrosion tested specimens were examined under an optical microscope to determine the attack penetration depth. Depth of penetration measurements were measured at 20° intervals around the perimeter of the entire specimen. It was determined that LTHC attack after 60 hours was not very significant for this series of specimens. The 60 hour cut specimens were resalted and run for two additional cycles for a total of 100 hours. Another set of specimens was run for 100 hours following the given hot corrosion testing procedure. The LTHC test data is listed in Table IV .

A scanning electron microscope (SEM) was used to take photographs of the mounted specimens. These photographs, which can be found in Figures B.2-B.15, show the following: a) the coating prior to hot corrosion testing and b) the pitting which resulted from LTHC attack. Continuous electron microprobe scans were made on the as-received specimens to determine the nickel, aluminum, platinum, and chromium element composition (as a weight percent) of the coating prior to hot corrosion testing. The nickel and chromium compositions were adjusted to reflect their known compositions in the superalloy substrates. The aluminum compositions have not been adjusted, but are known to have registered lower weight percent levels because of interference from a thin gold film which was required for conductivity in the SEM. The microprobe scan plots can be found in Figures B.16-B.29.

III. RESULTS AND DISCUSSION

A. MICROSTRUCTURE

The microstructure of the fourteen specimens were analyzed using the SEM photographs and microprobe scan data as shown in Figures B.2-B.29. Exact phase identification was not possible because of the lack of appropriate phase diagrams and X-ray diffraction data. The structures of platinum-aluminides and chromium-aluminides have previously been examined. However, there is a dearth of information on chromium modified platinum-aluminides which made structure analysis extremely difficult.

The composition difference between the two substrates did not affect the general structure of the coatings. It can be seen in Table III that the principal difference between the two superalloys is the chromium content (16% for IN-738 and 10% for IN-100). This difference was not enough to cause the structures to be radically different although it did affect the relative composition levels of elements within the general coating structure. The main differences in structure could be attributed to manufacturing process variations.

The platinum-aluminide samples on both substrates exhibited the classic microstructures associated with inward diffusion, low temperature high activity (LTHA) and outward diffusion, high temperature low activity (HTLA) diffusion aluminide coatings [Ref. 23]. The LTHA platinum-aluminide coatings (Figures B.2a and B.9a) have a single-phase, four-zone structure. The surface zone consists of a high platinum content Pt-Al outer layer. Exact phase identification is in question, although the literature mentions $PtAl_2$ and Pt_2Al_3. Present thinking is that $PtAl_2$ predominates. The outer intermediate zone consists of chromium and platinum rich precipitates in an NiAl matrix. The inner intermediate zone is a region of single-phase beta-NiAl, denuded of any other phases or substrate elements. The innermost zone is the so-called interdiffusion zone which consists of refractory metal elements and carbides from the substrate in a beta-NiAl matrix. [Ref. 24]

The HTLA platinum-aluminide coatings (Figures B.3a, B.4a, B.10a, and B.11a) have a two-phase, three zone structure. The surface zone consists of a thin layer of $PtAl_2$ with some NiAl, which covers a platinum-containing beta-NiAl matrix with $PtAl_2$ precipitates. The intermediate zone is a nickel-rich NiAl matrix with chromium-rich precipitates. The innermost zone is similar to the LTHA platinum-aluminide. It consists primarily of refractory metal carbides in a NiAl matrix. [Ref. 25]

The LTHA chromium-aluminide specimens exhibited microstructures consistent with those previously seen and discussed in past studies. These chromium-aluminide coatings (Figures B.5a and B.12a) have the standard three-zone structure associated with LTHA aluminides. It can be seen from Figures B.19 and B.26 that the outer zone has a high chromium content. However, because chromium has very low solubility in NiAl, the outer zone consists of a NiAl matrix enriched to the full extent of chromium's solubility (approximately 3 atom percent) with an alpha-chromium precipitate. The intermediate zone is single-phase beta-NiAl, denuded of phase and substrate elements. The innermost interdiffusion zone consists of carbides, primarily chromium carbides, in a NiAl matrix and is identical in structure to those previously discussed. [Ref. 26]

The HTLA chromium modified aluminide specimen structures varied drastically from the microstructures seen in the past. The classic structure has an outer layer of single-phase NiAl with the possibility of a few chromium particles from the pack mix embedded near the surface by the outward diffusion of nickel through NiAl. In general, the chromium is distributed in an underlayer between the NiAl and interdiffusion zone because of the nickel extraction and diffusion outward to react with the aluminum being deposited. [Ref. 26]

The two HTLA chromium-aluminide specimens (Figures B.6a and B.13a) did not have this classic structure. It can be seen from Figures B.20 and B.27 that here was a considerable amount of chromium at the surface, possibly a layer of alpha-chromium. Underneath this layer was NiAl, at first with little chromium, but then with quite substantial amounts of a chromium-rich precipitate which is probably alpha-chromium. The interdiffusion zone is similar to that found in the LTHA chromium-aluminide, but possibly with more chromium enrichment.

It must be remembered that the family of diffusion aluminide coatings is not constrained to a purely "outward" or "inward" type of structure. There is a range of possible structures between these two extremes which can be obtained by carefully and precisely varying the aluminum pack composition and the diffusion treatment. The two HTLA chromium-aluminide specimens exhibited a microstructure which had characteristics attributed to a combination of both classic outward and inward diffusion structures. They exhibited a chromium precipitate in a NiAl matrix and a high chromium surface layer as in a pure LTHA chromium aluminide. However, this chromium precipitate was concentrated more in the region near the interdiffusion zone rather than the region near the surface. This concentration of chromium near the interdiffusion zone is similar to the more narrow entrapment of chromium found in a pure HTLA chromium aluminide. The HTLA chromium-aluminide specimens have a hybrid microstructure which could be attributed to a variation in manufacturing procedure. This structure is apparently the result of the aluminum content being in the stoichiometric region where aluminum and nickel both diffuse at comparable rates. [Ref. 27]

The chromium modified platinum-aluminides have microstructures which are even more complex because of the addition of a second modifying element. They still follow the general inward or outward diffusion mechanisms and structures found in straight aluminides. The dearth of studies in the literature investigating chromium modified platinum-aluminide structures and a lack of phase identification and X-ray diffraction data, which was not in the scope of this study, makes a detailed analysis of these microstructures extremely difficult.

The two chromium modified platinum-aluminide coatings, designated Process B and Process D, differ primarily in the order in which the modifying elements, platinum and chromium, are applied. Process B has the coating elements applied in the following order: 1) platinum, 2) chromium, and 3) aluminum. Process D reverses platinum and chromium to get the following order: 1) chromium, 2) platinum, and 3) aluminum. In both cases investigated, the aluminizing process involves a HTLA outward diffusion type process.

Process B (Figures B.7a and B.14a) exhibits a three-zone structure which is similar to an outward diffusion formed platinum-aluminide. It can be seen from Figures B.21 and B.28 that the outermost zone appears to have a surface layer which is predominantly a chromium-rich Pt-Al phase (possibly $PtAl_2$) with some nickel. This Pt-Al phase grades into a two-phase $PtAl_2$/beta-NiAl structure with a chromium-rich precipitate dispersed throughout. These chromium-rich particles from the chromizing process may be alpha-chromium. There is no possibility of pack mix entrapment because all of the HTLA aluminizing processes in this study involved vapor deposition out of the pack. The intermediate zone is NiAl which is essentially free of precipitates. The interdiffusion zone appears to be primarily chromium carbides with some NiAl as previously described and discussed.

Process D has microstructures which differ greatly between the two substrates. This difference is probably the result of a variance in processing and not the substrates per se. Process D on IN-738 (Figure B.8a) has a fairly thick surface layer which appears from Figure B.22 to be nickel and chromium-rich $PtAl_2$ (although no data could be found in the literature identifying such a phase). The rest of the structure is similar to those displayed in the HTLA chromium-aluminide specimens. The intermediate zone appears to have a beta-NiAl matrix with an increasing amount of alpha-chromium precipitate. The interdiffusion zone is probably chromium and refractory metal carbides in NiAl. Again, this structure appears to be a combination of inward and outward diffusion structures apparently as a result of the particular aluminization process and diffusion treatment condition used.

Process D on IN-100 (Figure B.15a) has a thin surface layer of what appears to be $PtAl_2$ (Figure B.29). The rest of the structure is similar to that exhibited by classic HTLA chromium-aluminides. The intermediate zone is NiAl with a small amount of chromium particles near the surface. There is an interlayer where most of the chromium is trapped, then the diffusion zone with chromium carbides in NiAl. This structure, with the high and rather limited concentration of platinum and chromium at the surface, suggests an initial inward diffusion of aluminum which entraps the platinum and chromium at the surface followed by subsequent outward nickel diffusion. This results in chromium enrichment in the inner coating zone and a large denuded NiAl zone essentially free of platinum and chromium.

In the manufacturing of Process B, platinum is the first modifying element to be deposited. In the manufacturing of Process D, chromium is the first modifying element deposited. It is interesting to note that the structures of the resulting chromium modified platinum-aluminides is most closely related to the aluminide modified by the element which is first applied.

B. LOW TEMPERATURE HOT CORROSION TESTING

It can be readily seen from the LTHC test results in Table IV that the most resistant coatings for LTHC attack out of those tested were the HTLA platinum-aluminide, Process A (a second type of two-phase HTLA platinum-aluminide), and Process D (a chromium modified platinum-aluminide). These coatings provided excellent resistance regardless of which substrate was used. The LTHA chromium-aluminide did provide an equivalent resistance, but only for the structure that was applied to the lower chromium IN-100. The test results must be correlated with the microstructural analyses to determine why all of the coatings performed as they did. SEM photographs of the pitting attack resulting from LTHC are presented in Figures B.2b-B.15b. Note, these photos do not represent the average penetration depths or the worst or least hot corrosion areas, but are simply representative of the degradation morphology on each coating.

The LTHA platinum-aluminide did not display particularly good resistance to LTHC. Although it had a surface layer of $PtAl_2$ which does have improved LTHC resistance over the unmodified aluminides, once this barrier was breached, hot corrosion proceeded rapidly. This is illustrated by looking at the 60 hour results found in Table IV . At the end of 60 hours, the LTHA platinum-aluminide was showing resistance on a level with the HTLA platinum-aluminides. It must be noted that relative lifetime ranking is therefore a function of the point of time of examination.

The HTLA platinum-aluminide and Process A coatings did have a good resistance to LTHC attack. Underneath the primarily $PtAl_2$ surface layer was a thick two-phase zone of $PtAl_2$ in NiAl. Even after this surface layer was penetrated, the $PtAl_2$ precipitate provided some hot corrosion protection. In other testing, the continuous $PtAl_2$ layer has been reported as the most LTHC resistant structure with the two-phase $PtAl_2$/beta-NiAl structure performing little better than the unmodified aluminide. In these tests, lifetime may be related to the thickness and perfection of this continuous $PtAl_2$ surface layer.

The chromium-aluminides did not have much resistance to LTHC attack even though chromium has long been recognized as providing LTHC protection. This may be understood by looking at Figure B.26, the composition distribution for the LTHA chromium-aluminide on IN-100. This coating, the only chromium-aluminide to provide very good protection, was also the only coating to have an extremely high level of chromium at the surface. The other LTHA chromium-aluminide had a much higher average chromium composition throughout the coating, but it was not concentrated at the surface at such a high level. The HTLA chromium modified aluminides had fairly high chromium compositions near or at the interdiffusion zone. This is not sufficient for good LTHC protection because the high chromium composition must be at the surface for significant LTHC resistance.

The chromium modified platinum-aluminides differed greatly in LTHC resistance. Process D, which exhibited a good resistance, had a high $PtAl_2$ content with little nickel or chromium, again confirming the perception that a $PtAl_2$ surface layer is beneficial. Process B, which had poor resistance, had less continuous $PtAl_2$ with high amounts of nickel (probably as NiAl) and some chromium. In both coatings, the most important factor for LTHC resistance appeared to be the $PtAl_2$ content in the surface layer because neither coating had a high concentration of chromium at the surface. The order in which the modifying elements were applied greatly affected the structure. Applying chromium first and then platinum prior to aluminizing allows the platinum to form an effective layer of $PtAl_2$ at the surface, while applying the platinum first and then chromizing results in a lower platinum content in the surface layer upon subsequent aluminizing.

IV. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be made on the basis of the microstructural analyses and the specific LTHC testing performed on five baseline chromium or platinum modified aluminides and two chromium modified platinum-aluminides coated on two different substrates.

1. Chromium gives effective resistance to LTHC attack only when it is concentrated at a high critical composition level at the coating surface.
2. Because of the apparently high critical chromium level required for LTHC resistance, the slight differences in composition of the two substrates, IN-738 and IN-100, did not greatly affect either the coating structure or the coating resistance to LTHC.
3. Platinum gives effective resistance to LTHC attack when it is concentrated at the surface as $PtAl_2$ with little or no dilution by Ni. A dense reserve layer of $PtAl_2$ in NiAl does provide some additional protection.
4. The structure of the chromium modified platinum-aluminide is most dependent on the sequence of modifying element addition and hence the resulting structure and surface composition.

5. Chromium modified platinum-aluminides have good LTHC resistance when $PtAl_2$ is concentrated at the surface. This can apparently be accomplished only when chromium is added first and platinum is added second prior to HTLA aluminizing. When this order is reversed, the chromium can actually be detrimental to LTHC resistance by diluting the protection provided by $PtAl_2$.

6. Proper processing is required to ensure that the correct coating structures are obtained. The structure, and hence the LTHC resistance properties of the coating, can be affected greatly by variations in the manufacturing process.

This study was an initial attempt to provide some insight into the value of chromium modified platinum-aluminides. Although the two coatings involved in this investigations are obviously not panaceas to the problem of LTHC, one coating, Process D, did show some promise and warrants further investigation. Following are the recommendations for future studies:

1. Further alterations to chromium modified platinum-aluminide processing should be made and investigated. Some of these alterations may include:

a) The use of LTHA pack cementation methods for the final aluminizing treatment.

b) The use of chromizing as a post-aluminizing treatment.

2. The phases in the microstructures should be determined by X-ray diffraction to complement the analysis done by SEM photomicrographs and electron microprobe scans.

3. The specimens should be run in a HTHC test to determine the HTHC resistance of chromium modified platinum-aluminides. This information on behavior and degradation mechanisms will provide additional material to develop the overall understanding of the behavior of these unique and important protection systems.

APPENDIX A

TABLES I-IV

TABLE I

SUMMARY OF HOT CORROSION MECHANISMS

Possible Propagation Modes for Hot Corrosion of
Superalloys by Na_2SO_4 Deposits

I. Modes Involving
Fluxing Reactions

- Basic
- Acidic

II. Modes Involving
A Component of
the Deposit

- Sulfur
- Chlorine

I. Fluxing Modes.

A. Basic Processes

1. Dissolution of Reaction Product Barriers, (i.e. AO) Due to Removal of Sulfur and Oxygen from the Na_2SO_4 by the Metal or Alloy:

$$SO_4^{2-}\ \text{(sulfate deposit)} \rightarrow 1/2\ S_2\ \text{(for reaction with alloy)} + 3/2\ O_2\ \text{(for reaction with alloy)} + O^{2-}\ \text{(for reaction with AO)}$$

Reaction between AO and oxide ions can follow 2 courses:

(a) Continuous dissolution of AO

$$A(\text{alloy}) + 1/2\ O_2 + O^{2-} \rightarrow AO_2^{2-}$$

Na_2SO_4 is converted to Na_2AO_2 and attack is dependent on amount of Na_2SO_4 initially present.

(b) Solution and reprecipitation

$$A(\text{alloy}) + 1/2\ O_2 + O^{2-} \rightarrow AO_2^{2-}(\text{solution}) \rightarrow AO(\text{precipitate}) + O^{2-}$$

A supply of SO_3 is required in order for attack to proceed indefinitely, otherwise attack will stop when melt becomes sufficiently basic at precipitation site.

Table I
Summary of Hot Corrosion Mechanisms (cont'd.)

B. Acidic Processes

1. Gas Phase Induced

(a) Formation of ASO_4 in Na_2SO_4:

$$A(alloy) + SO_3 + 1/2\ O_2 \rightarrow A^{2+} + SO_4^{2-}$$

Continuous solution of ASO_4 in Na_2SO_4 requires continuous supply of SO_3 and O_2 from gas.

(b) Solution and Precipitation of AO in Na_2SO_4 Due to Reduction of SO_3:

$$A(alloy) + SO_3\ (from\ gas) \rightarrow A^{2+} + SO_3^{2-}\ (in\ melt)$$

$$A^{2+} + SO_3^{2-} + 1/2\ O_2\ (from\ gas) \rightarrow AO\ (precipitate) + SO_3$$

(c) Nonprotective Reaction Product Barrier formation due to rapid removal of base element (e.g. Co, Ni) from alloy by molten deposit (33).

(d) Solution and Precipitation of AO as a Result of Negative Gradient in Solubility of AO in Na_2SO_4 as in B.

2. Alloy Phase Induced

(a) Solution of AO in Na_2SO_4 Modified by Second Oxide from Alloy (i.e. BO_3).

Sequence:

i. Modification of Na_2SO_4 by BO_3

$$B(alloy) + 3/2\ O_2 + SO_4^{2-} \rightarrow BO_4^{2-} + SO_3$$

ii. Solution reaction for AO, Na_2SO_4 becomes enriched in ABO_4

$$A(alloy) + B(alloy + 2O_2 \rightarrow A^{2+} + BO_4^{2-}$$

or

iii. Solution and repricipitation

$$A(alloy) + B(alloy) + 2O_2 \rightarrow A^{2+} + BO_4^{2-} \rightarrow AO + BO_3$$

II. Salt Component Effects

A. Sulfur

$$A(alloy) + 1/2\ S_2\ (from\ deposit) \rightarrow AS$$

$$AS + 1/2\ O_2 \rightarrow AO\ (nonprotective) + 1/2\ S_2$$

B. Chlorine

$$A(alloy) + 1/2\ Cl_2\ (from\ deposit) \rightarrow ACl\ (gas)$$

$$ACl + 1/2\ O_2 \rightarrow AO\ (nonprotective) + 1/2\ Cl_2$$

TABLE II

LIST OF SPECIMENS

Coatings	Process
LTHA Platinum-Aluminide	1) Platinizing - Electroplate 2) Diffuse at 870C for ½ hour 3) Aluminizing - LTHA process * 4) Diffuse at 1080C for 4 hours
HTLA Platinum-Aluminide	1) Platinizing - Electroplate 2) Diffuse at 870C for 4 hours 3) Aluminizing - HTLA process ** 4) Diffuse at 1080C for 4 hours
Process A	1) Platinizing - Electroplate 2) Aluminizing - HTLA process 3) Diffuse at 1080C for 4 hours
LTHA Chromium-Aluminide	1) Chromizing - Pack Cementation at 1060C for 7 hours 2) Aluminizing - LTHA process 3) Diffuse at 1080C for 4 hours
HTLA Chromium-Aluminide	1) Chromizing - Pack Cementation at 1060C for 7 hours 2) Aluminizing - HTLA process 3) Diffuse at 1080C for 4 hours
Process B	1) Platinizing - Electroplate 2) Chromizing - Pack Cementation at 1060C for 7 hours 3) Aluminizing - HTLA process 4) Diffuse at 1080C for 4 hours
Process D	1) Chromizing - Pack Cementation at 1060C for 7 hours 2) Platinizing - Electroplate 3) Aluminizing - HTLA process 4) Diffuse at 1080C for 4 hours

* LTHA process in most industrial applications involves chemical vapor deposition in the pack at approximately 760C for 1 hour.

** HTLA process in most industrial applications involves chemical vapor deposition at 1080C for 4 hours. Specimens in this study were aluminized out of the pack.

TABLE III

COMPOSITION OF SUPERALLOY SUBSTRATES

Substrate	Ni	Cr	Co	Al	Ti	Mo	W	Other
IN-738	61.0	16.0	8.5	3.4	3.4	1.7	2.6	Ta=1.7
IN-100	60.0	10.0	15.0	5.5	4.7	3.0	-	V=1.0

TABLE IV

RESULTS OF LTHC TESTING

SPECIMEN IDENTIFICATION		DEPTH OF PENETRATION (microns)			
Substrate	Coating	Run 1 60 Hr	Run 2 100 Hr	Run 3 100 Hr	Avg of 100 Hr
IN-738	LTHA Platinum-Aluminide	28	58	48	53
IN-738	HTLA Platinum-Aluminide	27	42	37	40
IN-738	Process A	29	48	36	42
IN-738	LTHA Chromium-Aluminide	48	78	63	70
IN-738	HTLA Chromium-Aluminide	36	53	46	50
IN-738	Process B	32	52	56	54
IN-738	Process D	23	44	34	39
IN-100	LTHA Platinum-Aluminide	26	63	41	52
IN-100	HTLA Platinum-Aluminide	24	41	37	39
IN-100	Process A	28	42	34	38
IN-100	LTHA Chromium-Aluminide	26	36	38	37
IN-100	HTLA Chromium-Aluminide	42	66	65	66
IN-100	Process B	41	72	61	66
IN-100	Process D	27	26	41	34

APPENDIX B

FIGURES B.1-B.29

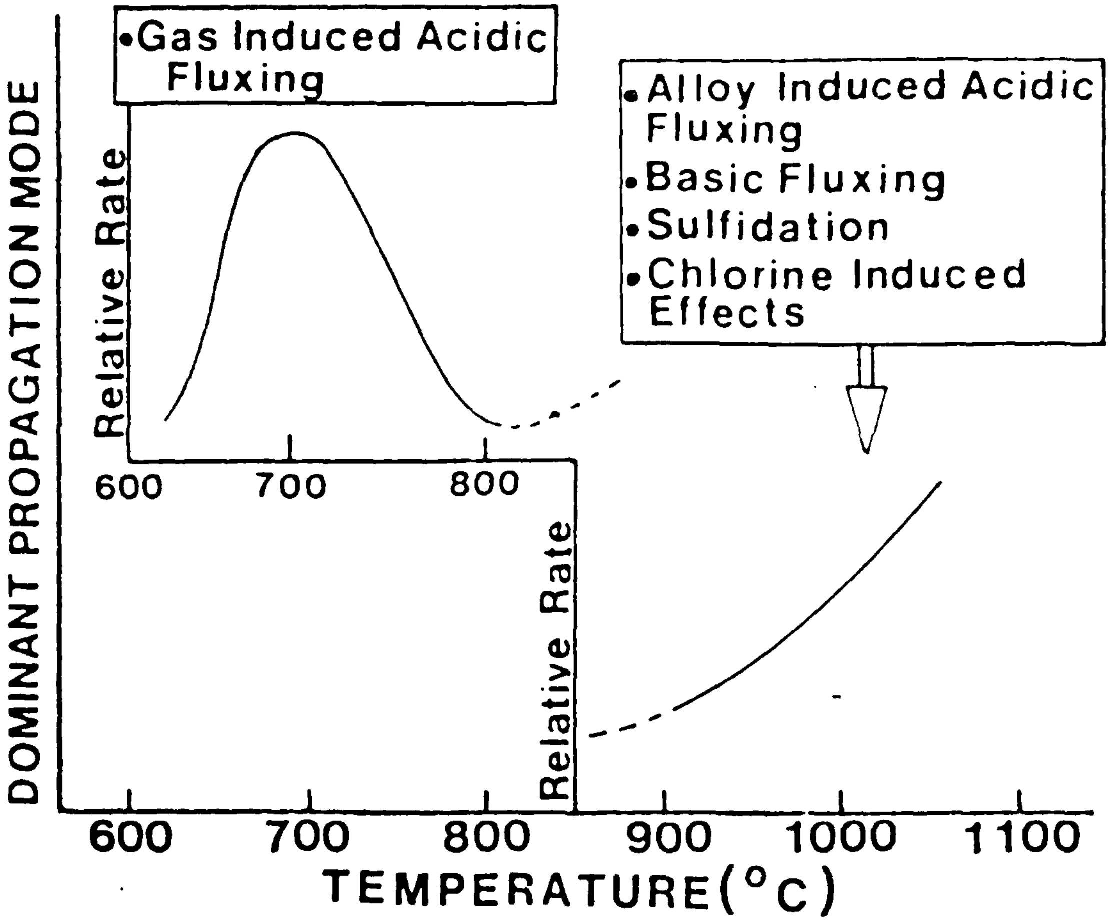

Figure B.1 Relative Rates of Attack.

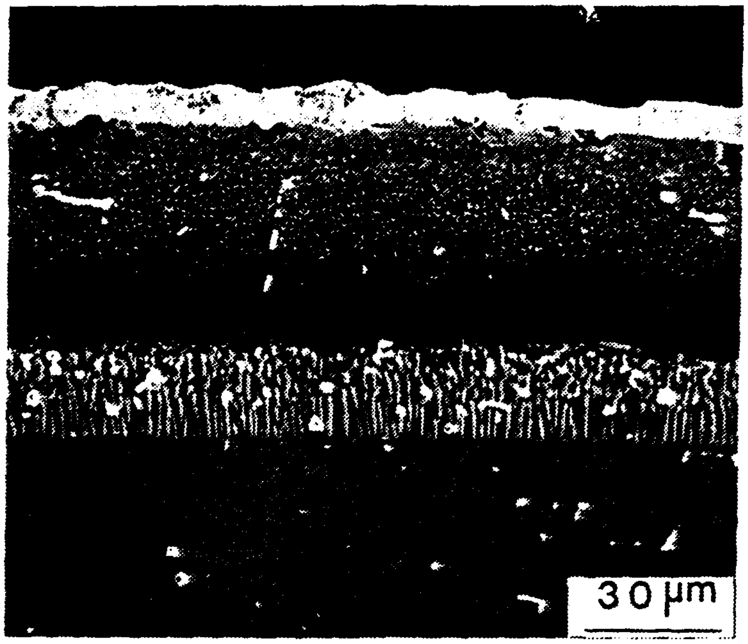

Figure B.2a LTHA Platinum-Aluminide / IN-738 (as-received).

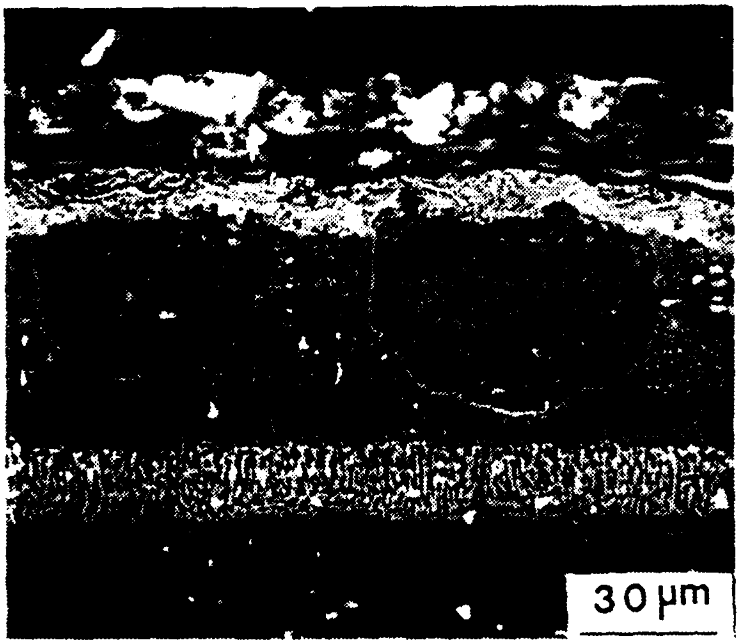

Figure B.2b LTHA Platinum-Aluminide / IN-738 (LTHC-100 hrs).

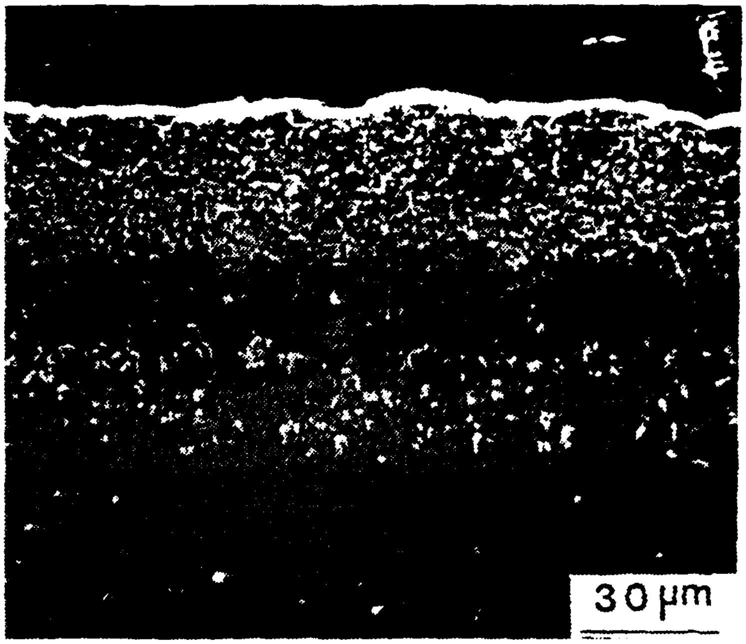

Figure B.3a HTLA Platinum-Aluminide / IN-738 (as-received).

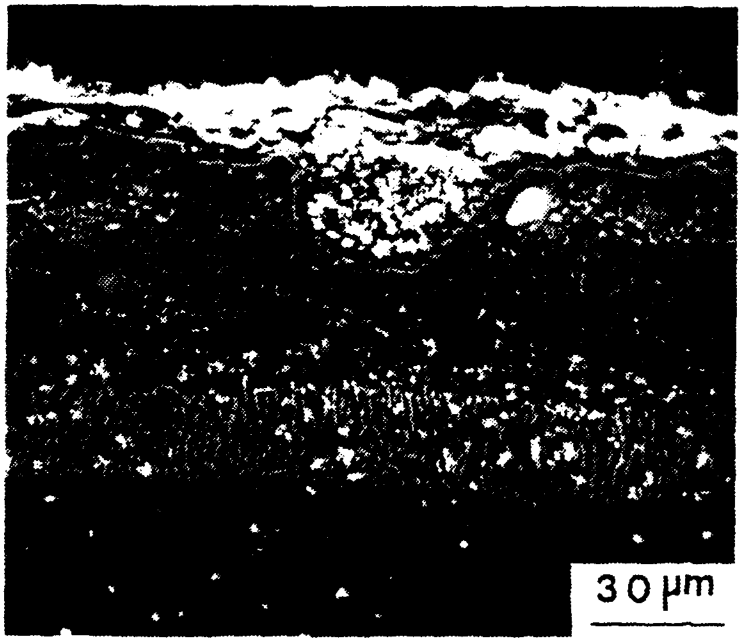

Figure B.3b HTLA Platinum-Aluminide / IN-738 (LTHC-100 hrs).

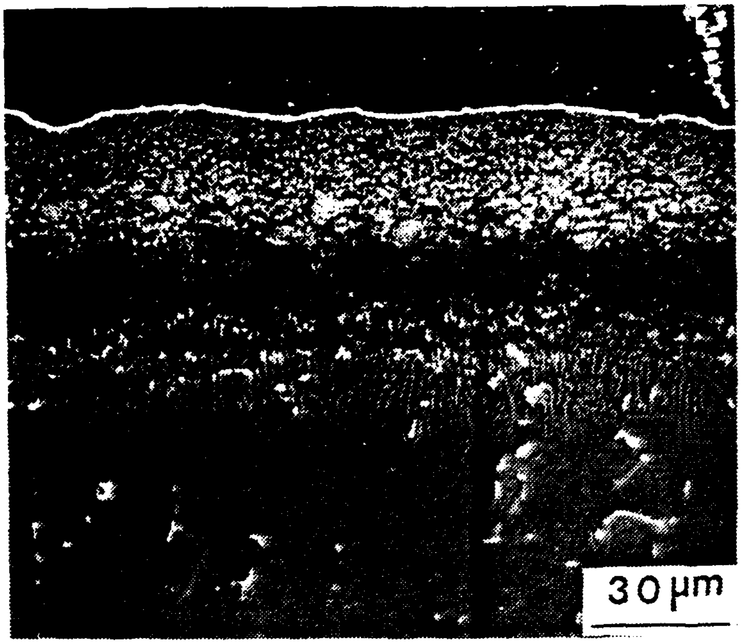

Figure B.4a   Process A / IN-738 (as-received).

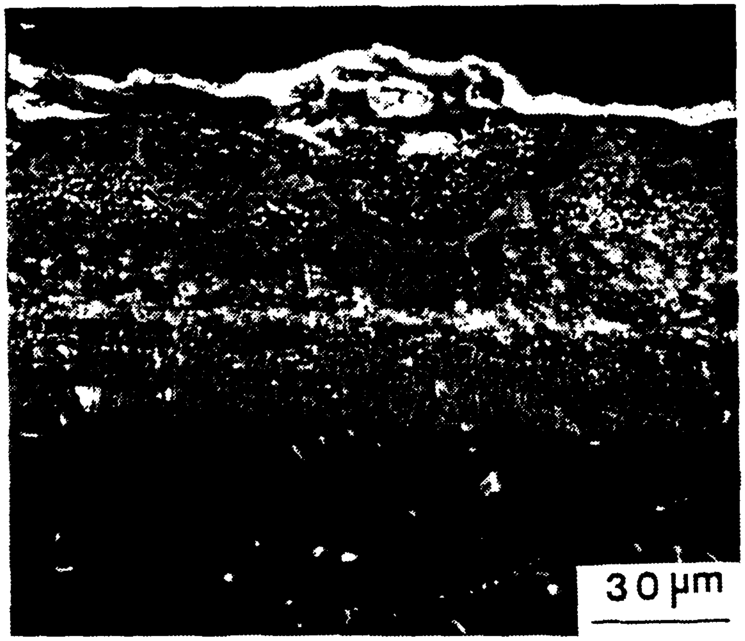

Figure B.4b   Process A / IN-738 (LTHC-100 hrs).

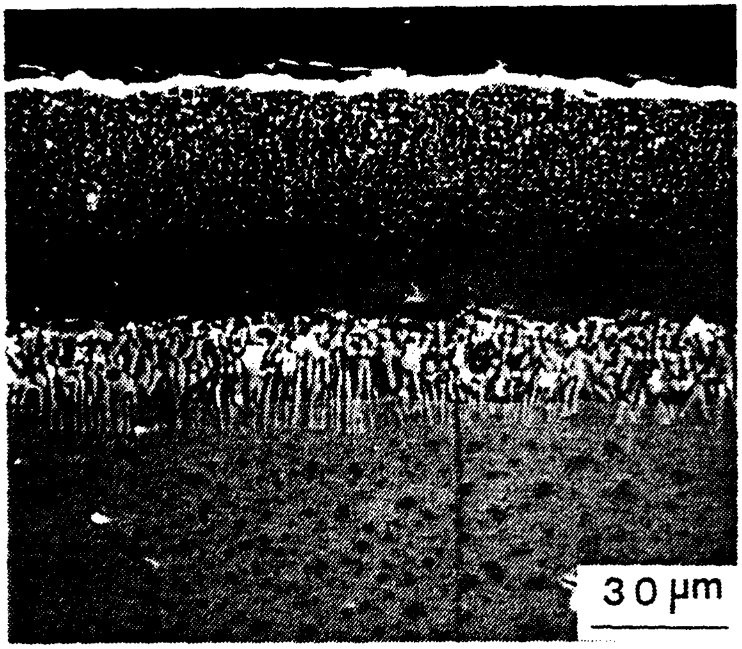

Figure B.5a LTHA Chromium-Aluminide / IN-738 (as-received).

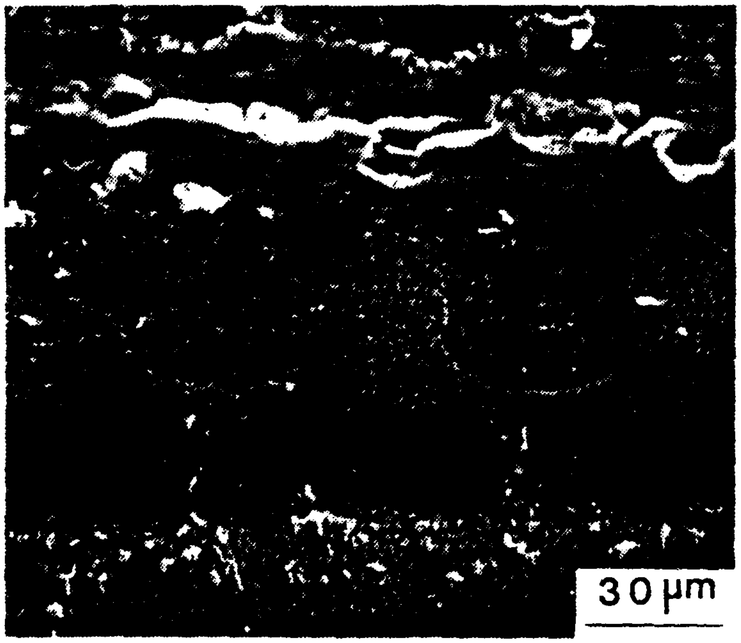

Figure B.5b LTHA Chromium-Aluminide / IN-738 (LTHC-100 hrs).

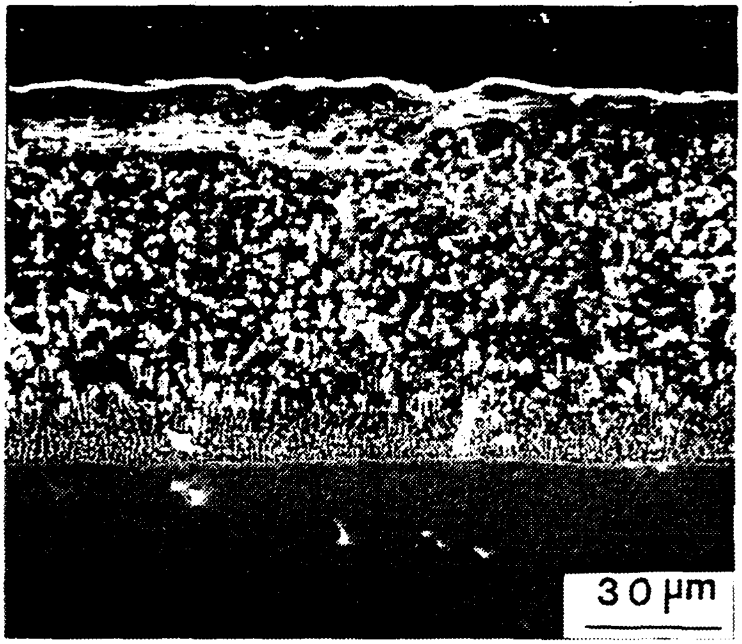

Figure 3.6a HTLA Chromium-Aluminide / IN-738 (as-received).

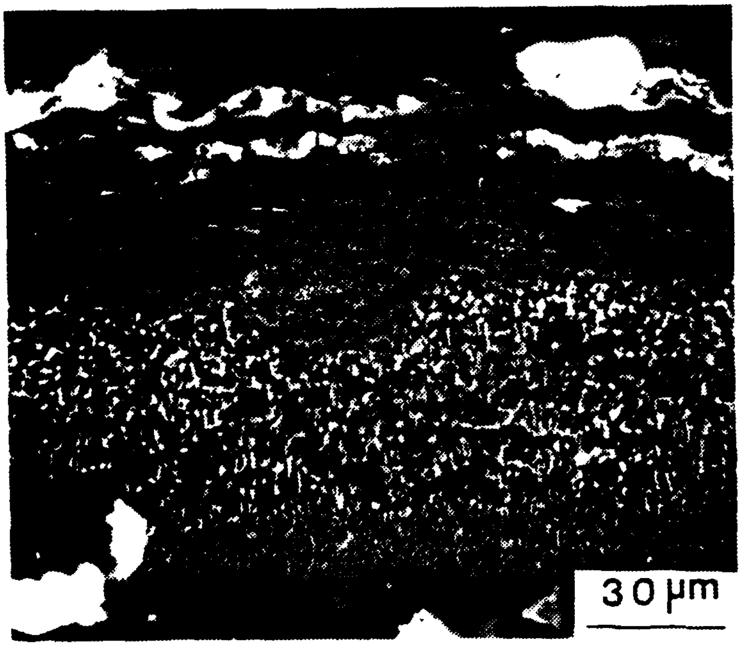

Figure 3.6b HTLA Chromium-Aluminide / IN-738 (LTHC-100 hrs).

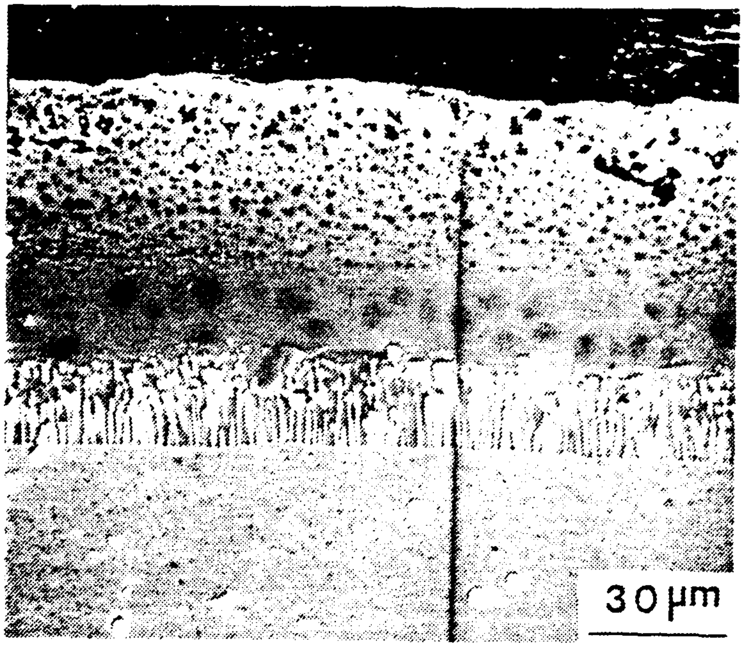

Figure B.7a Process B / IN-738 (as-received).

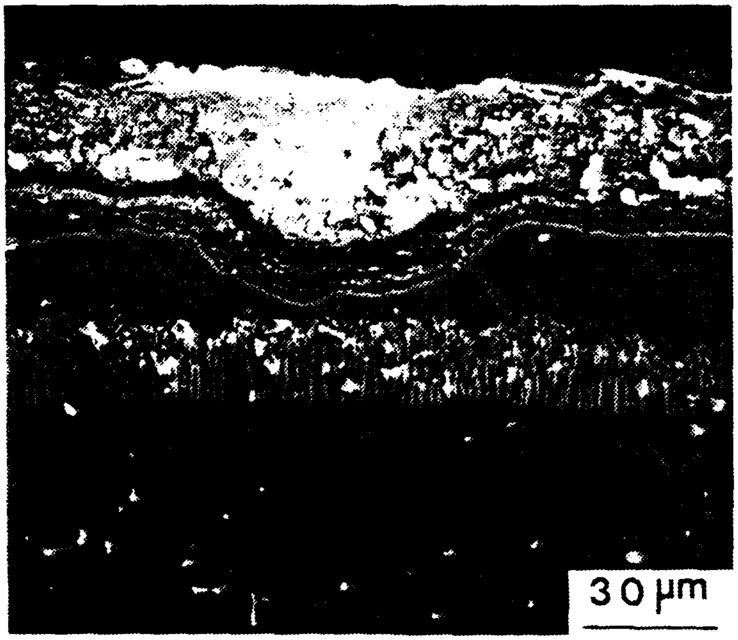

Figure B.7b Process B / IN-738 (LTHC-100 hrs).

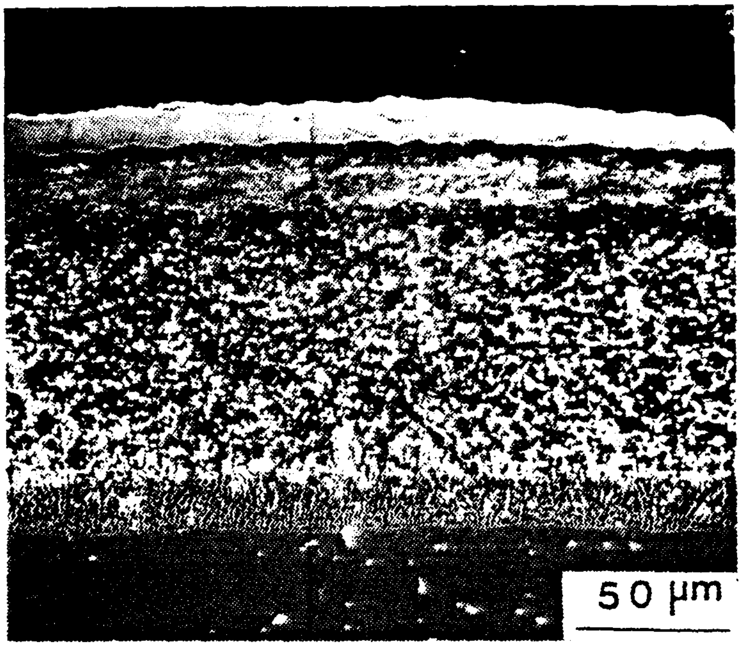

Figure B.8a Process D / IN-738 (as-received).

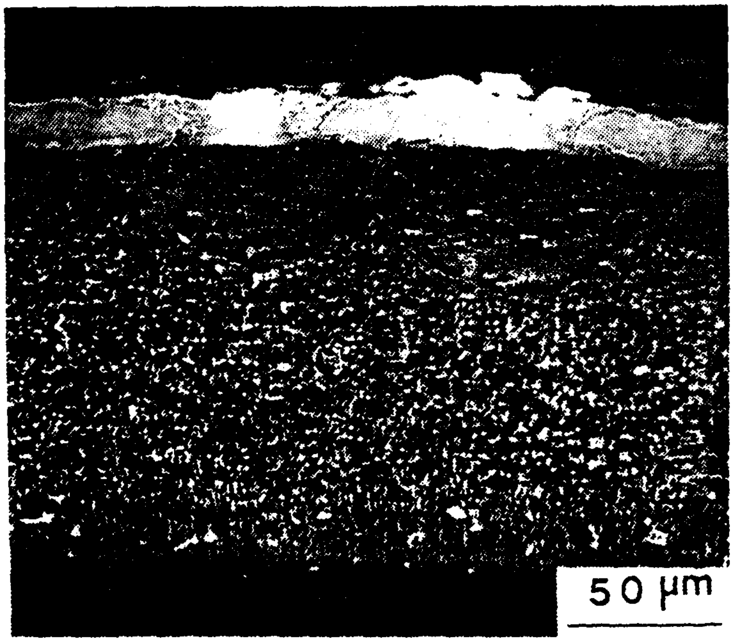

Figure B.8b Process D / IN-738 (LTHC-100 hrs).

Figure B.9a LTHA Platinum-Aluminide / IN-100 (as-received).

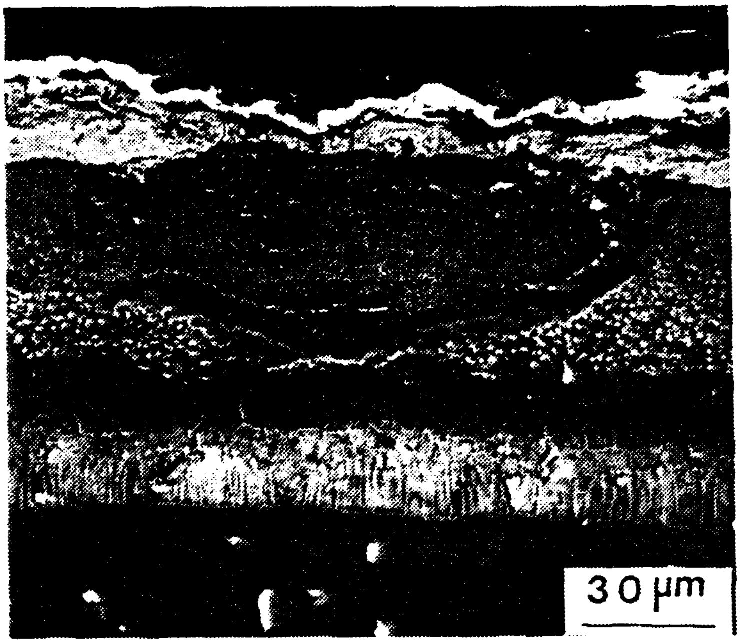

Figure B.9b LTHA Platinum-Aluminide / IN-100 (LTHC-100 hrs).

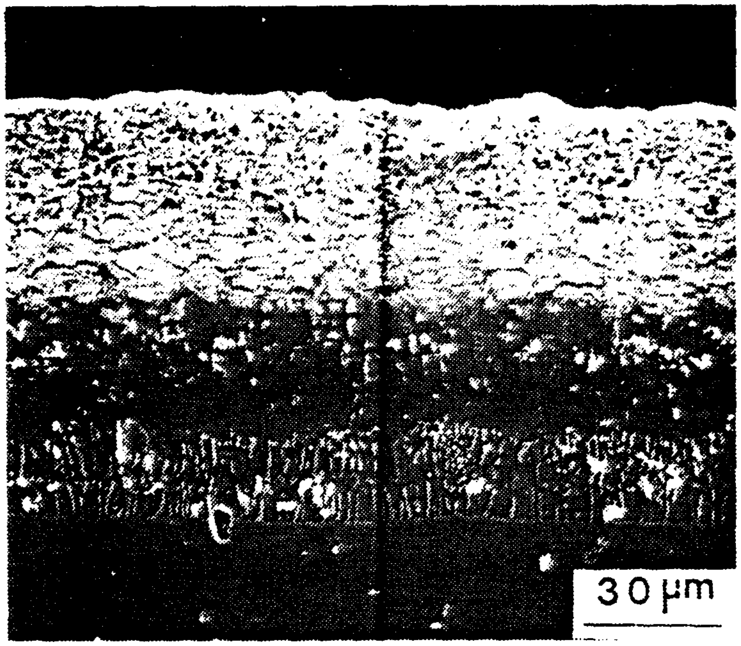

Figure B.10a HTLA Platinum-Aluminide / IN-100 (as-received).

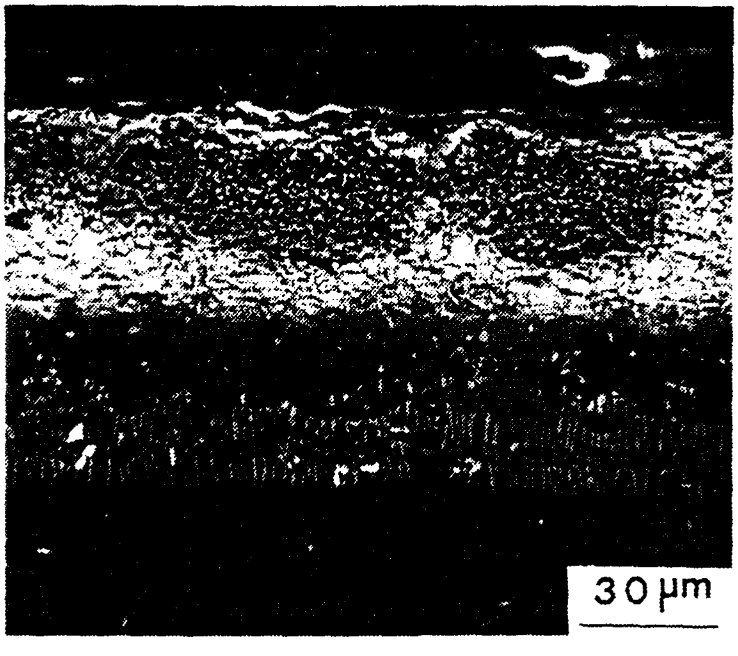

Figure B.10b HTLA Platinum-Aluminide / IN-100 (LTHC-100 hrs).

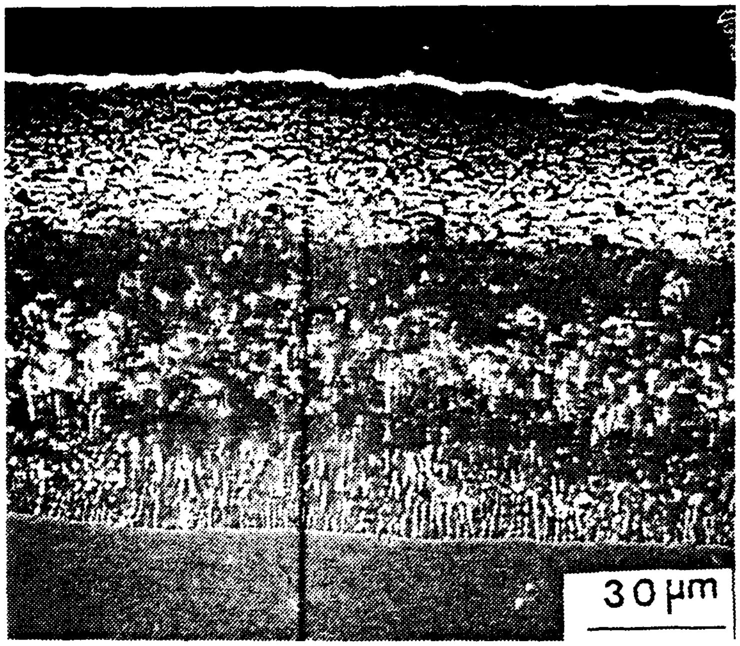

Figure B.11a Process A / IN-100 (as-received).

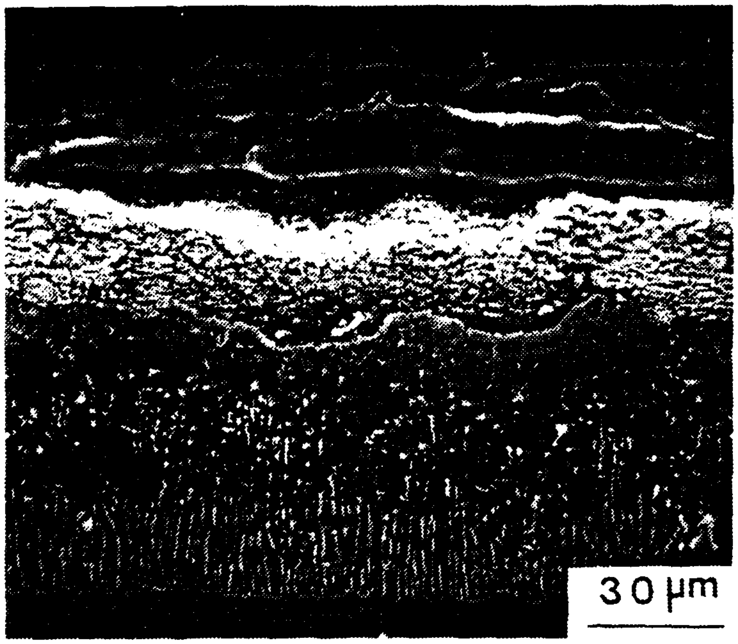

Figure B.11b Process A / IN-100 (LTHC-100 hrs).

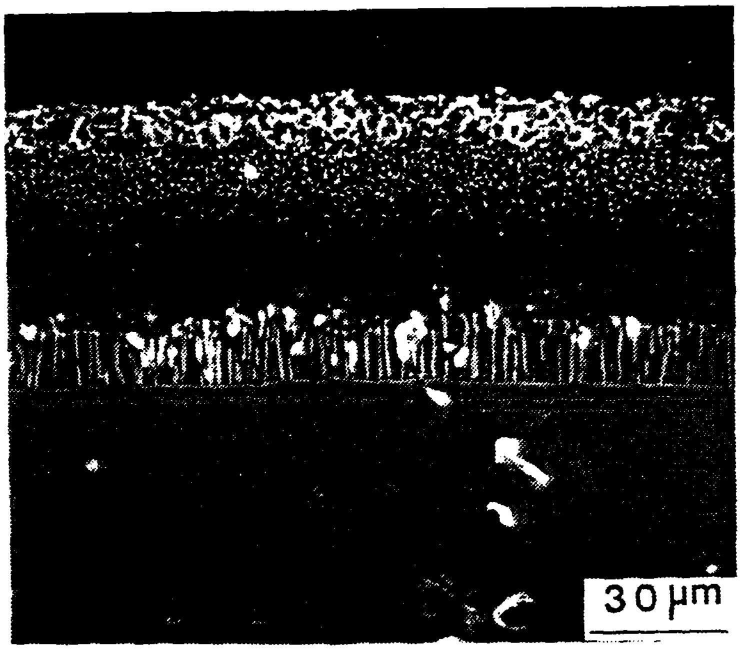

Figure B.12a LTHA Chromium-Aluminide / IN-100 (as-received).

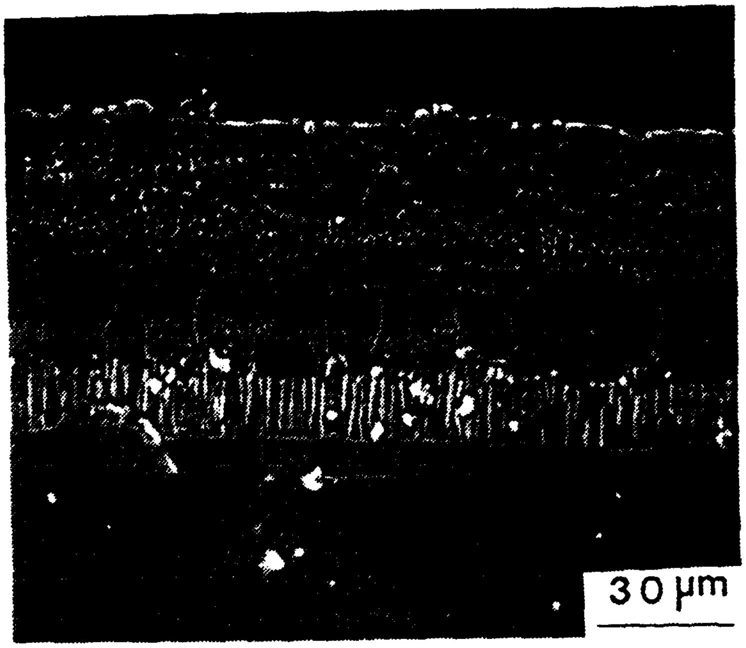

Figure B.12b LTHA Chromium-Aluminide / IN-100 (LTHC-100 hrs).

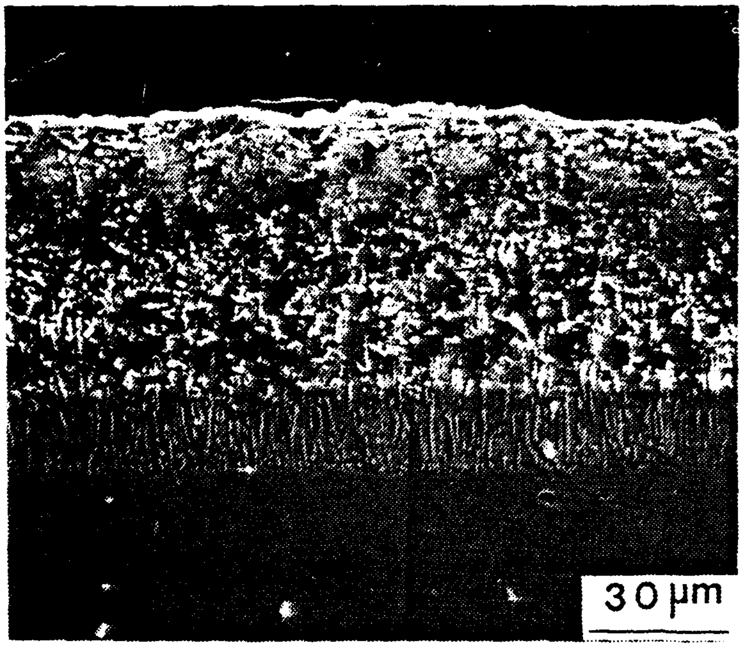

Figure B.13a HTLA Chromium-Aluminide / IN-100 (as-received).

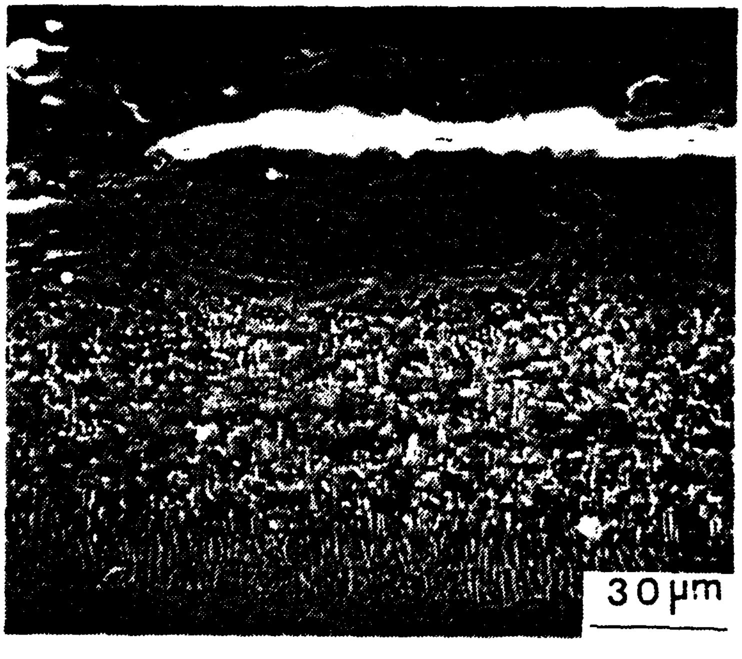

Figure B.13b HTLA Chromium-Aluminide / IN-100 (LTHC-100 hrs).

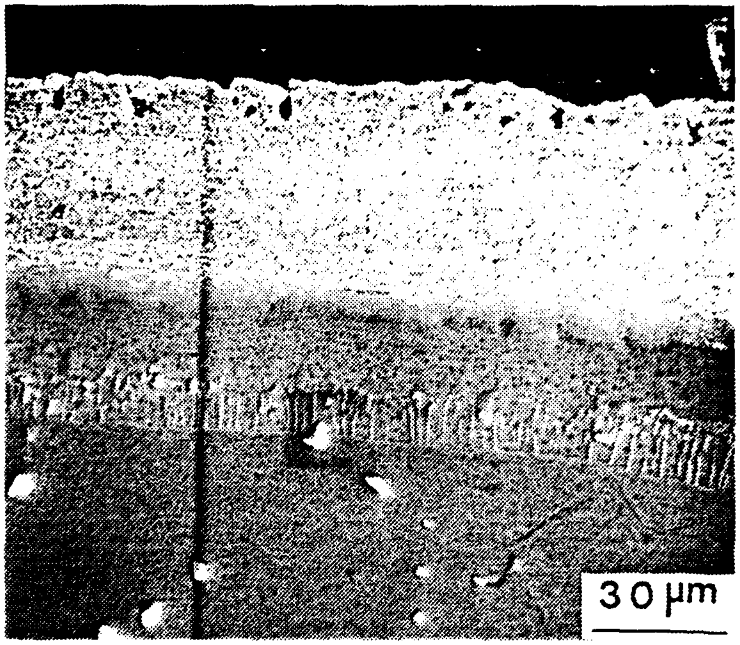

Figure B.14a Process B / IN-100 (as-received).

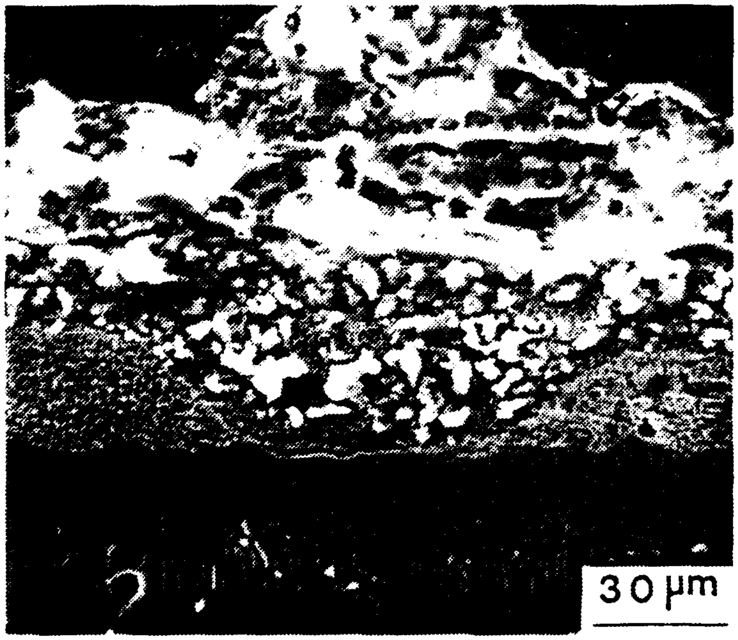

Figure B.14b Process B / IN-100 (LTHC-100 hrs).

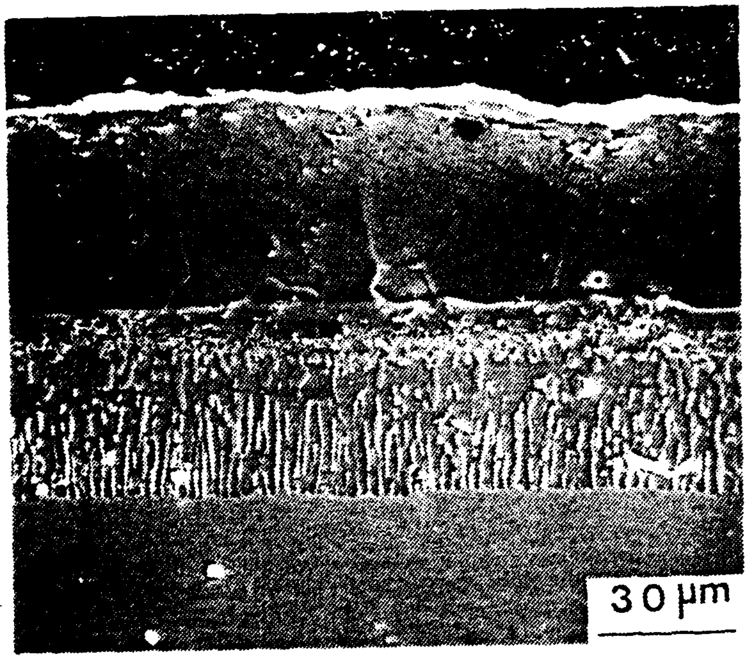

Figure B.15a Process D / IN-100 (as-received).

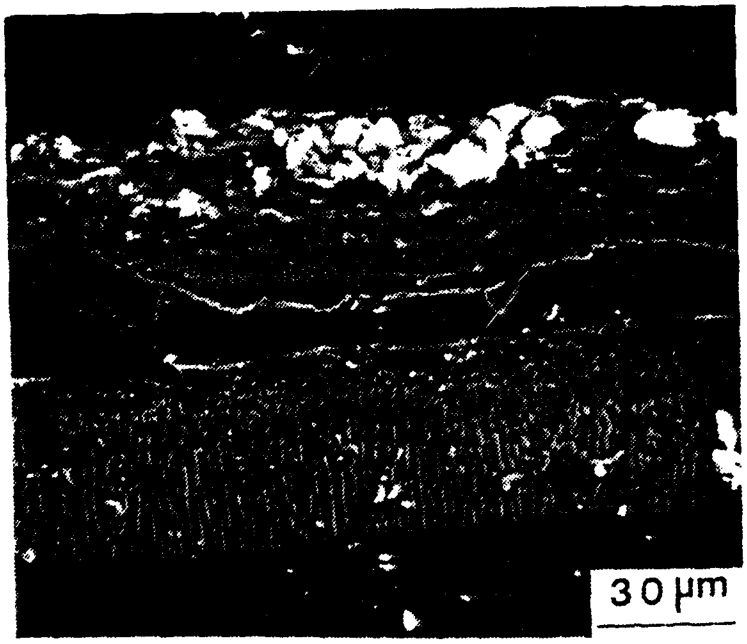

Figure B.15b Process D / IN-100 (LTHC-100 hrs).

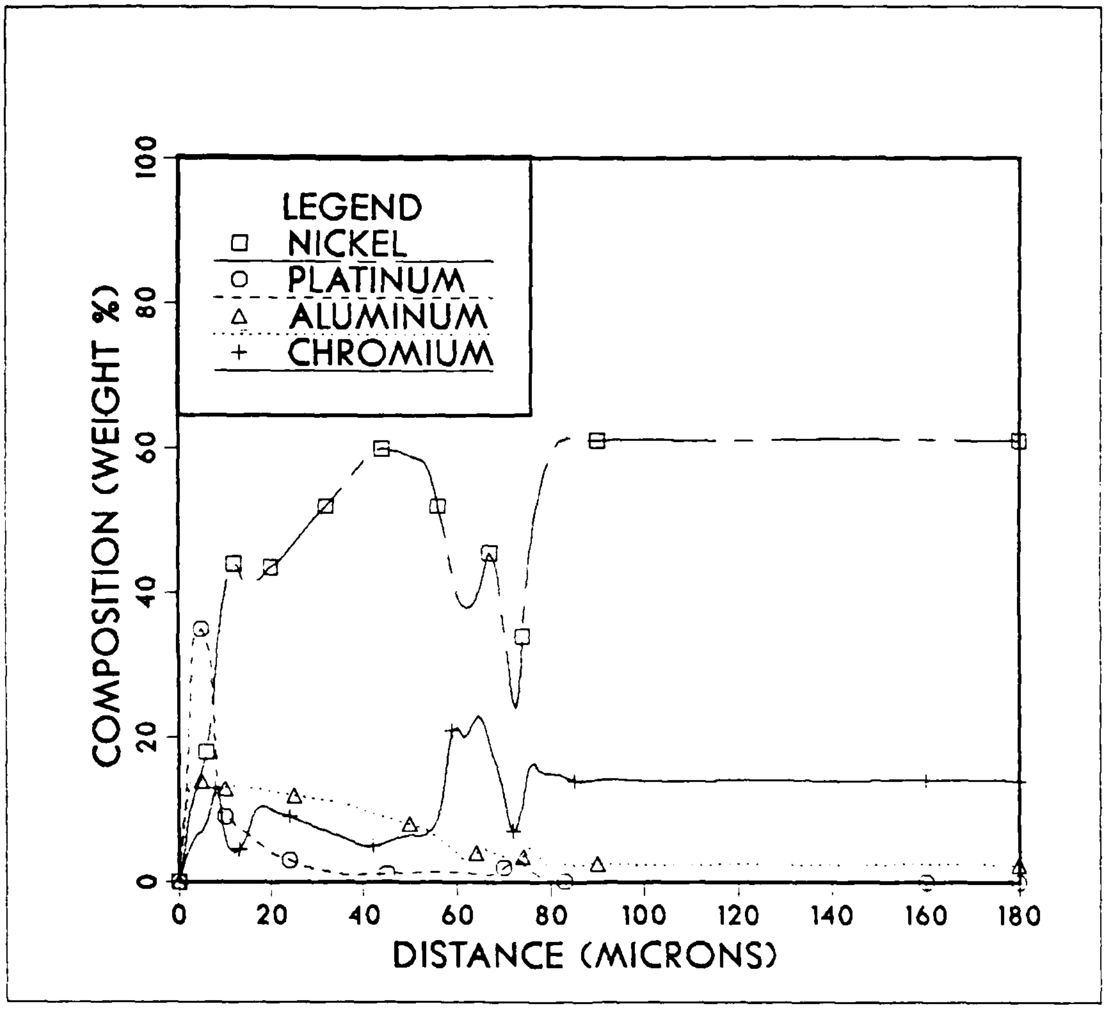

Figure B.16 Composition of LTHA Platinum-Aluminide / IN-738.

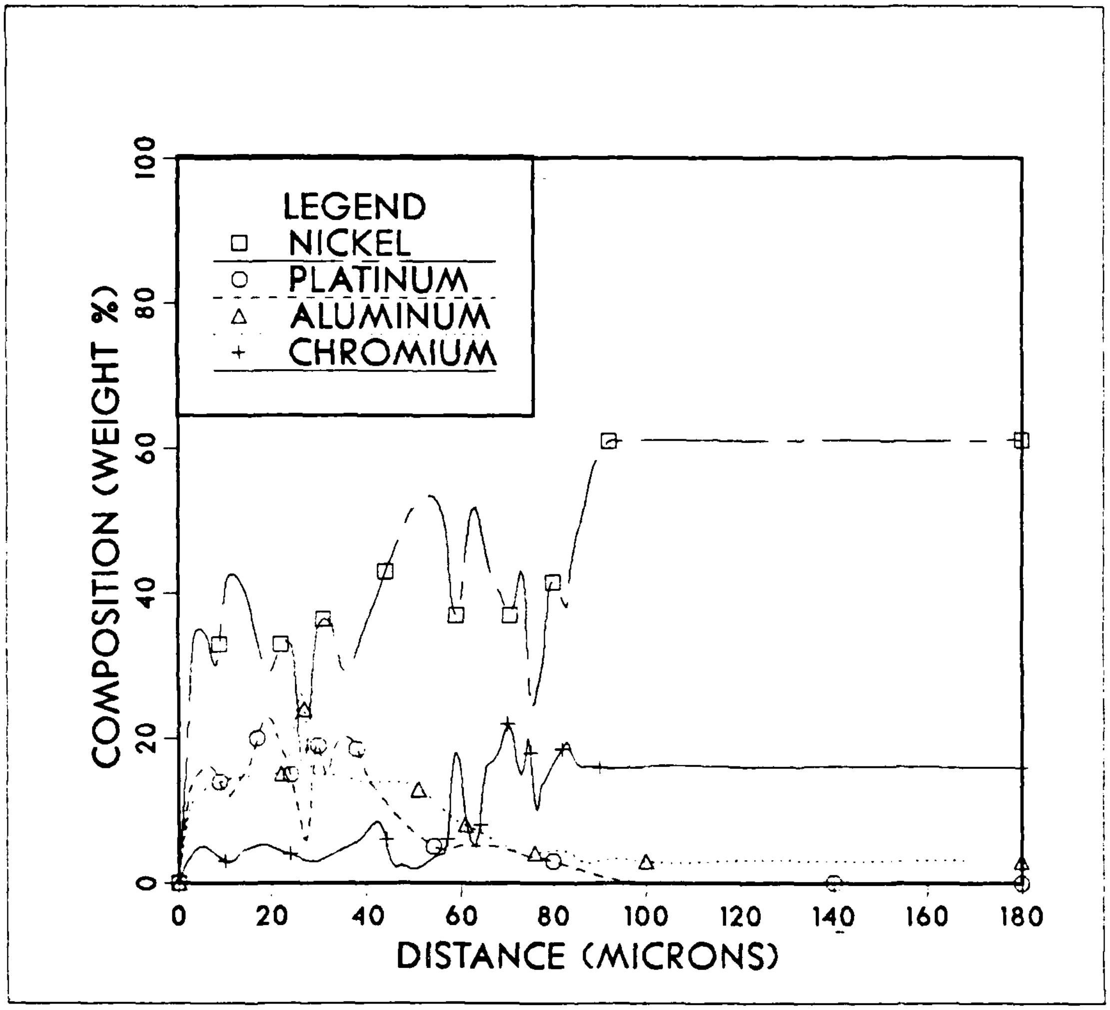

Figure B.17 Composition of HTLA Platinum-Aluminide / IN-738.

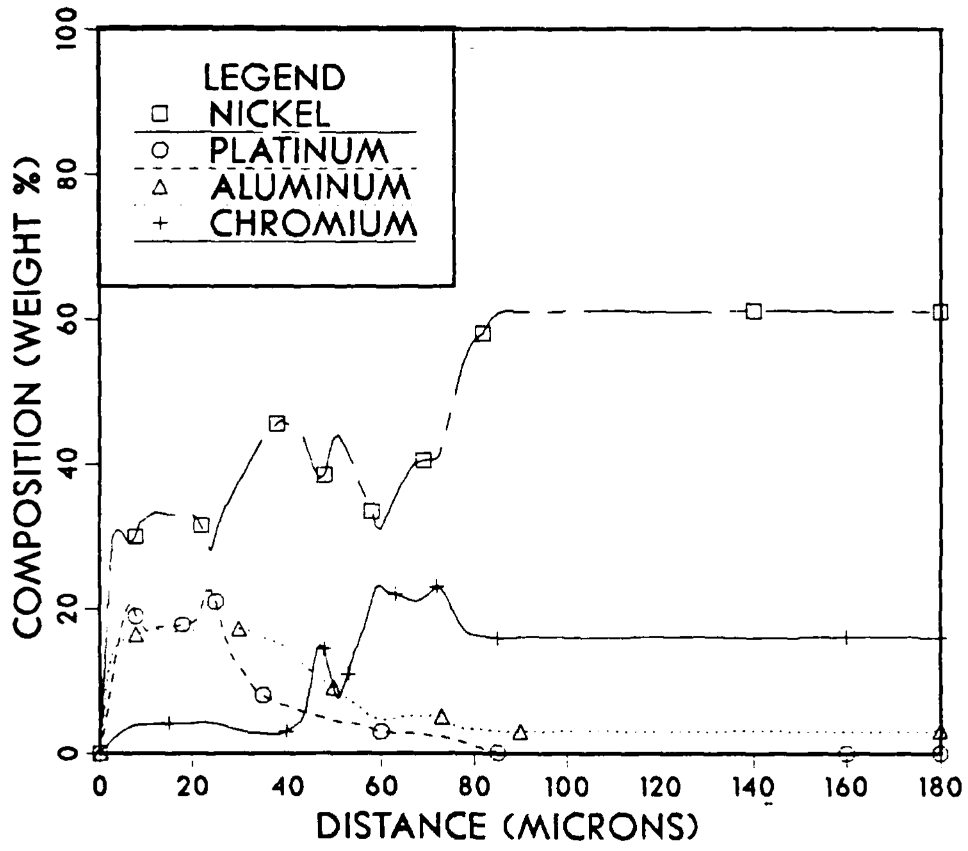

Figure B.18 Composition of Process A / IN-738.

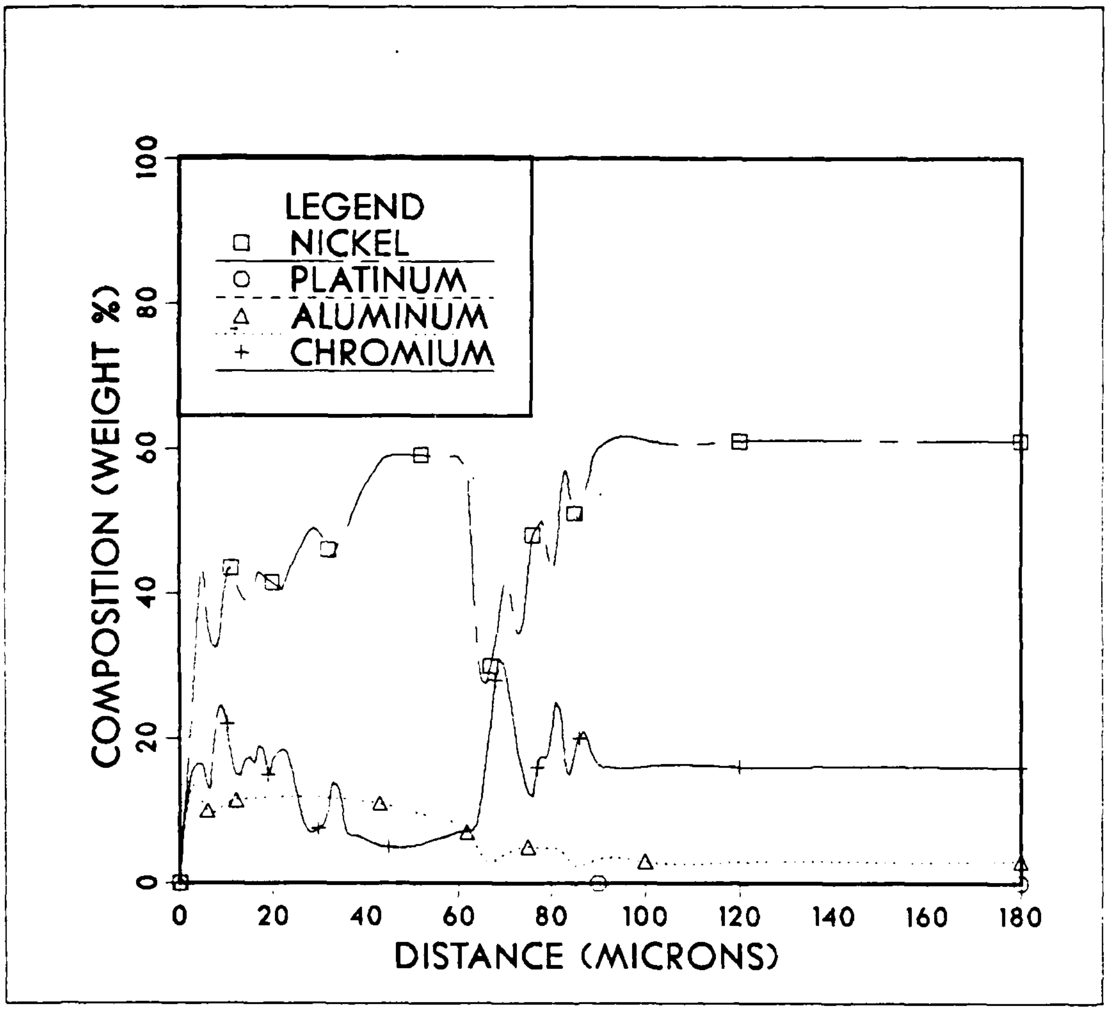

Figure B.19 Composition of LTHA Chromium-Aluminide / IN-738.

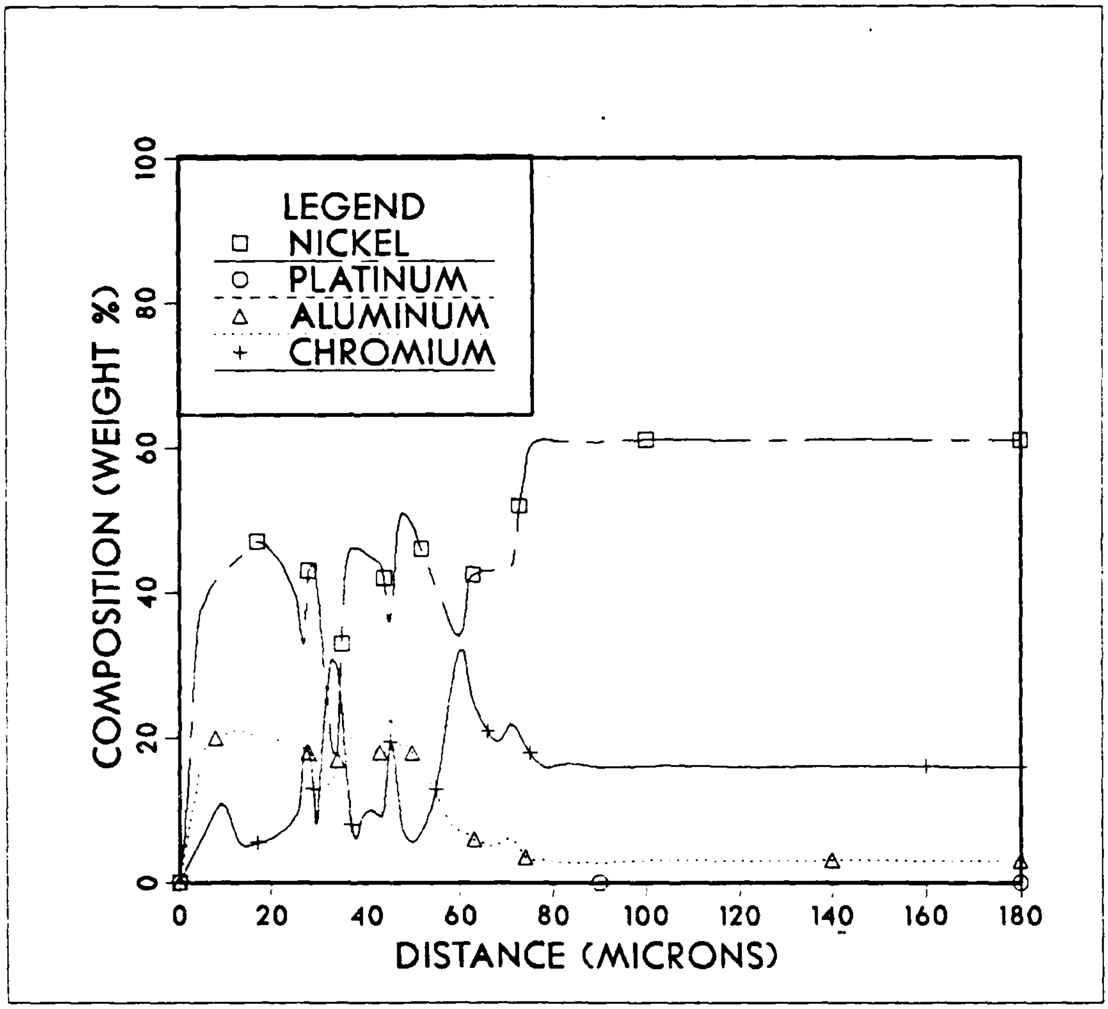

Figure B.20 Composition of HTLA Chromium-Aluminide / IN-738.

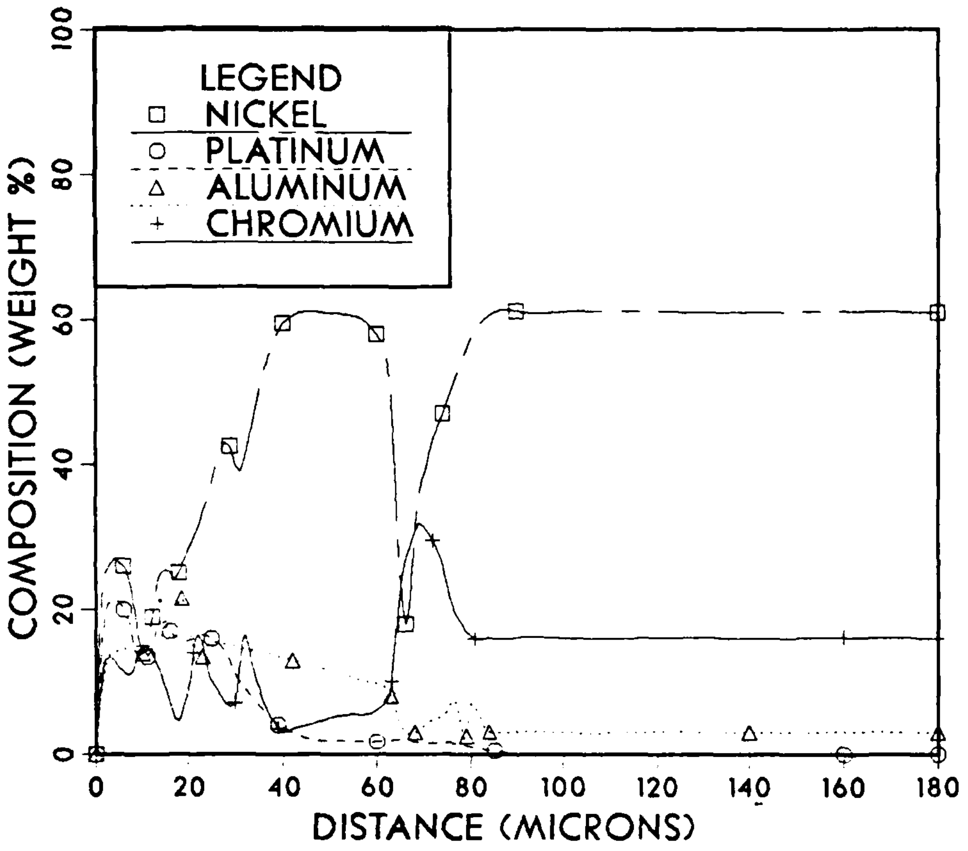

Figure B.21 Composition of Process B / IN-738.

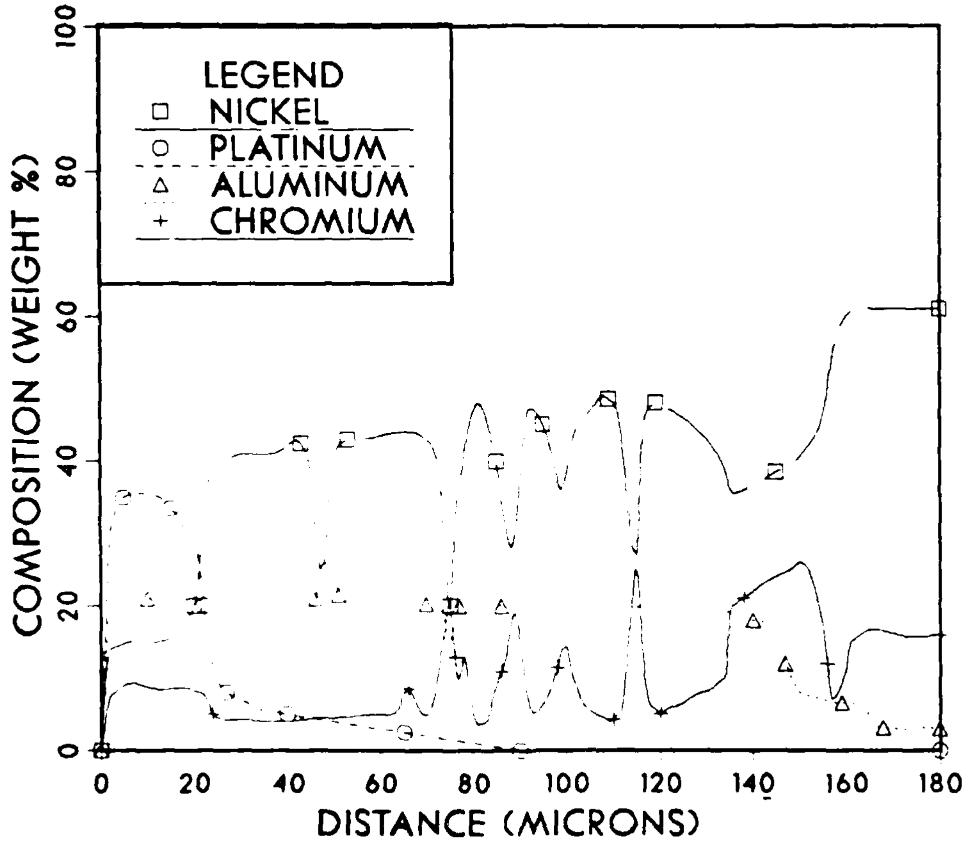

Figure B.22 Composition of Process D / IN-738.

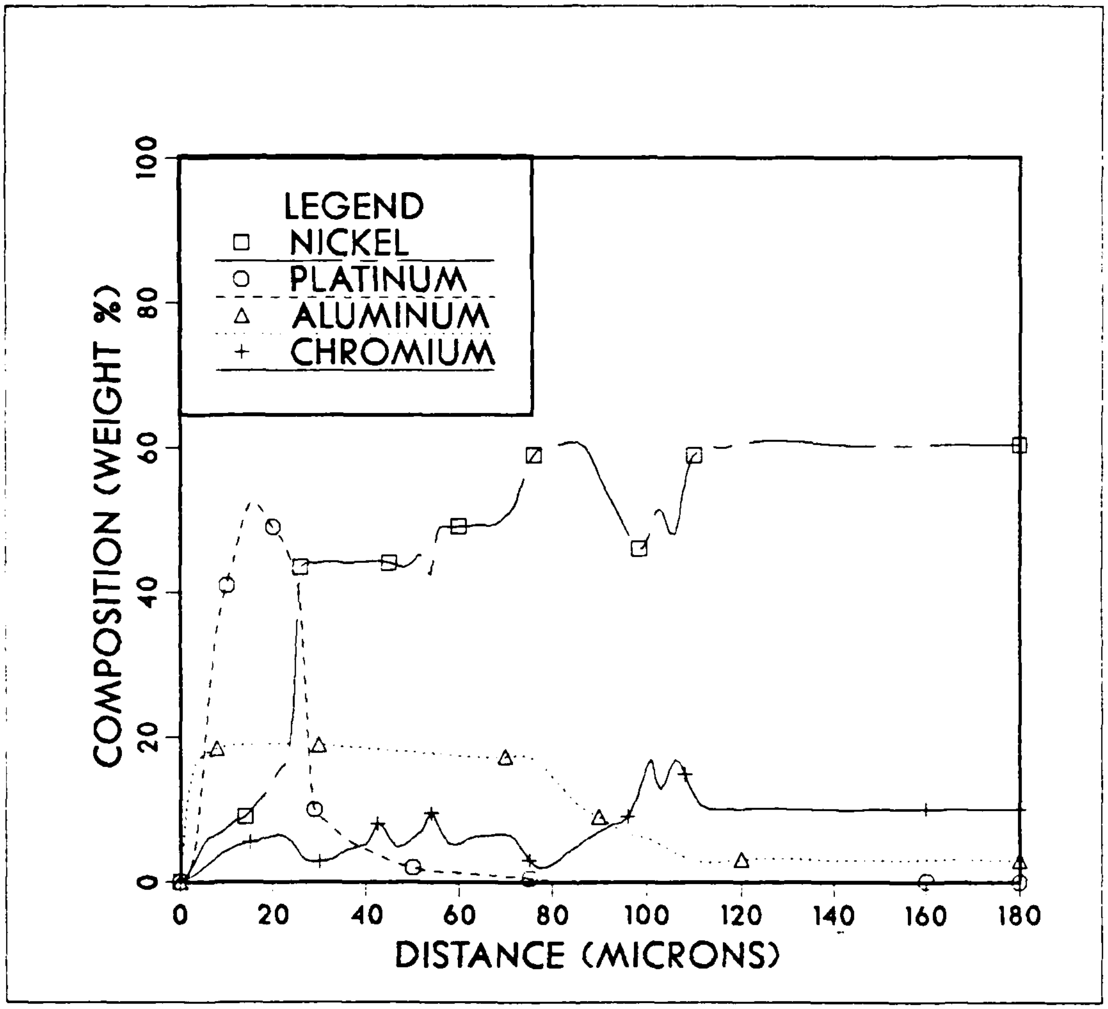

Figure B.23 Composition of LTHA Platinum-Aluminide / IN-100.

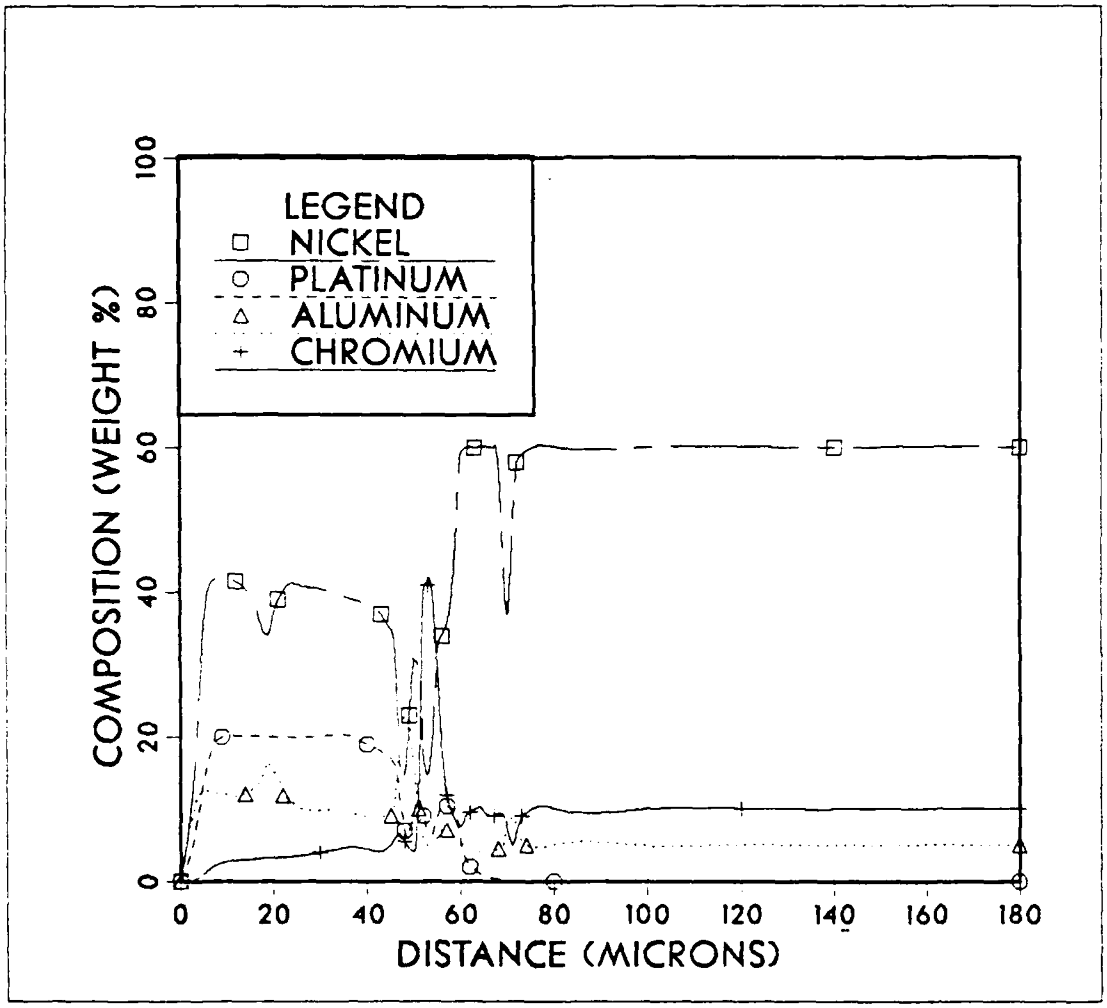

Figure B.24 Composition of HTLA Platinum-Aluminide / IN-100.

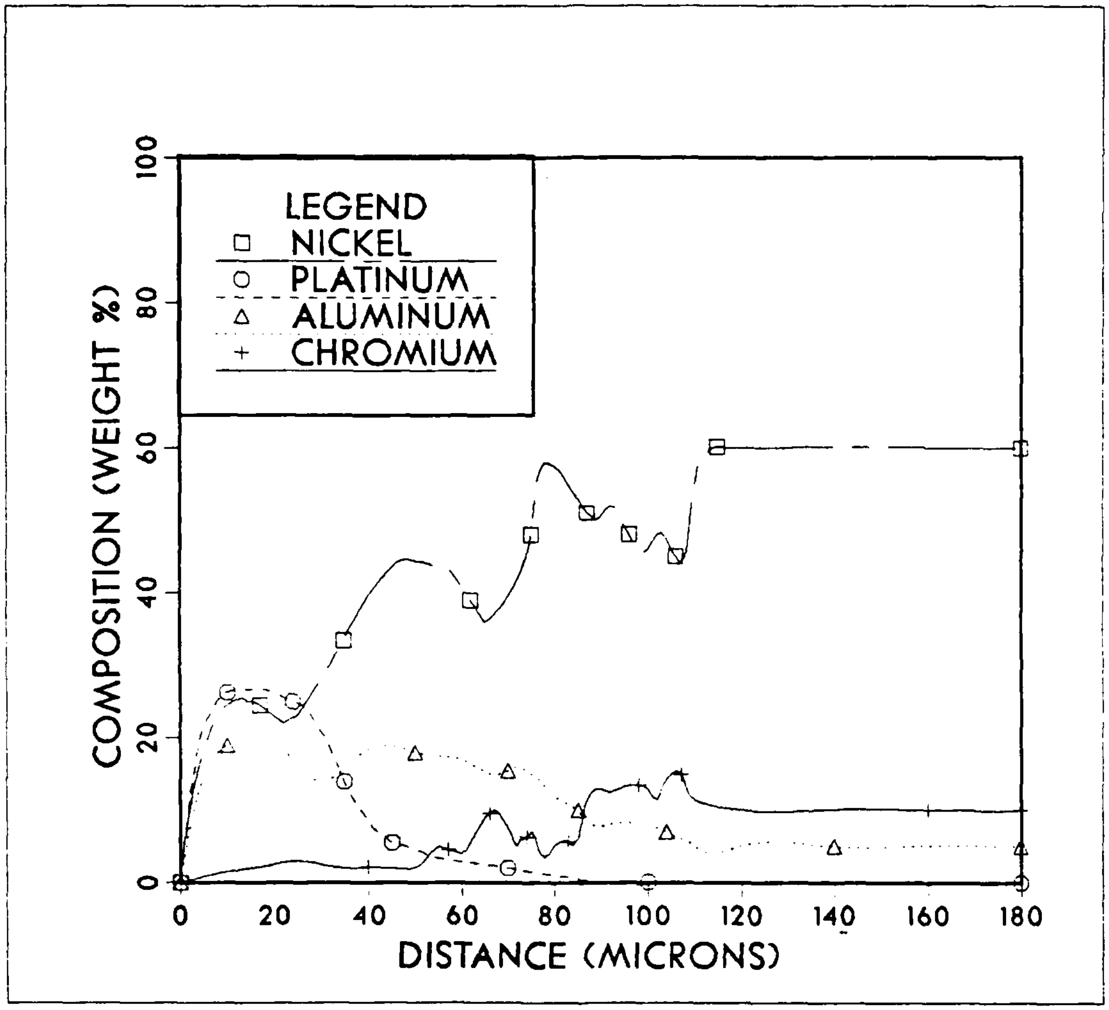

Figure B.25 Composition of Process A / IN-100.

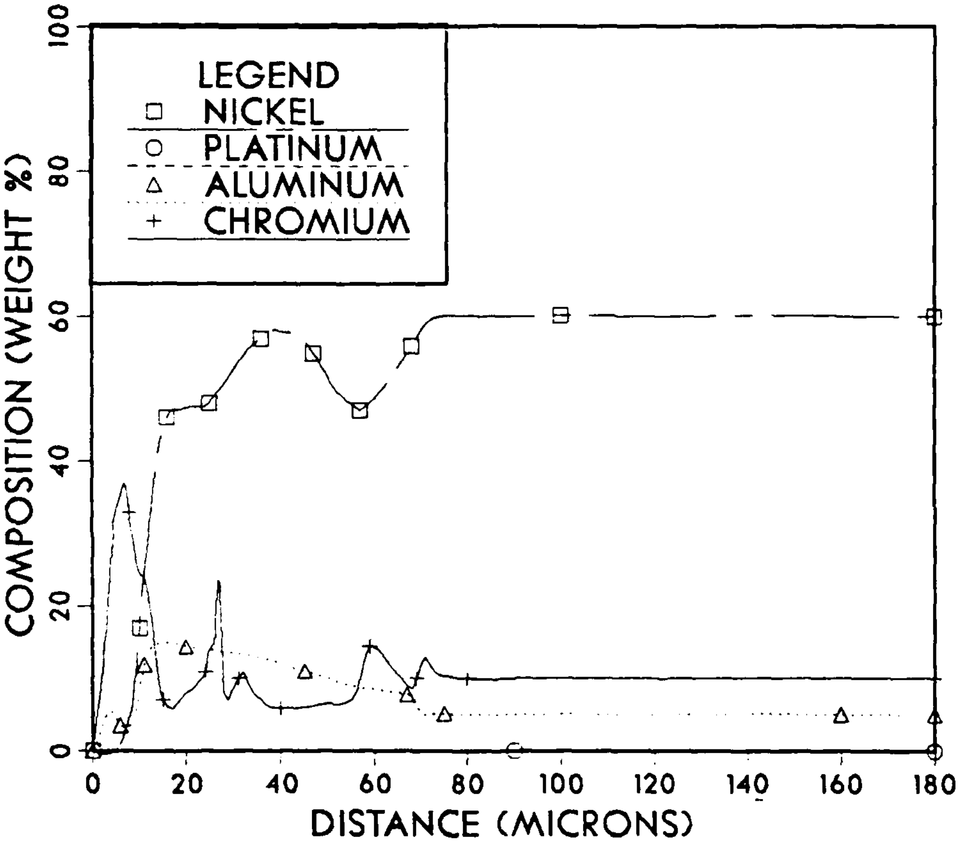

Figure B.26 Composition of LTHA Chromium-Aluminide / IN-100.

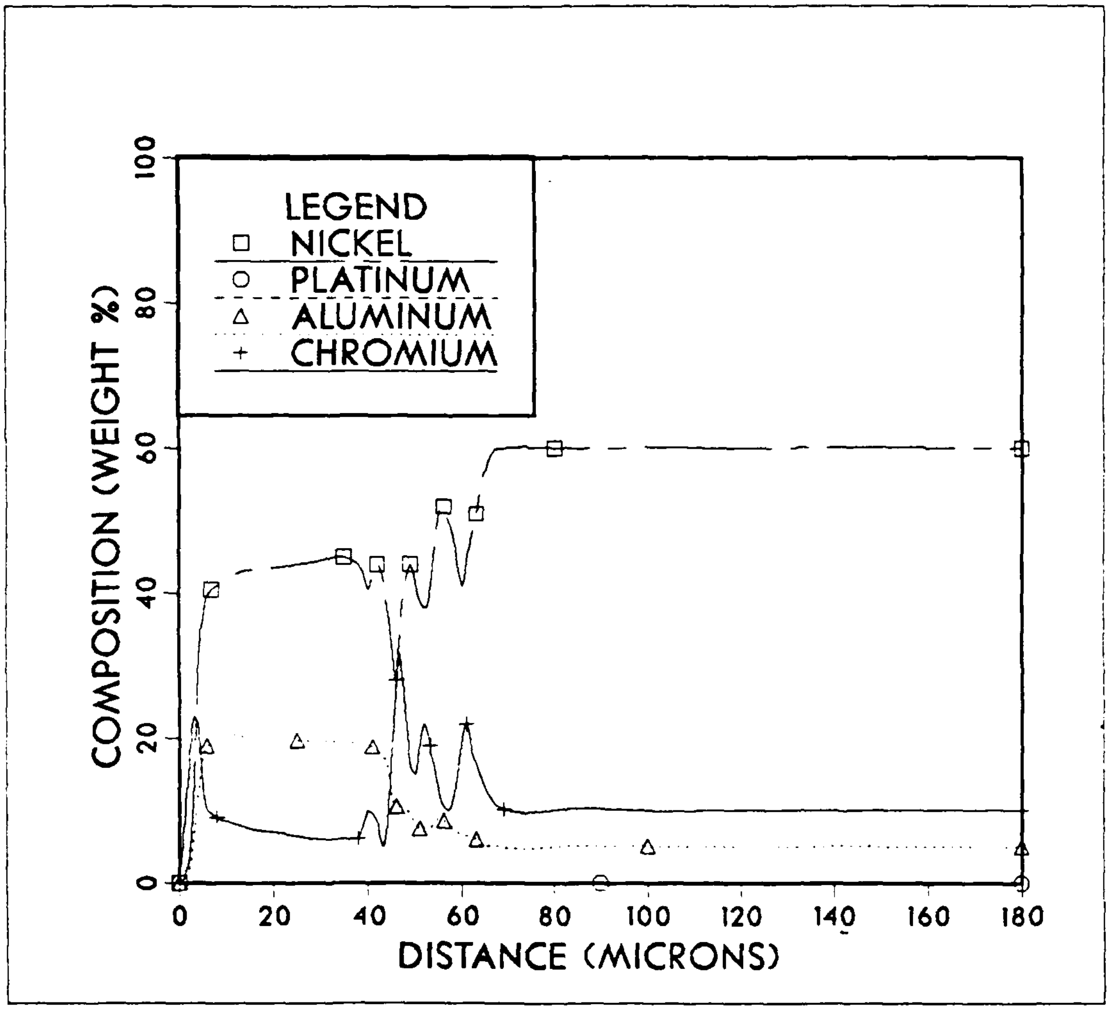

Figure B.27 Composition of HTLA Chromium-Aluminide / IN-100.

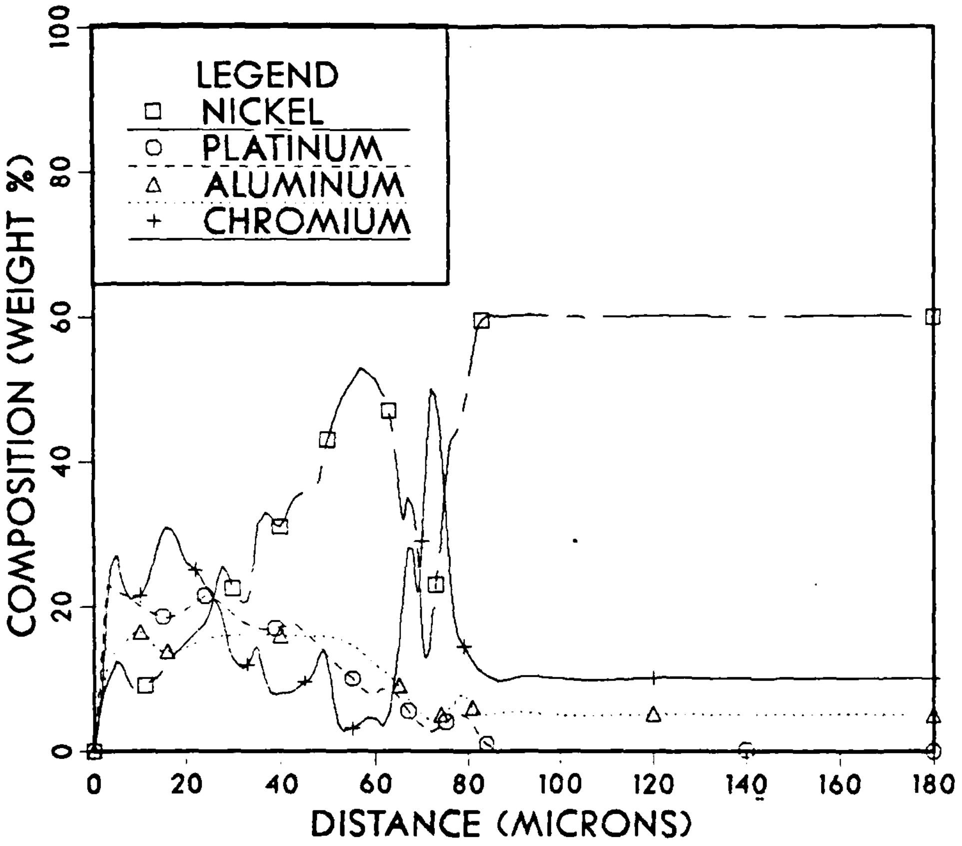

Figure B.28 Composition of Process B / IN-100.

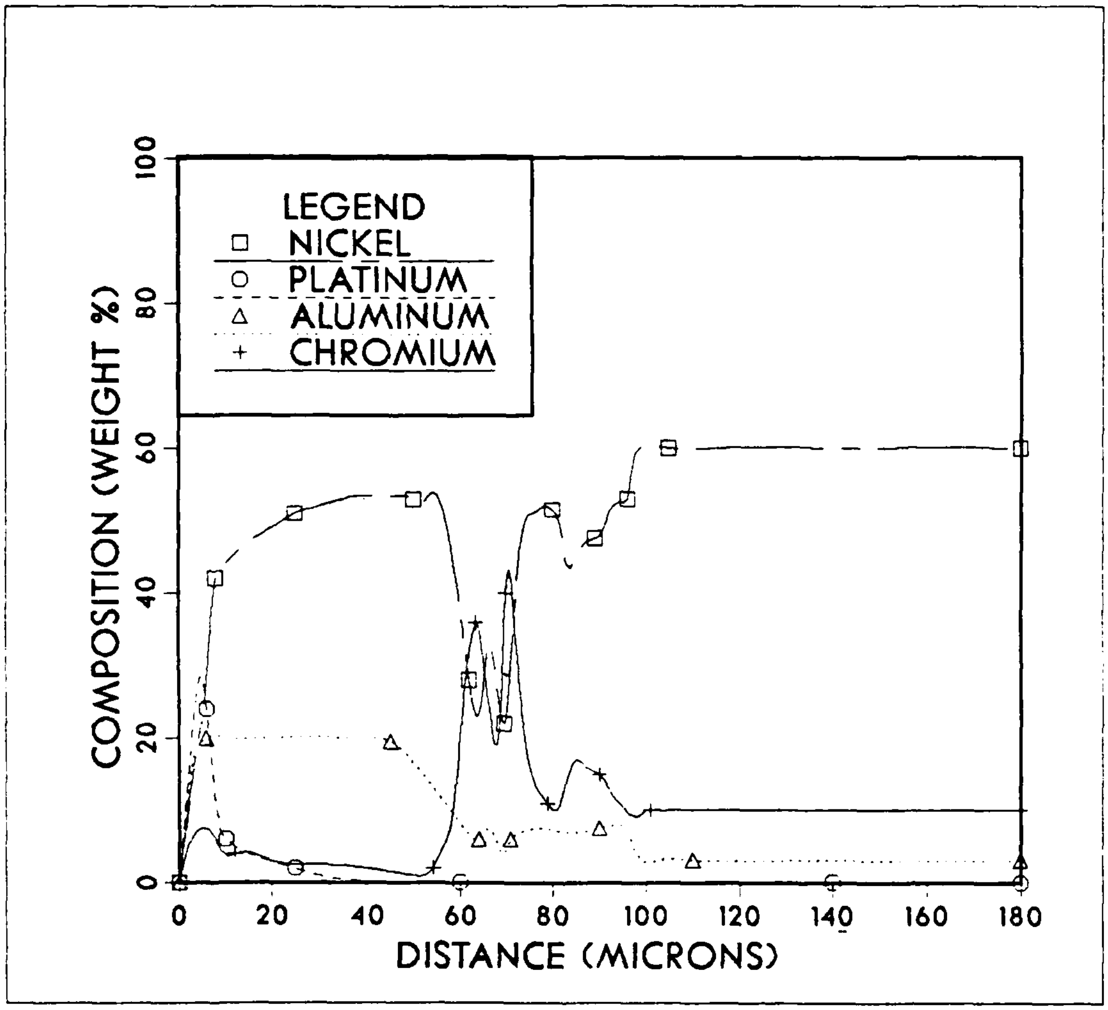

Figure B.29 Composition of Process D / IN-100.

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