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SURFACE TREATMENTS FOR NICKEL AND NICKEL-BASE ALLOYS

By C. M. Jackson and A. M. Hall

Prepared Under the Supervision of the
Research Branch, Redstone Scientific Information Center
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ABSTRACT

This report presents the state of the art in regard to selected surface treatments for nickel and nickel-base alloys. The treatments covered are diffusion coating, electroplating, electroless plating, hard facing, surface treatment for purposes of lubrication, explosive hardening, peening, burnishing and planishing. Each treatment is described and its application to nickel and nickel-base alloys is discussed. Reasons for using the treatment are covered, as are the effects of the operations involved on appropriate properties of nickel and nickel-base alloys. Problem areas are defined and recommendations for research are made.

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*Principal Investigators, Battelle Memorial Institute, Contract No. DA-01-021-AMC-11651(Z)

NASA-GEORGE C. MARSHALL SPACE FLIGHT CENTER

PREFACE

This report is one of a series of state-of-the-art reports being prepared by Battelle Memorial Institute, Columbus, Ohio, under Contract No. DA-01-021-AMC-11651(Z), in the general field of materials fabrication.

The published information used in preparing this report was obtained from technical articles, reports on Government-sponsored research programs and manufacturers' brochures. In locating this material, the following information centers, libraries, and abstract services were searched for the period from 1960 to the present:

Redstone Scientific Information Center
Defense Documentation Center
Defense Metals Information Center
The Libraries of Battelle Memorial Institute
STAR Abstracts
ASM Review of Metal Literature.

In addition, the NASA Research and Technology Program Digest Flash Index FY64 and the NASA Program Summary FY65 were searched for information on current programs and research reports.

To ensure that the property data presented in this report are up to date, and to obtain information on current shop practices, personal contacts were made, by telephone or visit, with 40 individuals from 35 organizations. The individuals and companies are listed in the Appendix.

The authors wish to thank each of these individuals and their organizations for their contributions. They also wish to thank Vernon W. Ellzey and Albert G. Imgram, Battelle Project Technical Coordinators, and Walter H. Veazie, Jr., and Mrs. Barbara A. Gilmour, Battelle Information Specialists, for their efforts during the progress of this program.

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C. M. Jackson and A. M. Hall

Prepared for

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In Cooperation with

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Under the Supervision of

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SURFACE TREATMENTS FOR NICKEL AND NICKEL-BASE ALLOYS

SUMMARY

Treatments have been developed that can improve a number of characteristics of the surfaces of articles made from nickel and nickel-base alloys. Among the characteristics that can be improved are lubricity, resistance to corrosive attack by oxidizing and/or sulfur-containing atmospheres, and resistance to wear, erosion, and fatigue. Surface treatments may be grouped into two categories, coating treatments and mechanical treatments.

COATING TREATMENTS

Coating treatments include diffusion coating, electroplating, electroless plating, hard-facing and surface treatment for purposes of lubrication.

Most diffusion coatings used in the United States for nickel-base alloys are rich in aluminum. They are used primarily to protect parts of aircraft, marine and automotive gas-turbine engines from the degrading effects of the service environment. There is still much room for improvement in these coatings, particularly those for engines that will be used near the sea. Under such circumstances, the salt content of the air, combined with sulfur from the jet fuel causes a new, severe type of sulfidation attack.

Diffusion coatings based on boron have been developed in the Soviet Union as a means of obtaining very hard cases on nickel and nickel-base alloys.

Nickel and nickel alloys generally are not electroplated or electroless plated, both because they are not used in applications in which plating is required and because they often inherently possess the corrosion resistance or other attribute for which plates are applied. In the relatively few applications where they are electroplated, care must be taken to first remove the passive surface film that occurs naturally on these materials.

Hard facings are not often applied to nickel or nickel-base alloys. However, examples are known in which hard facings have imparted to these materials the required resistance to steam erosion, erosion-corrosion, or wear.

Surface treatments have been developed that provide nickel and nickel-base alloys with lubricity under conditions in which oils and greases would deteriorate. Examples of such conditions are high temperature and high vacuum.

The following summary tabulation will serve as a general guide in selecting coating surface treatments on the basis of the characteristic desired to be imparted to nickel and nickel-base alloys. As noted, some of the treatments are known to have been applied to this general type of material*, while other treatments apparently have not; however, there is a strong possibility for the latter being applied successfully.

Desired Characteristic	Corresponding Surface Treatment	Known to be Satisfactory[a]	Expected to be Satisfactory[b]
Resistance to oxidation at high temperatures	Diffusion coating[c]	Nickel-base superalloys	
Resistance to sulfidation at high temperatures	Diffusion coating[c]	Nickel-base superalloys	
Resistance to erosion by flames and hot gases	Diffusion coating[c]	Nickel-base superalloys	
Resistance to impact by foreign objects	Diffusion coating[c]	Nickel-base superalloys	
Resistance to mechanical and thermal fatigue, including thermal shock	Diffusion coating[c]	Nickel-base superalloys	
Hard case	Boron diffusion coating	Nickel and nickel-base alloys	
Hard case	Carbon diffusion coating	Chromized Hastelloy B	
Resistance to attack by chemicals	Electroplating of gold[d]		✓
Resistance to wear	Electroless plating of nickel-phosphorus alloy		✓
Resistance to erosion by steam	Hard facing with a -base alloy	Inconel 600	
Resistance to erosion-corrosion	Hard facing with a nickel-base alloy	Duranickel	
Resistance to wear	Hard facing with tungsten carbide plus 9 per cent cobalt	Monel K-500	
Resistance to wear	Hard facing with tungsten carbide in a Nichrome matrix	Waspaloy and Udimet 700	
Lubricity at elevated temperatures	Application of film of lead monoxide	Inconel X-750	
Lubricity at elevated temperatures	Application of ceramic-bonded calcium fluoride coating	Inconel X-750 and René 41	
Lubricity under conditions of ultrahigh vacuum	Vapor deposition of gold	Nickel, Ni-Cr alloys and Ni-Re alloys	
Resistance to wear	Hard facing with nickel-base and cobalt-base hard facing alloys and with tungsten carbide	Nickel and Monel 400	
Lubricity at elevated temperatures	Application of molybdenum disulfide or tungsten disulfide	✓	

(a) Known to have been used to impart the noted characteristic to nickel and/or nickel-base alloys. When specific alloys are mentioned, this should not be interpreted as limiting the possible applicability of the treatment to these alloys alone.

(b) Would be expected to impart the noted characteristic to nickel and/or nickel-base alloys.

(c) Aluminum, aluminum-rich, beryllium-base, zirconium, or zirconium-chromium diffusion coatings.

(d) Other electroplated coatings might also be suitable for these and other specific purposes.

---

*When specific alloys are known to have been treated, they are listed. Such a listing should not be interpreted as limiting the possible applicability of the treatment to other alloys, however.

MECHANICAL TREATMENTS

Mechanical surface treatments such as burnishing, explosive hardening, peening and planishing are not used to any great extent for nickel and nickel-base alloys. When used, they serve a variety of functions from improving surface finish to increasing fatigue strength and surface hardness, to reducing the occurrence of weld cracking. Improvements in mechanical properties arise largely as a result of the residual compressive stress established in the surface of the metal by the treatments.

The following tabulation will aid in the selection of mechanical surface treatments to impart particular desired characteristics to nickel and nickel-base alloys. Included are treatments that are known to have been applied to this general type of material*, and treatments that have not been applied but show promise of successful use.

		State of the Art	
Desired Characteristic	Corresponding Surface Treatment	Known to be Satisfactory(a)	Expected to be Satisfactory(b)
Mirror-like, smooth surface	Burnishing	✓	
Smooth surface	Planishing		✓
Cold working of welds(c)	Planishing	Inconel 600 and Inconel 718	
Cold working of welds(c) and wrought components	Peening		✓
Prevention of cracking in weldments(d)	Peening	René 41 and Astroloy	
Improved fatigue strength	Peening		✓
Correction of distortion in weldments and in unwelded components	Peening		✓
Increased strength of weldments	Explosive hardening		✓
Increased tensile and yield strengths	Explosive hardening	✓	
Improvement of resistance to stress-corrosion cracking	Peening		✓

(a) Known to have been used to impart noted characteristic to nickel and/or nickel-base alloys. When specific alloy names are given, these are the ones on which it is known that the treatment has been successful; it is expected that it would also be successful on other nickel-base alloys.

(b) Would be expected to impart noted characteristic to nickel and/or nickel-base alloys.

(c) To induce recrystallization upon subsequent heating, and thereby cause the mechanical properties of the weld to more closely match those of the wrought parent metal.

(d) By counteracting the tensile stresses developed in the joint by the shrinkage that occurs on cooling after welding.

Note: Applications of mechanical treatments for the purpose of forming are not included in this tabulation.

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*When specific alloys are known to have been treated, they are listed. Such a listing should not be interpreted as limiting the possible applicability of the treatment to other alloys, however.

INTRODUCTION

Nickel and nickel-base alloys are used extensively in essentially all areas of industry. Applications range from such items as special plumbing fixtures to structurals in aerospace vehicles. The many and varied applications of these metals are based primarily on their resistance to corrosion by specific environments, to their formability and to their strength and resistance to creep at elevated temperatures. Other important uses of these metals capitalize on the wide variety of electrical and magnetic properties obtainable in nickel-base alloys, and on the ability of the alloys to retain ductility and toughness at cryogenic temperatures (Ref. 1).

Advancing technologies are, nevertheless, continually challenging the ability of nickel-base alloys to provide new combinations of mechanical, physical, and chemical characteristics. Often, different characteristics are required of the surface of an article than of the interior; in many service environments the surface characteristics are the more important of the two. Accordingly, to prevent the characteristics of the surface from limiting the usefulness of an otherwise acceptable alloy, various surface treatments have been developed to improve these characteristics.

Frequently, the purpose of the surface treatment is to provide resistance to corrosive attack, such as oxidation or sulfidation, to lubricate rubbing surfaces, or to improve resistance to wear or erosion; these goals may often be accomplished by altering the nature of the surface by the application of a coating. In other cases, the purpose of the surface treatment is to increase the fatigue resistance of material subjected to alternating stresses; this may often be accomplished by establishing a compressive state of stress in the surface by a mechanical surface treatment such as peening. Or, the purpose may be to induce recrystallization in welds upon subsequent heat treatment, in which case the cold work induced by peening or planishing may be effective. For improvement of surface finish, burnishing and planishing may be helpful.

The purpose of this report is to present the state of the art in regard to surface treatments for nickel and nickel-base alloys. Covered are diffusion coating, electroplating, electroless plating, hard facing, coating for lubrication, burnishing, explosive hardening, peening and planishing. Other surface treatments such as grinding, machining, and vacuum metallizing are not included, inasmuch as they are covered in detail in other reports being prepared by Battelle Memorial Institute for the Redstone Scientific Information Center.

For each surface treatment considered, details of the procedures used to accomplish the treatment on nickel and nickel-base alloys* are given whenever available. Likewise, data showing the effect of the surface treatments on the properties of nickel and nickel-base alloys are presented when available and appropriate. The availability of procedural details and test data was limited because some of the treatments are not used very extensively for nickel and nickel-base alloys and because of the proprietary nature of some of the processes discussed.

COATING TREATMENTS

Coatings are applied to the surfaces of metals to impart some specific characteristic to them. Characteristics of frequent interest are lubricity and resistance to corrosive attack, fatigue, and erosion. The use of a coating is generally cheaper and more technically feasible than attempting to fabricate the entire part from the coating material; in fact, in many cases such a fabricated part would lack some other vital characteristic such as strength or toughness.

The coating treatments discussed in this report are diffusion coating (by such methods as pack cementation, vapor plating, slurry processes, and hot dipping), electroplating, electroless plating, hard facing, and processes used to apply coatings for the purpose of lubrication.

An article that is to be coated can generally be of the same design as an article that would be used uncoated in the same application. However, three additional points should be considered when designing a part that is to be coated:

(1) Sharp radii should be avoided; such radii promote weak areas in the coating and serve as stress raisers during rapid heating and cooling (Ref. 14). Corners and edges

---

*The nominal chemical compositions of the nickel-base alloys for which specific treatments are given in this report are shown in Table I on page 7.

should not just be broken, but should be radiused and smoothly blended into adjacent surfaces (Ref. 15).

(2) Blind holes and small-diameter holes should be avoided. These are difficult to coat, the degree of difficulty being a function of the coating method employed (Ref. 14).

(3) Strength requirements should be taken into account; coatings should not be considered as contributing to the load-carrying ability of the part. This is a particularly important consideration in the case of diffusion coatings on thin sections, in which the diffusion zone may occupy a large fraction of the cross-sectional area (Ref. 14).

The importance of obtaining a clean surface prior to coating cannot be overemphasized. The presence of dust, dirt, oxides, oil, grease, fingerprints, or similar contaminants on the surface of a part being coated can result in the formation of a coating that is discontinuous, has poor adhesion, and contains chemical species that are detrimental to the properties desired of the coating.

Specific cleaning procedures for preparing the surfaces of nickel and nickel-base alloys prior to coating are generally regarded as proprietary; this is particularly true in the case of cleaning prior to the application of diffusion coatings. In the few cases for which significant information regarding cleaning procedures was available, it was included in this report.

Among the methods that are used to clean metal surfaces in general prior to the application of coatings are polishing on a cotton wheel, vapor blasting, grit blasting, and pickling (Ref. 16).

There is no indication that pickling is used to any great extent to prepare nickel or nickel-base alloys for coating-type surface treatments. Readers interested in this topic should contact the producer of the particular alloy, inasmuch as pickling procedures vary considerably from alloy to alloy. Two particularly knowledgeable sources of information on pickling are the Huntington Alloy Products Division of The International Nickel Company, Inc., and Stellite Division, Union Carbide Corporation.

Degreasing can be accomplished by washing in a warm detergent, rinsing and drying in an oven (Ref. 16), or by the use of

TABLE I. SELECTED N

Name	Originator	Form	C	Cr	Co	Mo	
Wrought Nickel	The International Nickel Company, Inc.	Wrought	0.06	--	--	--	
TD Nickel	E. I. du Pont de Nemours & Company, Inc.	Wrought	--	--	--	--	
Monel K-500	The International Nickel Company, Inc.	Wrought	0.15	--	--	--	
Inconel X-750	The International Nickel Company, Inc.	Wrought	0.04	15.0	--	--	
Astroloy	General Electric Company	Wrought	0.06	15.	15.	5.25	
Monel 400	The International Nickel Company, Inc.	Wrought	0.12	--	--	--	
Inconel 600	The International Nickel Company, Inc.	Wrought	0.04	15.8	--	--	
Mar-M 200	Martin Metals	Cast	0.15	9.0	10.0	--	12
Udimet 500	Special Metals, Inc.	Wrought	0.15(a)	17.5	16.5	4.0	
		Cast	0.10(a)	19.0	18.0	4.0	
Udimet 700	Special Metals, Inc.	Wrought	0.15(a)	15.0	18.5	5.2	
René 41	General Electric Company	Wrought	0.12(a)	19.0	11.0	9.75	
		Cast	0.09	19.0	11.0	9.75	
Hastelloy B	Stellite Division, Union Carbide Corp.	Wrought or cast	0.10	0.6	2.5(a)	28.0	
Hastelloy X	Stellite Division, Union Carbide Corp.	Wrought or cast	0.10	22.	1.5	9.0	0
Waspaloy	Pratt & Whitney Aircraft	Wrought or cast	0.10(a)	19.5	13.5	4.25	
IN-100	The International Nickel Company, Inc.	Cast	0.18	10.0	15.0	3.0	
Alloy 713C	The International Nickel Company, Inc.	Cast	0.12	12.5	--	4.20	
B-1900	Pratt & Whitney Aircraft; James F. Baldwin	Cast	0.10	8.0	10.0	6.	0
Inconel 718	The International Nickel Company, Inc.	Wrought	0.04	18.6	--	3.1	

(a) Maximum.
(b) Low.

EL-BASE ALLOYS

Nominal Composition, per cent									Reference
Ni	Cu	Fe	Al	Ti	Cb	V	B	Other	
99.5	0.05	0.15	--	--	--	--	--	--	2
98.0	--	--	--	--	--	--	--	2.0ThO$_2$	3
Bal	29.5	1.00	2.80	0.50	--	--	--	--	2
Bal	0.05	6.75	0.80	2.50	0.85	--	--	--	2
Bal	--	--	4.4	3.5	--	--	0.03	--	4
Bal	31.5	1.35	--	--	--	--	--	0.90Mn, 0.15Si, 0.005S	2
Bal	0.10	7.2	--	--	--	--	--	0.20Mn, 0.20Si	4
Bal	--	--	5.0	2.0	1.0	--	0.015	--	4
Bal	0.15$^{(a)}$	4.0$^{(a)}$	2.90	29.0	--	--	0.010$^{(a)}$	--	5,6
--	0.10$^{(a)}$	2.0$^{(a)}$	3.00	3.00	--	--	0.010$^{(a)}$	--	
Bal	--	1.0$^{(a)}$	4.25	3.5	--	--	0.05$^{(a)}$	--	4
Bal	--	5.0$^{(a)}$	1.50	3.15	--	--	0.006	--	7,8
Bal	--	5.0$^{(a)}$	1.65	3.15	--	--	0.003$^{(a)}$	--	7,8
Bal	--	5.0	--	--	--	0.30	--	0.8Mn, 0.7Si	4
Bal	--	18.5	--	--	--	--	--	0.5Mn, 0.5Si	4
Bal	0.50$^{(a)}$	2.0$^{(a)}$	1.30	2.90	--	--	0.006	0.07Zr	9
Bal	--	(b)	5.5	5.0	--	1.0	0.015	0.05Zr	10
Bal	--	(b)	6.10	0.80	2.20	--	0.012	0.10Zr	11
(a) Bal	--	0.35$^{(a)}$	6.0	1.0	0.10$^{(a)}$	--	0.015	4.3Ta, 0.07Zr	12
Bal	0.10$^{(a)}$	18.5	0.40	0.90	5.0	--	0.004	--	2,13

organic solvents. Among the solvent-cleaning methods that have been found to be satisfactory for degreasing nickel and nickel-base alloys are mechanical scrubbing with perchloroethylene, vapor degreasing with trichloroethylene, and vapor degreasing with perchloroethylene (Ref. 17). Trichloroethylene, acetone, and other solvents have been found in some cases to leave a residue film on the work. This film cannot be tolerated on parts that are to be coated. The film can be removed with a hot (180 to 200 F) solution of 10 to 20 per cent sodium carbonate (soda ash) and 10 to 20 per cent trisodium phosphate in water. Sodium hydroxide (caustic soda) may be used instead of sodium carbonate if desired. After immersion in the solution for 10 to 30 minutes, the metal should be thoroughly rinsed with water (Ref. 17).

DIFFUSION COATING

As the name implies, the formation of diffusion coatings involves the diffusion of atoms of chemical elements. In practice, the atoms of an alloying element (or elements) are deposited on the surface of the metal to be coated (often called the substrate) by any one of a number of methods. Heat treatment at high temperatures, either concurrently with the deposition of the alloying elements or as a subsequent operation, causes the atoms to diffuse into the substrate, forming a multielement coating that generally consists of a number of phases of varying composition.

The choice of a specific coating procedure (including coating method – pack cementation, vapor plating, slurry process, or hot dipping – and coating materials) for diffusion coating a given nickel-base alloy depends on the environment to which the alloy will be subjected in service. A procedure that yields a coating that affords satisfactory protection under a given set of operating conditions may not be the choice when other conditions are involved.

For a given service environment, the optimum coating procedure depends on the substrate to be coated. This circumstance arises because the final diffusion coating is formed by the interaction of the atoms of the deposited material with those of the substrate; hence, the phases present in the coating and their composition are affected by the chemical composition of the substrate material.

Published data are quite limited in regard to coating method – substrate combinations and test environments. Accordingly, because the substrate material and the service environment govern the choice of coating method it is usually not possible to select a specific diffusion-coating procedure on the basis of published data alone. Instead, a commonly used procedure for choosing a coating for a given application is to have vendors coat parts or coupons for comparison testing at the customer's plant.* The best test procedure is, of course, to subject the coated coupons to the actual service environment; however, this often cannot be accomplished and simulated exposure tests are used. If the economics of the situation are favorable, the vendor may undertake the development of a coating with the desired properties.

General Methods. Before presenting the specific methods used to obtain particular diffusion coatings, the four general methods used to apply diffusion coatings to nickel and nickel-base alloys will be discussed. These methods are pack cementation, vapor plating, slurry processes, and hot dipping. The four methods are alike in that each involves a thermal treatment at a high temperature to allow diffusion of the alloying elements inward from the surface of the substrate. This treatment is a separate operation in the slurry and hot-dipping process, while in pack cementation it occurs concurrently with deposition. In vapor plating the diffusion treatment can either be a separate operation or can occur concurrently with deposition. The four methods differ in regard to the manner in which the alloying element or elements are brought into contact with the surface.

Pack Cementation. In the pack-cementation method, parts to be coated are first cleaned and then packed in a mixture of powders (often called the "pack mix") in a metal retort. A schematic representation of a retort and its contents in a heat-treating furnace is given in Figure 1. Figure 2 shows typical retorts and furnaces for production-scale operations.

The pack mix consists of an inert filler material, a halide salt, and a source (a metal or alloy) of the elements to be deposited; all

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*Although equipment, materials, and instructions to apply coatings by plating or hard-facing techniques may be purchased and used successfully by any competent organization, commercial diffusion coatings usually must be applied by the companies that have developed these coatings.

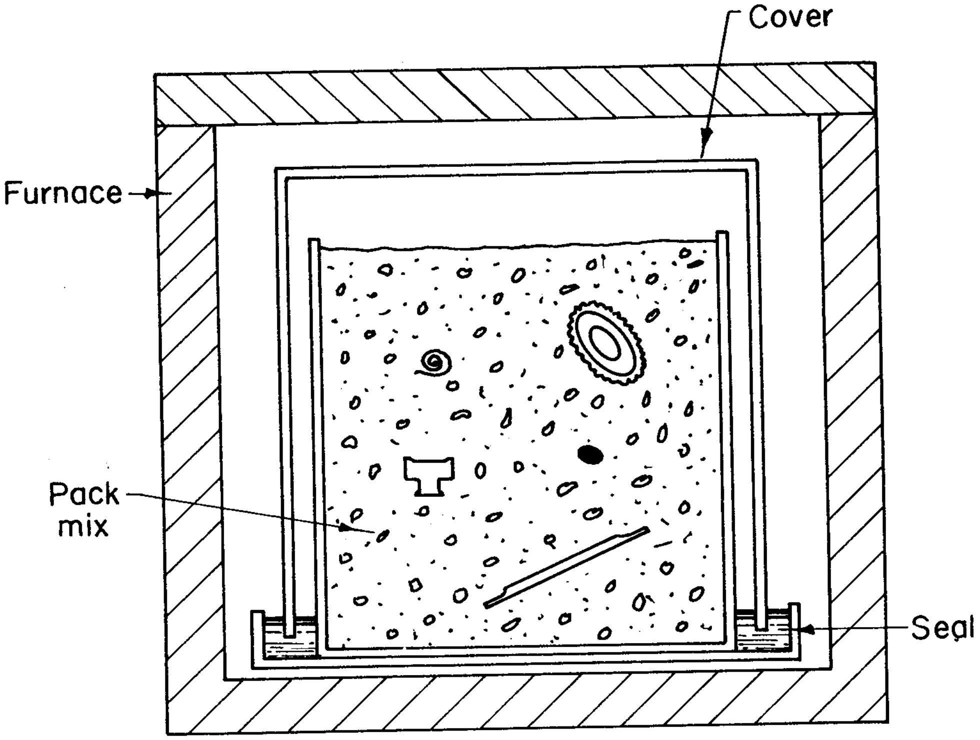

FIGURE 1. SCHEMATIC REPRESENTATION OF PACK-CEMENTATION RETORT AND CONTENTS IN HEAT-TREATING FURNACE (REF. 16)

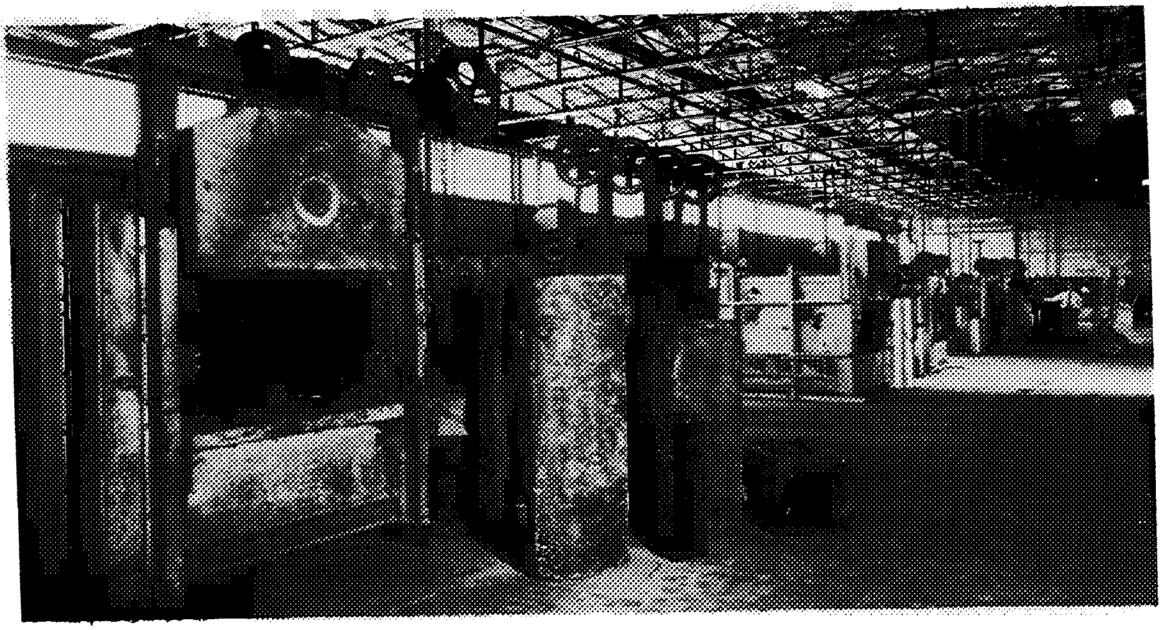

FIGURE 2. TYPICAL RETORTS AND FURNACES FOR PRODUCTION-SCALE PACK-CEMENTATION OPERATIONS

Courtesy of Chromalloy Division, Chromalloy Corporation.

materials are in powder form (Refs. 16, 18). The function of the inert material is to prevent sintering. The halide salt, often called the "activator", serves as a source of halogen atoms at elevated temperatures. The source material can be any metal that forms a halide that has a reasonably high vapor pressure.

After packing is accomplished, the retort is sealed by a method that will provide for easy opening and closing and heated in a furnace to a predetermined temperature. A common method of sealing is to use a fusible silicate material for the seal (Refs. 19, 20). By this method, gases can escape as the retort is heated, a good liquid seal is obtained at temperature and a solid silicate seal sufficient to withstand the contraction of gases within the retort is provided during cooling (Ref. 19). Breaking the seal when the retort has cooled sufficiently is a simple matter. In some cases, the retort is covered but not completely sealed. To prevent contamination, it is placed with other retorts in a large bell-type chamber in which an inert atmosphere is maintained (Ref. 21). Figure 3 is a photograph of such a chamber being removed from a pit-type heat-treating furnace at the conclusion of the diffusion treatment.

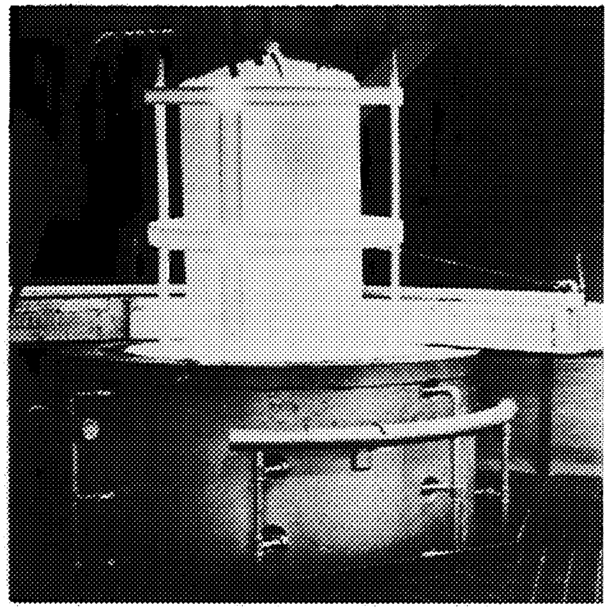

FIGURE 3. BELL-TYPE CHAMBER, CONTAINING PACK-CEMENTATION RETORTS, BEING REMOVED FROM FURNACE

Note gas lines entering top of chamber.

Courtesy of Misco Precision Casting Company and American Machinist.

Pack cementation may also be performed in a retort under vacuum conditions.

At temperature, the halogen atoms from the halide salt unite with atoms of the source material to form one or more metal halides. On coming in contact with the surfaces of the parts, molecules of this halide(s) decompose, whereupon the alloying element(s) deposits on the surface. The alloying elements then diffuse into the part and form a diffusion coating with atoms of the alloy while the retort is at temperature. The temperature at which pack cementation is carried out is chosen according to whether both diffusion in the substrate and the desired chemical reactions in the pack will occur at reasonable rates. Unfortunately, these temperatures may be so high and cooling rates so low that an undesirable microstructure results in the interior of the nickel-base-alloy parts. Accordingly, it is sometimes necessary to heat treat the parts after coating to establish the desired microstructure and properties.

A modification of the pack-cementation method involves the use of a remote activator in a method known as "halogen streaming" (Ref. 22). As shown in Figure 4, the halogen vapors are introduced into the retort under a ceramic gas-diffuser plate; the plate is held at a preselected temperature. The seal of the retort is made of sand, which allows the halogen to leave the retort while maintaining a positive pressure inside (Ref. 22).

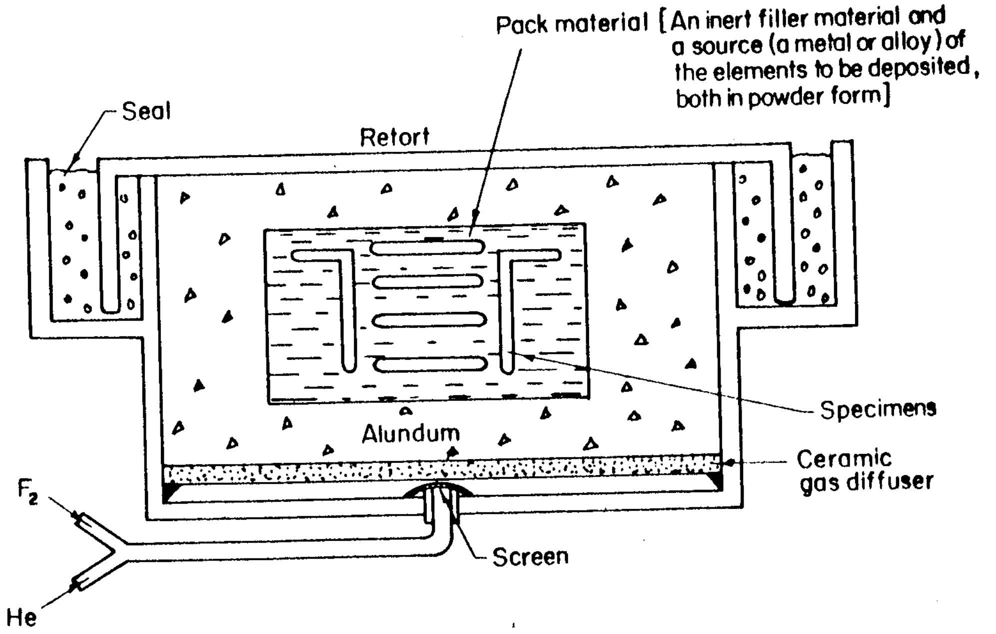

FIGURE 4. SCHEMATIC DIAGRAM OF RETORT AND CONTENTS IN HALOGEN-STREAMING PACK-CEMENTATION TECHNIQUE (REF. 22)

The advantages claimed for the use of halogen streaming instead of a halide salt as the source of the halogen are as follows(22):

(1) It permits regulation of the gas-flow rate and halogen content in the retort.

(2) It provides for introduction of the halogen to the retort to be started and discontinued when desired.

(3) It allows for the employment of different halogens in one operation, at any desired temperature.

(4) The retort can be purged and filled with inert gases at any time during processing.

(5) It eliminates the ion variable that is present when halide-salt activators are employed. When halide salts are used, these ions can enter into the vapor-phase reaction as impurities during the deposition process.

It is thought that the use of the halogen-streaming pack-cementation technique will enable greater process control and thereby the achievement of more reproducible and reliable coatings in the future (Ref. 22). Also, the technique should permit depositing different elements using the optimum halogen and application temperature for each, in a single coating cycle (Ref. 22).

Although at least two companies (Refs. 22, 23) are believed to be using this method to some extent at the present time, no data were available on coatings that were identified as having been produced by the halogen-streaming method.

A second modification of the pack-cementation method, namely, the slip-pack technique, is used when particularly large parts are to be coated. This technique involves applying a slurry of the pack materials to the part by spraying, dipping, or brushing. Heating to effect formation of the coating is usually performed in a furnace under an inert atmosphere.

The slip-pack technique is also used to repair coated parts in the field. In this case, the required heat is usually applied either by furnace heating (Refs. 16, 22), by torches (Refs. 16, 22), by radiant-heat lamps (Ref. 16), or by the ignition of an exothermic material over the patched area (Ref. 22). To prevent contamination of the coating when heat is applied by means of a torch or an air-atmosphere furnace, an over slurry of tabular alumina and a suitable binder (e.g., Na_2SiO_3) has been used (Ref. 22). The over slurry has not been employed when heating was accomplished by means of an exothermic reaction (Ref. 22).

The nature of the pack-cementation process allows complex shapes to be coated, the size of the parts being limited primarily by the size of available retorts and heat-treating furnaces. The large stator ring shown in Figure 5 is indicative of the size of parts that have been coated by this process.

FIGURE 5. STATOR RING COATED BY PACK CEMENTATION

Courtesy of Chromalloy Division, Chromalloy Corporation.

When items that will be later brazed, welded, or otherwise joined to other parts are to be coated, it is necessary that the areas to be joined be left uncoated. Examples of such areas are the machined root sections on turbine blades and the shroud sections on vanes (Ref. 24). These areas must be masked during the coating operation. The masking material must not be porous, must not react with the substrate at the temperatures involved in the pack-cementation process, and must be readily removable at the end of the coating cycle. The methods and materials used for masking are considered proprietary, and are the subject of patent applications.

Vapor Plating. The vapor-plating* method is occasionally used to produce diffusion coatings. A gaseous metal halide or organometallic compound, in an inert carrier gas, is passed over a clean heated substrate in a chamber at reduced pressure. The halide or organic compound thermally decomposes on the substrate, depositing a coating of the metal and liberating the halogen or organic vapor, which is pumped from the chamber. If the substrate temperature required for decomposition is high enough, diffusion may occur during the deposition process. Otherwise, the coated part must be given a separate diffusion heat treatment to form the desired coating.

Reactions other than thermal decomposition can be used to deposit metal on the substrate. One of these is the hydrogen reduction of a metal-containing chemical compound. In this case, hydrogen is also passed into the chamber with the gaseous compound and carrier gas.

The advantages of vapor plating are that the coating can be applied readily in bores and threaded holes, and that there is no chance (as there is in pack cementation) that powder will sinter to the surface being coated (Ref. 16). The disadvantage is that the required vacuum and gas-flow metering equipment and support fixtures are relatively expensive (Ref. 16).

Slurry Methods. There are two methods by which a slurry can be used to form diffusion coatings on nickel-base superalloys.** The primary difference between the two methods lies in whether or not the source material melts during the heat treatment used to form the diffusion coating.

In both methods, the slurry may be applied to cleaned substrates by spraying with a gun, dipping (see Figure 6), or brushing. The slurry is formed by blending the coating material in powder form and suspending the powders in a liquid carrier. In order to avoid roughness and agglomeration in the diffused coating, small particle sizes (in some cases below 325 mesh) must be used (Ref. 26). The primary requirements for the carrier are that it

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*The term "vapor plating" for the method described here is not universal. The method is known by a number of other names: chemical vapor deposition (which includes vapor plating and pack cementation), vapor streaming, and vapor cementation. The important point is to distinguish vapor plating from vacuum metallizing (also known as vacuum plating and physical vapor deposition), in which the feed material undergoes no deliberate chemical reaction at the substrate surface.

**In addition to the slip-pack technique, which utilizes a slurry but is generally classified as a variation of the pack-cementation technique because the slurry contains a halide activator.

hold the coating particles in suspension and not react with the substrate or form undesirable compounds in the coating. Organic liquids, which decompose on heating or burn off without forming carbides, are commonly used as carriers (Ref. 26).

FIGURE 6. DIP COATING OF TURBINE-ENGINE COMBUSTION LINER BY SLURRY PROCESS FOR SUBSEQUENT DIFFUSION TREATMENT (REF. 25)

Courtesy of Solar, a Division of International Harvester Co.

After the slurry has been dried in air, the part is heated in an inert atmosphere or in vacuum to allow diffusion of the coating materials to take place. In one slurry method, the coating materials in the dried bisque react without melting and diffuse into the substrate (Ref. 25). In the second technique (probably used only for aluminum-diffusion coatings and rarely on nickel-base-alloy substrates) the bisque is heated to 1300 to 1400 F, causing the aluminum in the bisque to melt and flow over the part. The part is then heated at a temperature of 1700 to 2200 F, to cause diffusion of the aluminum into the substrate and effect the formation of the desired coating (Ref. 25).

An advantage cited for slurry coating over pack cementation is that higher cooling rates are possible, thereby simplifying control of the microstructure of the finished part (Ref. 27). Another advantage, at least with the spray (Ref. 27) and brushing methods of application, is that masking of areas to be left uncoated is easier than in the case of pack cementation. A disadvantage of the dipping method of application is the difficulty in coating blind holes.

Hot Dipping. In the hot-dipping process, the cleaned substrate is immersed in a molten bath of the coating material.

Generally, but not always, the bath is covered with a flux to prevent oxidation of either the molten metal or the substrate as it is inserted (Figure 7) (Ref. 26). To prevent solution of the substrate in the liquid metal, the duration of the immersion is limited to a few minutes. On the other hand, this short time allows very little diffusion of the coating material into the substrate. Consequently, in order to obtain a diffusion coating the hot-dipped part must be heat treated. This is usually done in an inert atmosphere or in vacuum (Ref. 26).

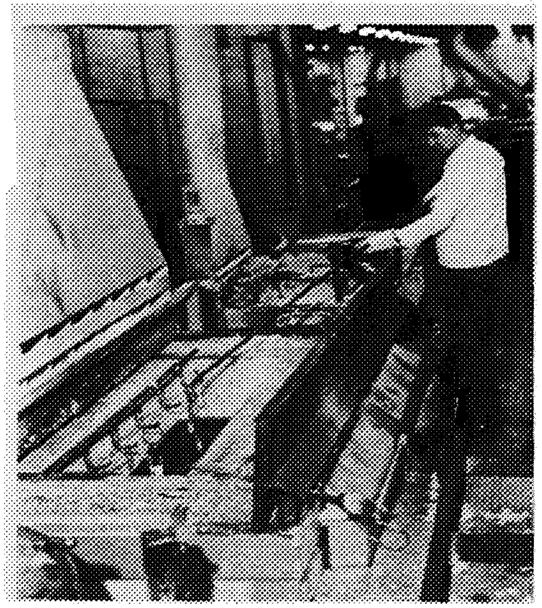

FIGURE 7. PRODUCTION EQUIPMENT FOR THE FORMATION OF ALUMINUM COATINGS BY HOT DIPPING SHOWING FLUXING SALT AND ALUMINUM FURNACES

Courtesy of Allison Division, General Motors Corp.

Disadvantages that are sometimes found in using the hot-dipping technique include the difficulty of obtaining a uniform coating on complex shapes or blind recesses, poor coverage on corners, and the formation of inclusions in the coating by oxidation as the substrate enters the bath (Ref. 26).

Applications for Diffusion Coatings. Most work on diffusion coatings for nickel-base alloys has been directed toward improving these alloys as materials of construction for critical components of aircraft, marine, and automotive gas-turbine engines. This is by far the largest volume application of diffusion-coated nickel-base alloys. Typical items of hardware in such engines that are often prepared from coated nickel-base alloys are combustion chambers, turbine blades, and turbine vanes (Figures 8, 9, and 10, respectively).

For reasons of economy, engine manufacturers and users strive to increase the time between engine overhauls and, by operating at

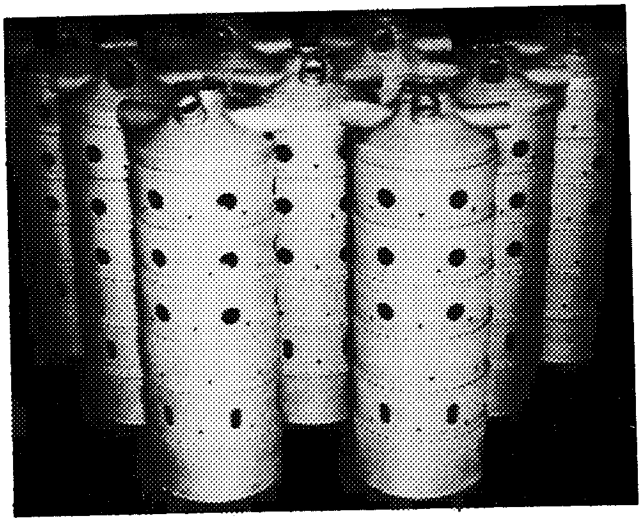

FIGURE 8. COMBUSTION CHAMBERS FABRICATED FROM HASTELLOY X AND PROTECTED WITH A DIFFUSED ALUMINUM COATING

Courtesy of Whitfield Laboratories, Inc.

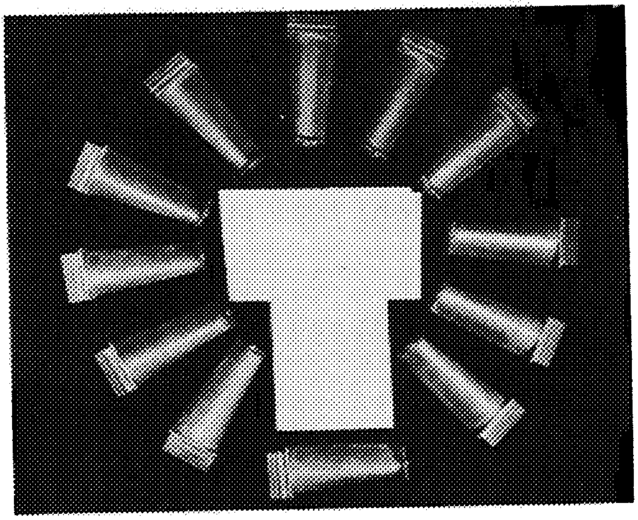

FIGURE 9. FIRST-STAGE TURBINE BLADES OF CAST WASPALOY PROTECTED WITH A DIFFUSED ALUMINUM COATING

Courtesy of Whitfield Laboratories, Inc.

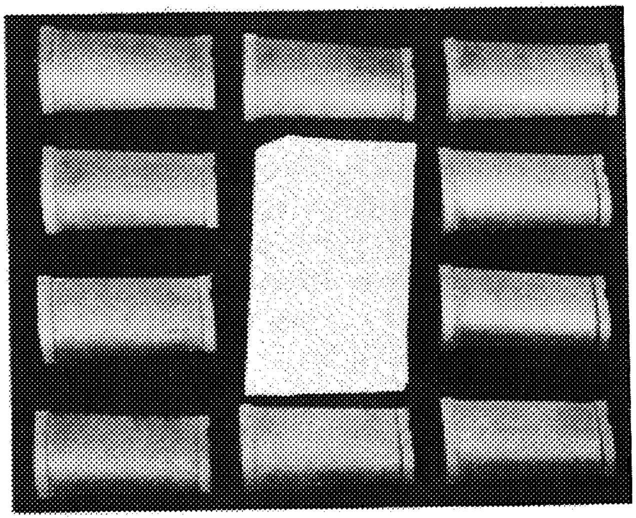

FIGURE 10. TURBINE VANES OF A NICKEL-BASE ALLOY PROTECTED WITH A DIFFUSED ALUMINUM COATING

Courtesy of Whitfield Laboratories, Inc.

higher temperatures, to increase engine efficiency.* However, as engine temperature is increased, the chemical attack of the turbine parts by the products of the combustion of the fuel increases. Thus, a trade-off between the two objectives has been necessary.

At the present time uncooled nickel-base-alloy blades and vanes are being used in gas-turbine engines at temperatures to 1700 F (Ref. 29). Covering the blades and vanes with a protective coating has substantially extended their useful life at these temperatures. When mechanically cooled hardware is used, operation to 2200 F should be possible if a suitable coating material were available to protect the outer metal surface. At the present time, however, available coatings have permitted operation only to 1900 F (Ref. 29).

Because of the complexity of both the corrosive environment and the time-temperature profile of materials in gas-turbine engines, diffusion coatings are called upon to concurrently provide a number of different types of protection to the underlying metal. Some of these are as follows:

(1) Resistance to high-temperature oxidation, both static and cyclic

(2) Resistance to sulfidation at high temperatures

(3) Resistance to erosion by flames and hot gases

(4) Resistance to impact by foreign objects, particularly at high temperatures

(5) Resistance to mechanical and thermal fatigue (including thermal shock).

The coating must also possess diffusional stability with the alloy and a melting point high enough to withstand momentary overshoots in operating temperature. No coating has yet been developed that completely meets the requirements of engine manufacturers and users, although research is in progress to develop such a coating. This work is sponsored by engine manufacturers, by coating vendors, and by the United States Government.

The military's interest in this problem is shown by the request for proposal (Ref. 30) for "the development of a coating which shall

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*A 3 per cent rise in temperature can increase power output by as much as 12 per cent (Ref. 28).

possess the capability to protect nickel and cobalt alloys in gas turbine hot-section components of advanced engines against environmental effects" issued in November, 1965, by the U. S. Air Force, and by the U. S. Navy's sponsorship of the Gas Turbine Engine Conference in April, 1965 (Ref. 31). At about the same time as the Conference, the Navy initiated an in-house program to study this type of corrosion and to develop an alloy to withstand it without the necessity of being coated (Ref. 32).

At present, the most pressing problem in these applications is one of sulfidation. This sulfidation is not to be confused with the type of sulfidation that producers and users of nickel and nickel-base alloys have been familiar with and have had to contend with for many years. [The latter type of sulfidation problem is thought to result from the reaction of sulfur-containing environments with areas of the alloy previously depleted in chromium, thereby forming a nickel-sulfur eutectic that melts at about 1190 F (Ref. 33).] Instead, the sulfidation referred to in gas-turbine technology is attributed to the action of a molten slag that is the product of a reaction between ingested seawater and sulfur contained in the combustion products of the fuel (Ref. 34). Very low concentrations of seawater in the ingested air are sufficient to promote the problem. Although there are a number of different theories regarding the specific mechanisms by which the attack occurs, it is generally agreed that (Ref. 34)

(1) A molten slag composed principally of Na_2SO_4 is formed.

(2) The protective oxide film on the surface of the superalloy is destroyed.

(3) The sulfur in the slag reacts with the superalloy, stimulating gross and rapid oxidation.

Most of the diffusion coatings that are being applied to nickel and nickel-base alloys in the United States are based on (or at least rich in) aluminum. A few proprietary beryllium-base, zirconium, and zirconium-chromium coatings have been developed but test data are not presently available. Boron diffusion coatings have also been investigated, but most of this work has been done in the Soviet Union. Carbon and nitrogen have been diffused into nickel and nickel-base alloys. Although such diffusion has usually been the result of accidental contamination from an environment during heat treatment or service rather than a deliberate attempt to form a diffusion coating, diffusion of carbon and diffusion of nitrogen will be considered as types of diffusion coatings in this report.

Having described the general coating methods, the applications for diffusion coatings, and the types of diffusion coatings, the state of the art of specific diffusion coatings for nickel and nickel-base alloys will be discussed. Coating techniques will be described in detail whenever possible, considering the proprietary nature of such information. Data on the properties of diffusion-coated nickel and nickel-base alloys will also be presented. Such data are very difficult to obtain, even by personal interview. Accordingly, in many instances the only available information was that given in manufacturers' literature. Due to the lack of standardization of test procedures and conditions, only seldom can a comparison be made of the performance of the various diffusion coatings that are presently available.

Following convention, the diffusion coatings will be categorized according to the composition of the source material in the pack, slurry, or hot-dip bath in these methods of application and according to the composition of the coating prior to diffusion in the case of the vapor-plating method. The composition of the actual coating, as noted earlier, depends not only on the composition of the source material but also on the substrate. Also, differences in the vapor pressures of the elements or halides of the elements in pack sources can cause the composition of the coating to vary considerably from that of the source.

Aluminum. The Aldip process of Allison Division, General Motors Corporation, is a hot-dip method that can be used to apply aluminum diffusion coatings to nickel-base and other alloys. The process usually involves four steps: (1) cleaning of the substrate, (2) preheating and fluxing, (3) coating, and (4) diffusion (Ref. 26). The coating bath is molten 1100-grade aluminum at about 1300 F with a layer of molten slag floating on top to prevent oxidation (Ref. 26) (Figure 7). The diffusion treatment is accomplished at temperatures between 1700 and 2150 F, depending on the substrate alloy and the intended application of the part (Ref. 26). Specific information regarding the Aldip process is considered proprietary (Ref. 35).

Lycoming Division, Avco Corporation, Stratford, Connecticut, also applies aluminum coatings to nickel-base alloys by hot dipping followed by diffusion (Ref. 36); its 601 coating is of this type. Details of the coating process are proprietary. The coating is said to significantly improve the oxidation resistance of nickel-base alloys without adversely affecting stress-rupture life (Ref. 36). No specific properties of nickel-base alloys with this coating were available to the authors.

A third hot-dip-type aluminum coating is Type I AD coating of Whitfield Laboratories, Inc., Bethel, Connecticut. The particular coating process used is said to permit close control of coating thickness (Refs. 37,38). No flux is used, thereby reportedly minimizing stop-off problems when only a portion of an object is to be coated (Ref. 37). Details of the dipping process, which is patented, are proprietary.

Thermal-fatigue tests have been performed on Waspaloy and on Inco 713 C, both uncoated and with Type I AD coating. The test method involved heating the samples for 10 seconds in each of four heating stations. The first station selectively heated the leading edge of the sample, and the other three stations heated the remainder of it. The samples were then blasted with air for 10 seconds in two cooling stations and cooled in stagnant air for a total of 20 seconds in the next two test stations (Ref. 38). For Waspaloy, the temperature cycles were from 200 to 2000 F; for Inco 713 C, they were from 350 to 2000 F. Results of the tests on the two materials are given in Figures 11 and 12. The data show the improvement in thermal fatigue obtained by using these coatings. Also shown in these figures are data for three other types of coatings, which will be discussed later in this report.

Another aluminum coating of Whitfield Laboratories is Type I AG, which is applied by a vapor-plating process (Ref. 37). Details of the process, which affords a high degree of control of coating thickness, are proprietary. The method is said to be particularly useful when complicated shapes, which might trap air or molten metal (as in a hot-dipping operation), are to be coated (Ref. 37).

Data showing the resistance to fatigue imparted by this coating to Waspaloy are given in Figure 11.

The A-11 coating of Martin Metals, Division of Martin-Marietta Corporation, Wheeling, Illinois, is an aluminum diffusion coating. The formation of the coating is a two-step process. First, the aluminum is deposited by a vapor-plating method onto a heated nickel-base-alloy substrate at about 300 F, through the thermal decomposition of an aluminum-containing chemical compound (Ref. 39). The use of such a low substrate temperature eliminates

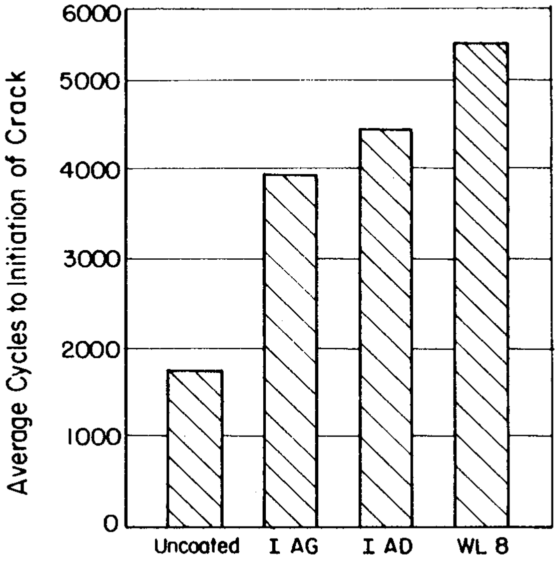

FIGURE 11. RESULTS OF THERMAL-FATIGUE TESTS (200 TO 2000 F) ON WASPALOY (REF. 38)

Uncoated and With Two Aluminum* and One Aluminum-Base** Diffusion Coatings

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*Types I AG and I AD coatings of Whitfield Laboratories, Inc., Bethel, Connecticut.

**Type WL 8 coating of Whitfield Laboratories, Inc., Bethel, Connecticut

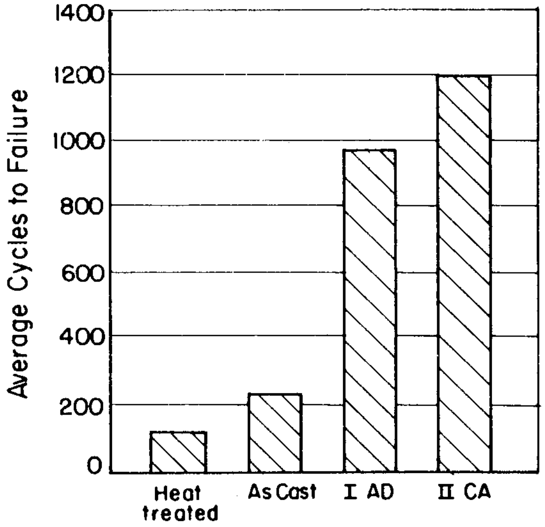

FIGURE 12. RESULTS OF THERMAL-FATIGUE TESTS (350 TO 2000 F) ON TURBINE VANES* OF INCO 713C (REFS. 37, 38)

Uncoated and With Aluminum** and Aluminum-Chromium*** Diffusion Coatings

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*Thickness of trailing edge 0.041 inch.

**Type I AD coating of Whitfield Laboratories, Inc., Bethel, Connecticut.

***Type II CA coating of Whitfield Laboratories, Inc., Bethel, Connecticut.

the possibility of the deposition process interfering with the prior heat treatment of the substrate. Second, the aluminum is diffused into the substrate, which is stimulated by heating at a temperature still below the range that would interfere with the microstructure established by the prior heat treatment of a nickel-base alloy (Ref. 39).

The stress-rupture properties of MAR-M200 and Udimet 700 with the A-11 coating are given in Figures 28 and 29 (Ref. 29), respectively. The figures are given and discussed on pages 66 and 67.

Results of ballistic-impact tests on MAR-M200 and Udimet 700 with the A-11 coating are given in Table XXIII. The test procedure is described in the section of this report that considers aluminum-silicon diffusion coatings.

Although the Martin Metals A-11 coating is not now commercial (Ref. 39), the U. S. Navy's continuing interest in it could possibly change the situation (Ref. 39).

The Ethyl Corporation, Baton Rouge, Louisiana, has an aluminum coating for nickel-base alloys that is formed by the thermal decomposition of an organometallic compound in a vapor-plating process (Ref. 40). To effect decomposition, the nickel-base-alloy substrate is heated to a temperature somewhat below 300 C (572 F). The properties of the coated alloys are said to be at least as good as those obtained when aluminum is applied by a pack-cementation process. Information regarding the coating procedure and data on the properties of nickel-base-alloys with the coating are considered proprietary (Ref. 40).

General Technologies Corporation, Alexandria, Virginia, is developing for commercial use a vapor-plating process whereby aluminum can be deposited on any substrate that can be heated to about 350 F (Ref. 41). The coating is formed by the thermal decomposition of aluminum alkyl (an organometallic compound) on the heated substrate in the presence of a carrier gas and a suppressant gas. The carrier gas may be argon or nitrogen; the type of suppressant gas used is proprietary. The latter gas inhibits unwanted reactions such as those that might lead to the formation of aluminum carbide or aluminum hydride. The advantages claimed for this process over hot dipping are that it is simple and clean and lends itself readily to production lines. Coatings can be deposited at production rates of 2 minutes per mil (0.001 inch) thickness (Ref. 41).

An aluminum diffusion coating was applied to TD Nickel in research at the Langley Research Center of the National Aeronautics and Space Administration (Ref. 42). Aluminum was applied by pack cementation at about 1700 F. The coated samples were then heated to effect diffusion of the aluminum and formation of the desired coating (Ref. 42). Details of the coating process, called "calorizing", were not published. Presumably, the coating referred to is the calorizing coating of The Calorizing Company, Pittsburgh, Pennsylvania (Refs. 43, 44).

Results of oxidation tests in static air on uncoated and coated TD-Nickel specimens at NASA Langley are given in Figure 13 (Ref. 42). The resistance to oxidation afforded by the coating is readily seen. The coating was found to be ductile if bent after exposure at 1800 to 2200 F, but cracked if an attempt was made to bend the specimen in the as-diffusion-coated condition or after exposure at 2300 or 2400 F (Ref. 42).

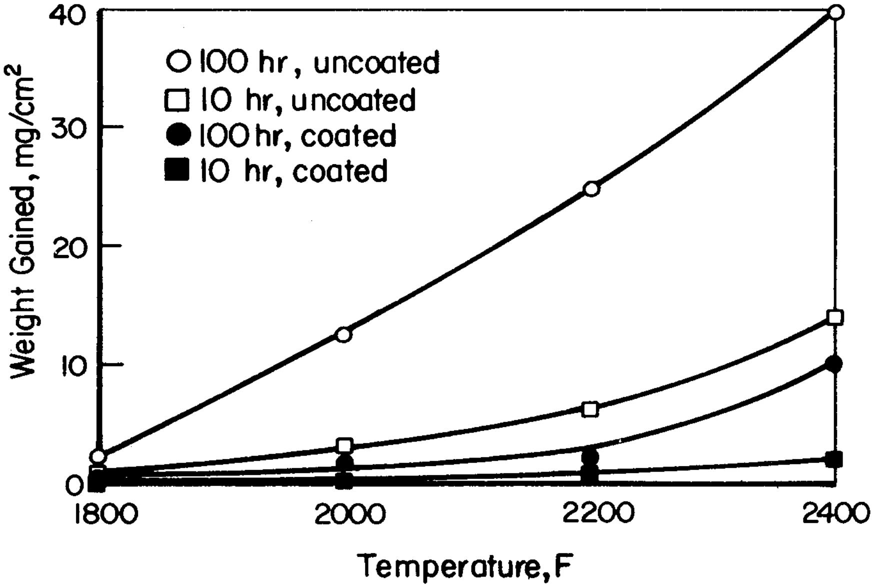

FIGURE 13. COMPARISON OF OXIDATION DATA FOR TD NICKEL SHEET (REF. 42)

Uncoated and With an Aluminum Diffusion Coating*, Exposed for 10 and 100 Hours in Static Air.

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*Presumably the calorizing coating of The Calorizing Company, Pittsburgh, Pennsylvania.

Coating 701 of Lycoming Division, Avco Corporation, Stratford, Connecticut, is an aluminum coating applied by pack cementation in vacuum (Ref. 36). The coating process yields reproducible coatings with good control of thickness; details of the process are proprietary (Ref. 36). Specific properties of nickel-base alloys with this coating were not available to the authors.

An aluminum coating, termed MDC-1A, is applied by Misco Precision Casting Company, Whitehall, Michigan, to nickel- and cobalt-base alloys (Ref. 24). Information regarding the coating method (believed to be pack cementation) or the properties of nickel-base alloys coated with it was not available to the authors.

Aluminum With Unidentified Nonmetallics. A diffusion coating that consists of aluminum with a dispersion of nonmetallics has been developed by the Misco Precision Casting Company, Whitehall, Michigan (Ref. 21). Called MDC-1, the coating is used under conditions in which improved vibrational fatigue characteristics, strength at high temperatures, oxidation resistance, and/or resistance to sulfidation are required. The coating is applied by a pack-cementation technique in which the substrates are prepared by grit blasting and vapor degreasing. After deposition, the coating is smoothened by polishing for a short length of time using a vibratory method (Ref. 24). Details of the pack-cementation process are proprietary.

This coating was applied to small (1/8 x 1/4 x 2-3/8 in.) investment-cast specimens of Inco 713 C in a program sponsored by the Bureau of Naval Weapons at Phillips Petroleum Company, Bartlesville, Oklahoma. The purpose of the research was to determine if a reduction in the maximum sulfur content of 0.4 weight per cent currently allowed in Grade JP-5 aviation turbine fuel would significantly reduce the corrosion of turbine-blade alloys used in high-performance engines in a marine environment (Ref. 45). The specimens were exposed under conditions that represent those in gas-gas-turbine engines of advanced design. The specimens were tested in the exhaust gas from a Phillips 2-inch combustor, using a constant combustor pressure of 15 atmospheres, air flow of 7200 pounds per hour, and fuel flow of 120 pounds per hour. The flow velocity of the gas ranged from 685 to 805 feet per second. Fuel-sulfur contents of 0.0002, 0.04, and 0.4 weight per cent were used. "Sea salt" was ingested at levels of 0, 1.0, and 10.0 ppm in air by the injection of synthetic "seawater" (per ASTM D665) into the quench zone of the combustor. Each specimen was exposed in a 5-hour test made up of

TABLE II. SUMMARY OF METAL-WEIGHT-LOSS DATA FOR COATED AND UNCOATED NICKEL-BASE ALLOYS (REF. 45)

Conditions Designed to Represent Those in Advanced Gas-Turbine Engines.

Temperature, F	Fuel Sulfur, wt %	"Sea Salt" in Air, ppm	Geometric Mean Weight Loss, mg/cm^2(a)				
			Inco 713 C	Inco 713 C With MDC-1(b, c)	IN-100	MAR-M200	Udimet 500
1800	0.0002	0	1.1	1.5	1.3	2.5	2.4
		1.0	2.2	1.1	6.8	39.5	4.5
		10.0	55.8	1.2	110.9	141.5	7.0
	0.04	0	2.5	1.2	4.4	5.2	2.7
		1.0	11.4	0.8	24.3	37.7	4.7
		10.0	45.9	1.7	113.3	104.4	5.8
	0.4	0	1.1	1.0	1.8	2.8	2.5
		1.0	16.8	2.0	26.9	24.4	4.0
		10.0	37.0	2.5	21.1	24.1	4.2
2000	0.0002	0	1.6	2.5	2.8	10.1	4.3
		1.0	2.9	1.4	10.6	74.7	6.7
		10.0	31.0	1.7	102.5	235.4	72.1
	0.04	0	1.5	0.6	3.5	7.2	4.3
		1.0	5.5	2.7	13.1	41.3	8.0
		10.0	20.2	1.7	118.7	76.2	25.0
	0.4	0	2.2	1.7	6.2	15.3	4.5
		1.0	5.6	1.4	32.3	37.7	6.7
		10.0	76.6	1.7	66.0	19.0	8.6
2200	0.0002	0	3.7	1.3	7.2	42.7	6.7
		1.0	6.0	0.8	9.8	73.4	7.9
		10.0	60.2	2.1	166.8	685.5	80.0
	0.04	0	10.2	1.2	32.6	82.2	6.5
		1.0	9.5	2.7	34.1	153.6	10.4
		10.0	111.1	2.1	220.2	591.0	8.7
	0.4	0	2.0	1.3	72.5	155.9	5.3
		1.0	10.6	1.6	58.2	184.4	12.4
		10.0	61.9	2.6	165.5	193.6	60.1

(a) Geometric mean is the antilogarithm of the average of the logarithms of the weight loss for three test specimens.

(b) MDC-1 coating of the Misco Precision Casting Company, Whitehall, Michigan.

(c) Some of the weight loss recorded here for the coated specimens occurred during cathodic cleaning after testing.

Note: 95 per cent confidence limits: lower = mean ÷ 2.15; upper = mean x 2.15

For example:

$0.9 < 2.0 \leq 4.3$

$9.3 < 20.0 \leq 43.0$

$93.0 < 200.0 \leq 430.0$.

five 1-hour cycles in which the specimen was exposed to the hot gas for 55 minutes, then cooled for 5 minutes with the fuel off. The exhaust temperatures used were 1800, 2000, and 2200 F, with the test specimens reaching 1840, 2040, and 2230 F, respectively (Ref. 45). As shown in Table II, the MDC-1 coating decreased considerably the weight loss of Inco 713 C under the test conditions. No other coatings were evaluated. Results for other alloys (uncoated) are also given in Table II for comparison.

In a test program sponsored by the Air Force, coated (MDC-1) and aged Waspaloy turbine blades were oscillated at their natural frequency at 1600 F. Cracking at the trailing edge was considered the condition of failure. At the termination of the test at 8 million cycles, none of the four blades coated with MDC-1 had failed. Of the 22 blades tested with four other coatings, 19 had failed by that time; 10 of these had failed before 2.4 million cycles (Ref. 24).

Four nickel-base alloys, uncoated and with the MDC-1 coating, were tested under static oxidation conditions in air at 2000 F for 100 hours. The weight-change data from these tests, indicative of the protection offered by the coating, are given in Table III (Ref. 24). No secondary diffusion occurred during the test period.

TABLE III. RESULTS OF 100-HOUR STATIC OXIDATION TESTS IN AIR AT 2000 F ON FOUR NICKEL-BASE ALLOYS (REF. 24)

Uncoated and With an Aluminum Diffusion Coating Containing Dispersed Nonmetallics(a)

Alloy	Condition	Weight Change, mg/cm^2
Udimet 500	Uncoated	-11.73
	Coated	+0.37
Inco 713 C	Uncoated	-2.93
	Coated	+0.10
IN-100	Uncoated	-97.0
	Coated	-0.49
Waspaloy	Uncoated	-4.49
	Coated	+0.18

(a) MDC-1 coating of Misco Precision Casting Company, Whitehall, Michigan.

Airfoil-shaped specimens of Udimet 500, IN 100, and Inco 713 C, both uncoated and with the MDC-1 coating, were tested in thermal shock at 1800 F (Ref. 24). Each cycle of the test involved heating the specimens to 1800 F in 25 seconds, holding at 1800 F for 35 seconds, cooling to below 300 F in 30 seconds, and holding under cooling water for an additional 60 seconds. The test was designed to simulate a range of conditions found in jet engines. The results of the tests, given in Table IV (Ref. 24), indicate the improvements in resistance to thermal shock imparted by the coating.

The tensile and stress-rupture properties of six nickel-base alloys measured on investment-cast specimens uncoated and with the MDC-1 coating are given in Table V (Ref. 24). It may be noted that the coating does not adversely affect tensile and stress-rupture properties under the stated conditions.

Aluminum With Unidentified Alloying Elements. NC-101 is an aluminum-base diffusion coating of Sylcor Division, Sylvania Electric Products, Inc., Hicksville, New York, that is applied by a pack-cementation process (Ref. 46). The alloying elements in the coating, as well as details regarding the deposition process, are proprietary (Ref. 47). The coating is said to have "some degree of room-temperature ductility" and to be "self-healing at operating temperatures".

Samples of IN-100, both uncoated and with the NC-101 coating, were tested for oxidation resistance by a cyclic method. Each cycle was 1 hour long and involved holding the samples in an oxyacetylene flame at 2000 F for 55 minutes and cooling them to 750 F for 5 minutes. Test results are given in Figure 14. It may be seen that the uncoated sample oxidized rapidly initially and then suffered severe spalling; the coated sample, however, formed a thin, protective oxide layer and remained stable until the end of the test, which was more than 350 hours (Ref. 46).

According to Sylcor (Ref. 47), this is the only releasable information on the effect of this coating on the properties or characteristics of nickel-base alloys. Many additional tests have been performed, but the data are considered proprietary by Sylcor's customers. In other cases, the coatings have been evaluated in Government-sponsored programs but were not identified by name when the data were given in the published reports (Ref. 47).

TABLE IV. RESULTS OF THERMAL-SHOCK TESTS ON THREE NICKEL-BASE ALLOYS (REF. 24)

Uncoated and With an Aluminum Diffusion Coating Containing Dispersed Nonmetallics(a)

Alloy	Condition	Weight Loss, per cent	Cycles at Which Cracking Occurred	Length of Crack at Conclusion, in.
		3000 Cycles at 1800 F(b, c)		
U500	Uncoated	1.855	600, 1400	5/16
	Coated	0.990	2800, —(d)	1/64
IN-100	Uncoated	1.695	—(d)	—(d)
	Coated	0.950	—(d)	—(d)
		4000 Cycles at 1800 F(b, e)		
U500	Uncoated	1.707	1400, 1600, 1600	1/8
	Coated	0.705	4200, 4200, 4200	1/64
IN-100	Uncoated	1.693	3000, 3000, 3000	1/8
	Coated	0.690	3800, —(d), —(d)	1/64
Inco 713 C	Uncoated	2.267	—(d)	—(d)
	Coated	0.502	—(d)	—(d)
		3000 Cycles at 1800 F(b, e)		
U500	Uncoated	1.565	800, 800	1/4, 3/8
	Coated	0.694	1800, 1800	1/8
Inco 713 C	Uncoated	1.333	—(d)	—(d)
	Coated	0.601	—(d)	—(d)

(a) MDC-1 coating of Misco Precision Casting Company, Whitehall, Michigan.

(b) One cycle consists of heating to temperature in 25 seconds, holding for 35 seconds, cooling to below 300 F in 30 seconds, and holding under cooling water for an additional 60 seconds.

(c) Weight loss is average of two samples.

(d) No cracking occurred during test.

(e) Weight loss is average of three samples.

TABLE V. TENSILE AND STRESS-RUPTURE PROPERTIES OF SIX NICKEL-BASE ALLOYS (REF. 24)

Uncoated and With an Aluminum Diffusion Coating Containing Dispersed Nonmetallics(a)

Alloy	Condition	Test Temperature, F	Tensile Strength, psi	Yield Strength, psi	Stress, psi	Life, Hours to Rupture	Elongation, per cent	Reduction of Area, per cent
			Tensile Data(b)					
Inco 713 C	Uncoated	RT	142,800	121,000	--	--	5.0	6.2
	Coated	RT	129,500	112,200	--	--	5.0	4.7
IN-100	Uncoated	1300	138,000	102,000	--	--	11.0	16.7
	Coated	1300	138,200	102,800	--	--	10.5	15.9
U-500	Uncoated	1200	145,500	--	--	--	10.0	13.8(c)
	Coated	1200	153,000	--	--	--	16.0	15.9(d)
MAR-M200	Uncoated	RT	138,200	128,500	--	--	4.5	7.9
	Coated	RT	146,200	131,800	--	--	7.0	13.8
			Stress-Rupture Data(b)					
Inco 713 C	Uncoated	1700	--	--	33,000	50.6	7.5	12.3
	Coated	1700	--	--	33,000	48.2	10.5	15.9
	Uncoated	1800	--	--	22,000	47.8	5.5	5.5
	Coated	1800	--	--	22,000	42.5	5.0	6.5
IN-100	Uncoated	1800	--	--	27,500	24.9	11.5	16.0
	Coated	1800	--	--	27,500	25.1	11.0	13.5
U-500	Uncoated	1700	--	--	25,000	47.8	15.2	24.7(c)
	Coated	1700	--	--	25,000	42.4	17.5	23.0(d)
MAR-M200	Uncoated	1800	--	--	29,000	41.5	2.0	2.4
	Coated	1800	--	--	29,000	47.6	2.5	3.6
Waspaloy	Uncoated	1500	--	--	47,500	74.8	6.1	9.4(e)
	Coated	1500	--	--	47,500	82.9	9.0	16.7(f)

(a) Coating MDC-1 of Misco Precision Casting Company, Whitehall, Michigan.
(b) Data obtained from investment-cast specimens
(c) Heat treated 2100 F/4 hr, 1975 F/4 hr, 1400 F/16 hr.
(d) Heat treated 1400 F/16 hr after coating.
(e) Heat treated 1975 F/4 hr, 1550 F/4 hr, 1400 F/16 hr.
(f) Heat treated 1550 F/4 hr, 1440 F/16 hr after coating.

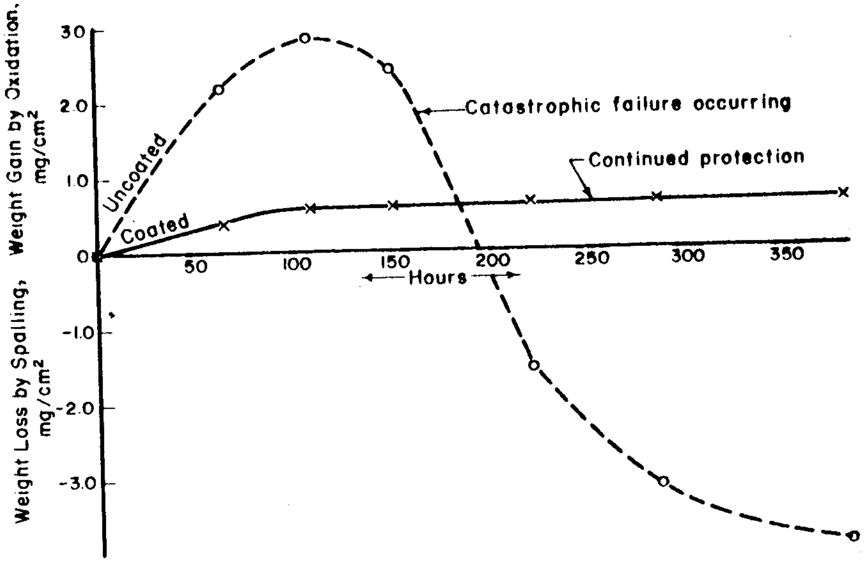

FIGURE 14. RESULTS OF CYCLIC OXIDATION TESTS ON IN-100 (REF. 46)

Uncoated and With an Aluminum-Base Diffusion Coating* in an Oxyacetylene Flame

Another aluminum-base coating with proprietary alloying elements is the MDC-7 coating of Misco Precision Casting Company, Whitehall, Michigan. The coating is said to be applied to nickel- and cobalt-base alloys to improve resistance to sulfidation and oxidation (Ref. 24). Information on the method of applying MDC-7 or on the properties of alloys with this coating was not available.

WL-8, a coating of Whitfield Laboratories, Inc., Bethel, Connecticut, has an aluminum base (Ref. 38). Details concerning the chemical composition and method of application of the coating are proprietary.

The improvement in resistance to thermal fatigue afforded Waspaloy by this coating is shown in Figure 11. The thermal-fatigue test is described on page 23.

Four coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York, fall into the "aluminum with unidentified alloying elements" category; the four are known as OT, MOT, UC, and SUD (Refs. 48-51). All are applied to nickel- and cobalt-base

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*NC-101 coating of Sylcor Division, Sylvania Electric Products, Inc.

alloys by the pack-cementation process. Details of the coating processes are proprietary, although it is known that they require temperatures of 1500 to 2100 F and times measured in tens of hours (Ref. 49).

Wedge-shaped specimens of the nickel-base superalloy B-1900 have been coated with OT, MOT, and UC and tested under oxidation-erosion conditions in the products of combustion of kerosene and air at 2100 F (Ref. 48). The specimens were wedge shaped in order to present radii approximating leading and trailing radii used in turbine buckets and nozzle guide vanes. The results of these tests, given as total weight change as a function of time at 2100 F, are plotted in Figure 15 (Ref. 48). It may be seen that all three coatings protect B-1900 under these conditions and that the OT and MOT coatings are somewhat more effective than is UC.

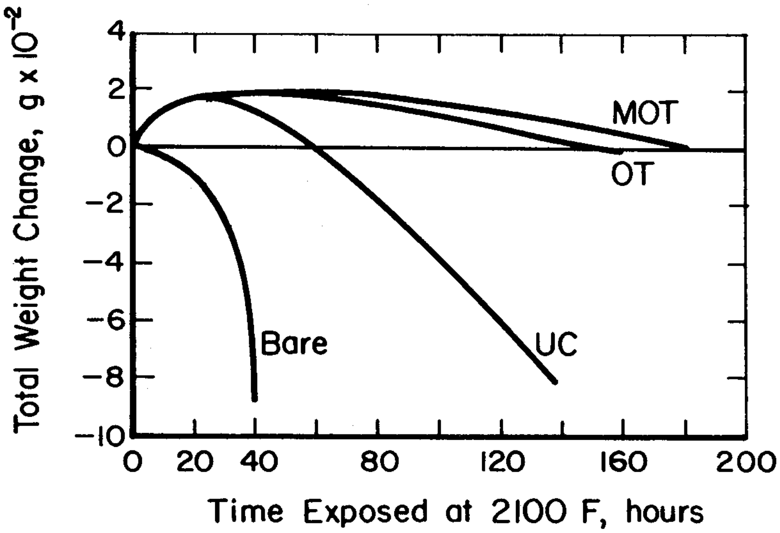

FIGURE 15. TOTAL WEIGHT CHANGE AS A FUNCTION OF TIME FOR B-1900 (REF. 48)

Uncoated and With Three Aluminum-Base Diffusion Coatings* Tested Under Oxidation-Erosion Conditions in the Products of Combustion of Kerosene and Air at 2100 F

Wedge-shaped specimens of Udimet 700, IN-100, and B-1900, each with the SUD coating, were tested under oxidation-erosion conditions in the products of combustion of kerosene and air at 2000 F.

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*MOT, OT, and UC coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

Results of the tests (Ref. 52) plotted as total weight change as a function of time are given in Figure 16.

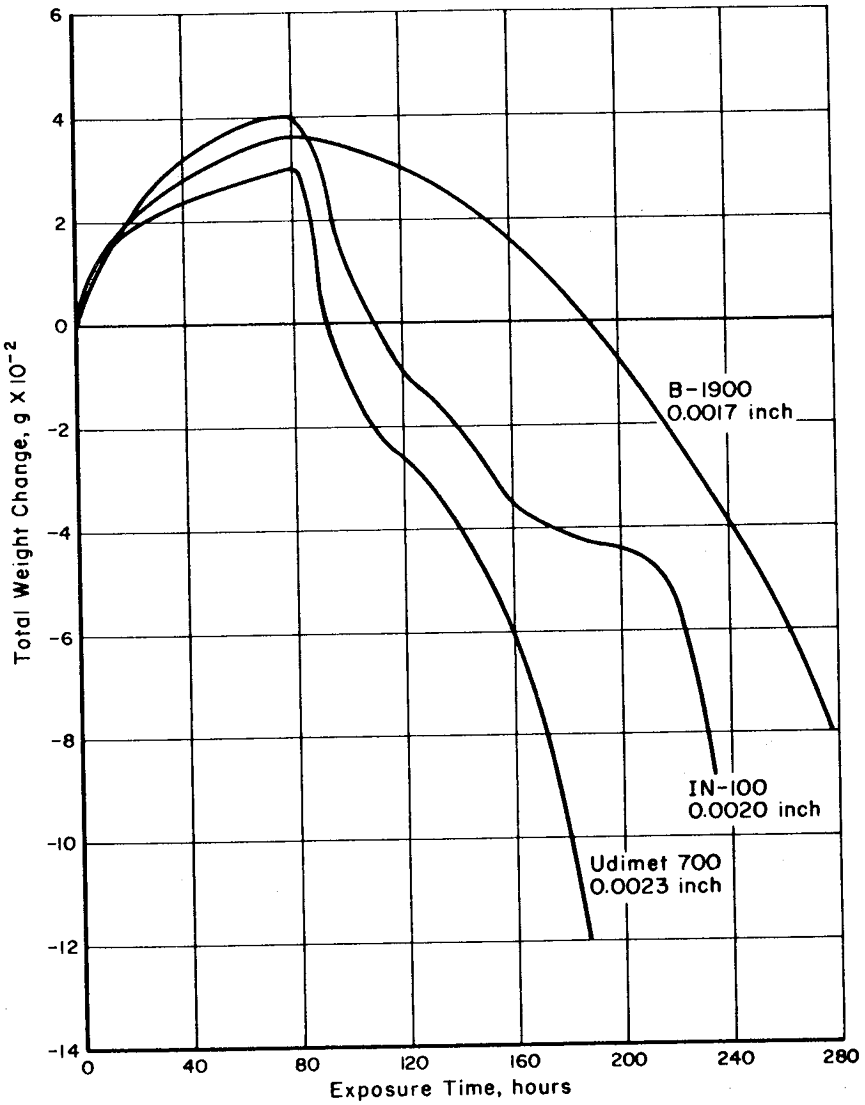

FIGURE 16. WEIGHT CHANGE AS A FUNCTION OF TIME FOR UDIMET 700, IN-100, AND B-1900 (REF. 52)

With an Aluminum-Base Diffusion Coating* Tested Under Oxidation-Erosion Conditions in the Products of Combustion of Kerosene and Air at 2000 F.

Thickness of coating is noted for each substrate.

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* SUD coating of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

The results of static-oxidation tests on B-1900 in air at 2200 F for uncoated specimens and specimens with the OT, SP-OT*, MOT, and SP-MOT* coatings are given in Figure 17 (Ref. 48).

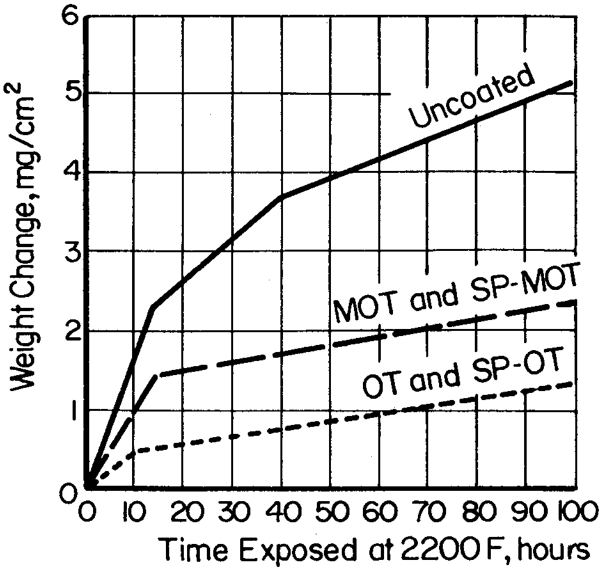

FIGURE 17. WEIGHT CHANGE AS A FUNCTION OF TIME FOR B-1900 (REF. 48)

Uncoated and With Aluminum-Base Diffusion Coatings* During Static Oxidation Testing in Air at 2200 F

*MOT, SP-MOT, OT, and SP-OT coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

In an effort to assess coated B-1900's resistance to damage by foreign objects such as might occur in a gas-turbine engine, specimens have been tested in a drop-weight-type impact tester designed by Chromalloy Division (Ref. 48). The results of the tests are given in Figure 18 (Ref. 48); the SAC coating mentioned in the figure is noted in the section of this report which deals with aluminum-chromium-silicon coatings. A comparison of these results with the microhardness values shown in Figure 19 (Ref. 48) indicates a reciprocal relationship between impact resistance and microhardness (Ref. 48).

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*SP-OT and SP-MOT are special formulations of OT and MOT.

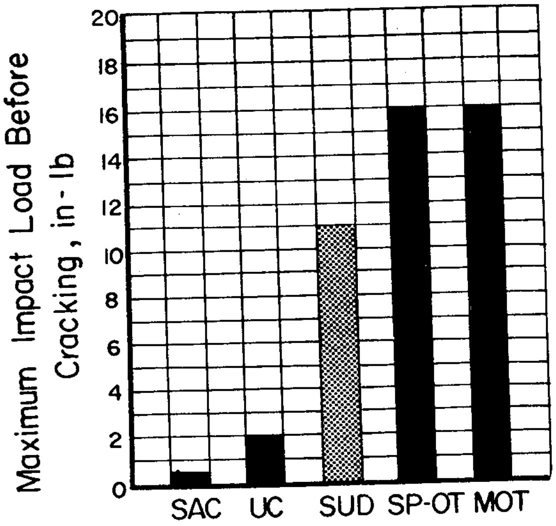

FIGURE 18. RESULTS OF IMPACT TESTS ON B-1900 WITH FIVE ALUMINUM-BASE DIFFUSION COATINGS* AT ROOM TEMPERATURE (REF. 48)

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*SAC, UC, SUD, SP-OT, and MOT coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

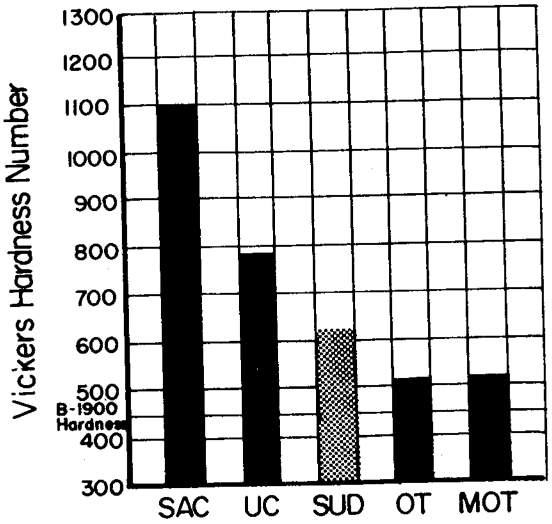

FIGURE 19. AVERAGE MICROHARDNESS OF FIVE ALUMINUM-BASE DIFFUSION COATINGS* ON B-1900 SUBSTRATES (REF. 48)

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*SAC, UC, SUD, OT, and MOT coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

Some concept of the meaning of the results of these tests is given in Figure 20 (Ref. 48). Shown in the figure are samples of B-1900 coated with SAC and with OT, which were stamped with letters. The SAC coating, with its relatively low resistance to impact and relatively high microhardness, spalled when stamped while the OT coating did not.

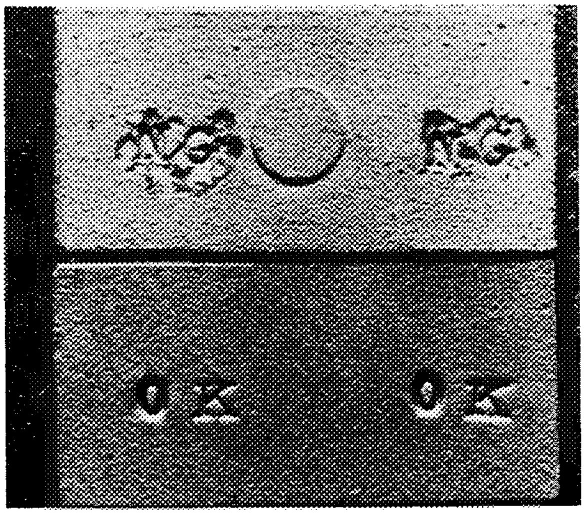

FIGURE 20. APPEARANCE OF SPECIMENS OF B-1900 WITH TWO ALUMINUM-BASE DIFFUSION COATINGS* AFTER STAMPING WITH LETTERS (REF. 48)

An aluminum-base diffusion coating to protect TD Nickel from oxidation at high temperatures has been developed by McDonnell Aircraft Corporation, St. Louis, Missouri (Ref. 53). It is applied as a slurry by dipping. After drying, the bisque-coated substrate is heat treated in argon at a "high temperature" to form a diffusion coating of an intermetallic compound about 0.0015 to 0.002 inch in depth. The chemical composition of both the slurry and the final coating are proprietary (Ref. 53).

TD Nickel with this coating has been found to withstand 50 1-hour cycles between 2200 F and room temperature (Ref. 53). (Cooling to room temperature should be particularly noted, because available thermal-cycling data on many coatings consider cooling to only 350 to 500 F. The difference between these temperatures and room temperature as a choice for the cold end of the cycle may affect considerably the results obtained.) (Ref. 53).

No other data on nickel or nickel-base alloys with this coating were available to the authors.

Aluminum-Chromium. Haynes Diffusion Coating No. C-20, developed by the Stellite Division of Union Carbide Corporation, is an aluminum-chromium coating applied to nickel- and cobalt-base alloys by a pack-cementation technique (Refs. 54, 55). Details of the coating process are proprietary.

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*SAC and OT coatings of Chromalloy Division, Chromalloy Corporation, West Nyack, New York.

Small cast disks (3/4-inch diameter, 5/32 inch thick) of Haynes IN-100 (IN-100) and Haynes 713 C (Inco 713 C) and chromium-plated Haynes 713 C were coated with Haynes Diffusion Coating No. C-20 and tested for 22 hours at 2000 F in the presence of flowing sulfur-bearing gases (Ref. 56). During the test the atmosphere was varied: it was reducing for 2 hours, oxidizing for 2 hours, reducing for 2 hours, and finally oxidizing for 16 hours. The reducing atmosphere consisted of a mixture of nitrogen (at a flow rate of 2 cfh), carbon dioxide (0.07 cfh), and hydrogen sulfide (about 0.04 cfh). The oxidizing atmosphere consisted of a mixture of air (2 cfh), carbon dioxide (0.07 cfh), and sulfur dioxide (about 0.04 cfh). All gases were bubbled through water prior to entering the furnace. The results of the tests, which show the protection afforded by the C-20 coating under these conditions, are given in Table VI (Ref. 56).

TABLE VI. RESULTS OF 22-HOUR HEAT TREATMENT OF COATED AND UNCOATED HAYNES ALLOYS IN-100 AND 713 C IN A SULFUR-CONTAINING GASEOUS ATMOSPHERE (REF. 56)

Alloy	Coating	Change in Weight, mg/cm^2	Amount of Spalling
713 C	None	Specimen almost completely converted to scale	
713 C	Chromium plated, followed by C-20 coating(a)	Severe attack	Heavy
713 C	C-20 coating(a)	-10 (loss)	Very light
IN-100	None	Specimen almost completely converted to scale	
IN-100	C-20 coating(a)	+14 (gain)	Light

(a) Haynes Diffusion Coating No. C-20.

This coating was also evaluated in another type of sulfur-bearing environment, a molten bath consisting of 99.5 per cent sodium sulfate and 0.5 per cent sodium chloride at 1652 F (Ref. 54). The substrate materials were again Haynes IN-100 and Haynes 713 C, which were tested in the uncoated condition and with aluminum-iron and aluminum-nickel coatings (Haynes Diffusion Coatings No. C-9 and C-3, respectively). The changes in weight noted after a 1-hour treatment in this bath, given in Table VII, indicate the substantial improvement afforded by the C-20 coating.

TABLE VII. CHANGE IN WEIGHT OF COATED AND UNCOATED HAYNES ALLOYS IN-100 AND 713 C (REFS. 54, 58)

After 1-Hour Treatment at 1652 F in a Molten Bath of 99.5 Per Cent Sodium Sulfate, 0.5 Per Cent Sodium Chloride

	Change in Weight, mg/cm^2			
	Uncoated	Al-Ni Coating(a)	Al-Fe Coating(b)	Al-Cr Coating(c)
IN-100	-71.3	-9.5	-9.4	+0.2
713 C	+2.4(d)	--	-12.2	+0.4

(a) Haynes Diffusion Coating No. C-3, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.
(b) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.
(c) Haynes Diffusion Coating No. C-20, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.
(d) Microstructure shows heavy sulfidation attack.

Haynes Diffusion Coatings C-20 and C-9 have also been applied to chromized TD Nickel (Ref. 57). Figure 21 gives the results of cyclic-oxidation tests (24-hour cycles) to 2200 F on these materials and on uncoated TD Nickel in dry air (Ref. 57). These tests show that both coatings afford considerable protection to TD Nickel under these conditions.

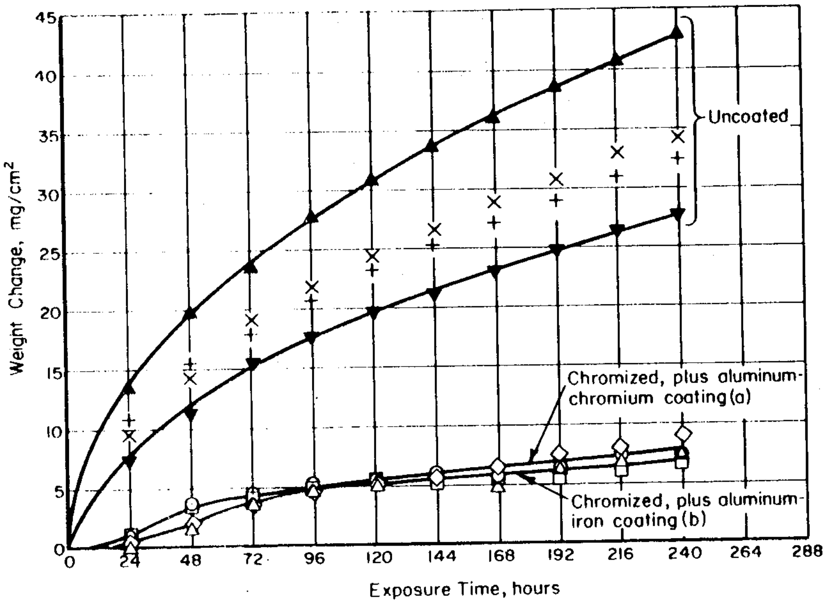

FIGURE 21. WEIGHT CHANGE VERSUS TIME FOR 2200 F CYCLIC-OXIDATION TESTS OF COATED AND UNCOATED TD NICKEL IN DRY AIR (REF. 57)

(a) Haynes Diffusion Coating No. C-20, Stellite Division, Union Carbide Corporation, Kokomo, Indiana

(b) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana

The effect of elastic and plastic strain on the oxidation protection afforded by Haynes Diffusion Coating No. C-20 in dry air has been investigated (Ref. 59). The substrate used was 0.020-inch-thick

Haynes R-41 (René 41) strip. Specimens were prepared and tested according to the procedures given in the Materials Advisory Board Report MAB-189-M. In no case was the effectiveness of the coating at 2200 or 2200 F reduced by prior deflection; the maximum deflection used was 0.015 inch in a 0.3-inch span.

A chromium-aluminum coating, known as MDC-6, was developed by Misco Precision Casting Company, Whitehall, Michigan. The coating is applied to nickel-base high-temperature alloys to provide improved resistance to sulfidation and oxidation (Ref. 24). Information was not available regarding the method by which MDC-6 is applied or the properties of nickel-base alloys with this coating.

Whitfield Laboratories, Inc., Bethel, Connecticut, produces an aluminum-chromium diffusion coating called Type II CA (the name will soon be changed to WL 1-1). Applied by a pack-cementation process, the chromium and aluminum can be deposited concurrently in the same pack or separately (the chromium being applied first) in a two-stage operation involving two packs (Ref. 38). Details of the coating process are proprietary.

Thermal-fatigue tests, performed in the manner described on page 23, have been performed on Inco 713 C with the Type II CA coating. The results of these tests, which indicate the improvement in fatigue resistance imparted by this coating, are given in Figure 12.

Lycoming Division, Avco Corporation, Stratford, Connecticut, has developed a chromium-aluminum coating for application to TD nickel. The coating, No. 703, is applied by pack cementation in vacuum (Ref. 36). Neither details of the coating process nor properties of TD nickel with this coating were available to the authors.

LTV Vought Aeronautics Division, Ling-Temco-Vought, Inc., Dallas, Texas, applies a chromium-aluminum diffusion coating by a process known as "Alchromizing" (Ref. 60). The process is a variation of pack cementation and is said to be cheaper than pack cementation when large parts are to be coated. A slip is prepared of a pack material (chromium, aluminum, inert filler material, and activator, all in powder form), using a phosphoric acid- or nitrocellulose-type carrier. The pack material is usually powdered aluminum oxide, while the activator is either anhydrous chromous chloride or potassium iodide. The slip is applied to the part to be coated either by immersion or by spraying. The slip-coated part is then placed in a retort under atmospheric conditions. A small additional amount of activator mixed with aluminum oxide powder is placed in the bottom of the retort. As the retort is heated to the temperature required to effect diffusion, the halogen evolved by the activator in the bottom of the retort purges most of the air from the retort; thus, the expense of an inert atmosphere is avoided. For nickel-base alloys the diffusion-treatment consists of heating for 2 to 6 hours at 1800 to 2000 F (Ref. 60). Typical coatings formed in this manner are 2 to 4 mils thick.

Property data for nickel-base alloys with this coating were not available to the authors.

Research done on an Air Force contract to E. I. du Pont de Nemours and Company, Inc., used two two-step pack-cementation processes to coat TD Nickel with a duplex chromium-aluminum coating in order to protect it from oxidation (Refs. 61-64). In both processes, chromium was deposited in the first step of the process, i.e., in the first pack, and aluminum was deposited in the second. One process, the details of which are proprietary, is carried out in vacuum using potassium fluoride as the activator. The other process involves the use of a 1-atmosphere pressure of argon, with ammonium chloride as the activator (Refs. 63, 64).

Specimens of TD Nickel were coated by means of the proprietary process, using temperatures and times, and achieving coating weights per unit area, as shown in Table VIII. Coated and uncoated samples were tested under conditions of cyclic oxidation; each cycle involved heating at temperature (2200 or 2400 F) for 24 hours in dried air at a flow rate of 13 cfm, cooling in still air to room temperature, and weighing along with any spalled oxide. Coating lifetime is defined as the time at which an abrupt increase occurred in the weight gain versus time curve; if no such abrupt increase is noted, lifetime was determined by the occurrence of excessive weight gain (>30 mg/cm^2) and by visual observation (Ref. 61). The results of these tests are given in Figures 22 and 23. The considerable protection afforded by these coatings, particularly those with the higher chromium and aluminum contents, is evident.

Similar tests were carried out on samples of TD Nickel with duplex chromium-aluminum coatings applied by the atmospheric pressure-ammonium chloride process. The pack materials, times, and temperatures used for the chromizing and aluminizing operations, as well as the average weight of chromium and aluminum deposited, are given in Table IX. The results of these tests were

TABLE VIII. CONDITIONS DURING COATING OF TD NICKEL CYCLIC-OXIDATION-TEST SPECIMENS (REF. 63)

Used was a Proprietary Vacuum-Pack Cementation Process Having Potassium Fluoride as the Activator

Chromizing Run	Run Conditions Time, hr	Temperature, F	Average Weight Cr or Al Deposited, g/cm^2	Chromized in Run
1	12	2250	0.0179	--
2	40	2250	0.0416	--
3	142	2250	0.0709	--
Aluminizing Run				
A	1	1400	0.0014	None
			0.0002	1
			0.0008	2
			0.0029	3
B	6	1400	0.0039	None
			0.0022	1
			0.0029	2
			0.0058	3
C	24	1400	0.0091	None
			0.0069	1
			0.0059	2
			0.0082	3

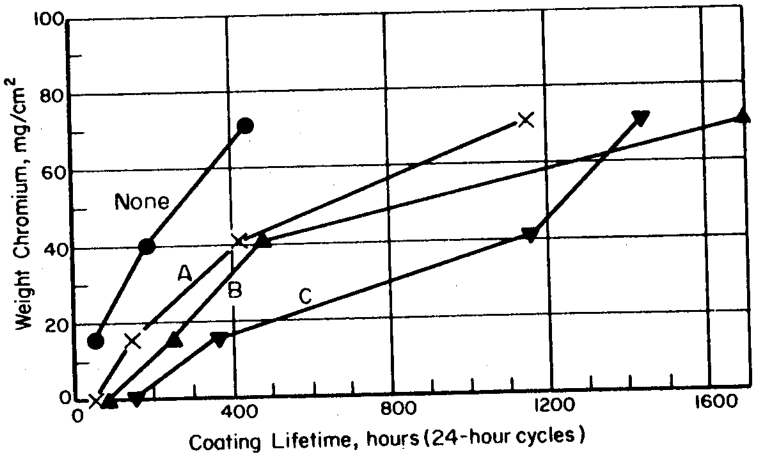

FIGURE 22. COATING LIFETIME VERSUS CHROMIUM CONTENT OF EXPERIMENTAL COATINGS ON TD NICKEL FOR 24-HOUR CYCLIC-OXIDATION TESTS AT 2200 F (REF. 63)

Letters next to curves refer to aluminizing runs described in Table VIII.

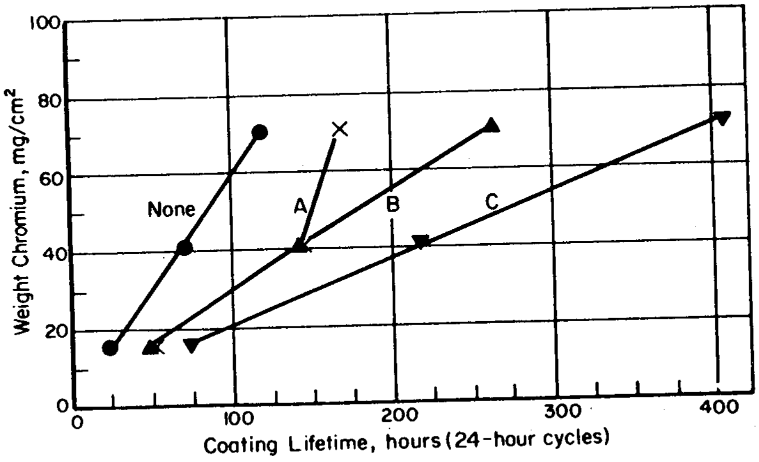

FIGURE 23. COATING LIFETIME VERSUS CHROMIUM CONTENT OF EXPERIMENTAL COATINGS ON TD NICKEL FOR 24-HOUR CYCLIC-OXIDATION TESTS AT 2400 F (REF. 63)

Letters next to curves refer to aluminizing runs described in Table VIII.

TABLE IX. PACK COMPOSITIONS AND RUN CONDITIONS FOR THE COATING OF TD NICKEL CYCLIC-OXIDATION-TEST SPECIMENS BY AN ATMOSPHERIC PRESSURE-AMMONIUM CHLORIDE-ACTIVATED PACK-CEMENTATION PROCESS (REF. 64)

Run Conditions		Average Weight Cr or Al Deposited, g/cm^2
Time, hr	Temperature, F	
	Chromizing Pack[(a)]	
24	2325	0.0432
24	2200	0.0053
24	2250	0.0249
	Aluminizing Pack[(b)]	
2.5	1800	0.0017 0.0022 0.0019
16	1800	0.0044 0.0057 0.0047
1.5	2100	0.0036 0.0062 0.0039
7.5	2100	0.0067 0.0145 0.0074

(a) 20 wt % Cr (-100 mesh); 0.5 wt % NH_4Cl; balance Al_2O_3 (-100 mesh).

(b) 20 wt % Cr (75 wt % Cr-25 Wt % Al) (-100 mesh); 0.1 wt % NH_4Cl; balance Al_2O_3 (-100 mesh).

found to be similar to those obtained for the coatings applied by the proprietary process.

Sheet specimens of TD Nickel were coated by the atmospheric pressure-ammonium chloride method according to the process described in Table X. Stress-rupture tests were performed on coated and uncoated specimens at 2000 and 2200 F, and tensile tests were performed at room temperature and 2000 F. The results of the stress-rupture tests are given in Tables XI and XII, and the tensile data are presented in Table XIII. It may be seen that, in general, there is little difference between the test results for coated and uncoated specimens; thus, the coating process does not lead to a degradation of mechanical properties.

In the same Air Force program (Refs. 61-64) Du Pont has codeposited chromium-aluminum coatings in a one-step process; the chromium and aluminum were deposited simultaneously in a single pack. It was found that by performing the codeposition under the conditions given in Table XIV coatings could be obtained whose behavior in cyclic-oxidation tests closely approximated that exhibited by a duplex chromium-aluminum coating; the duplex coating was deposited in a two-step process, using the conditions given in Table XV. The results demonstrate the feasibility of depositing such coatings in one-step processes, with the attendant economies.

In a subcontract from Du Pont on Contract No. AF 33(615)-1704, TRW Inc. is studying the application of a chromium-aluminum coating on TD Nickel by two-step pack cementation in vacuum. The variables being considered in studying the two steps (chromizing and aluminizing) in the process are as follows (Ref. 65):

(1) Pack composition (metal-refractory ratio)

(2) Pack-material particle size

(3) Time

(4) Temperature

(5) System pressure

(6) Type of activator

(7) Quantity of activator.

TABLE X. PROCESS DATA FOR THE COATING OF SHEET SPECIMENS OF TD NICKEL[a] (FOR MECHANICAL TESTING) BY AN ATMOSPHERIC PRESSURE-AMMONIUM CHLORIDE-ACTIVATED PACK-CEMENTATION PROCESS (REF. 64)

Chromizing Run	Run Conditions Time, hr	 Temperature, F	Average Weight Cr or Al Deposited, g/cm^2	Materials Coated
				Chromizing Pack[b]
C-108-A	30	2300	0.0306	Stress-rupture specimens, TD Nickel 978, transverse (0.090-in. sheet)
C-109-A	30	2300	0.0305	Stress-rupture specimens, TD Nickel 978, longitudinal (0.090-in. sheet)
C-110-A	30	2300	0.0323	Stress-rupture specimens, TD Nickel 1108, transverse (0.050-in. sheet)
			0.0326	Stress-rupture specimens, TD Nickel 1108, longitudinal (0.050-in. sheet)
			0.0331	Stress-rupture specimens, TD Nickel 918, transverse (0.030-in. sheet)
			0.0330	Stress-rupture specimens, TD Nickel 918, longitudinal (0.030-in. sheet)
			0.0330	Sheet tensile specimens, TD Nickel 1108, transverse (0.050-in. sheet)
			0.0327	Sheet tensile specimens, TD Nickel 1108, longitudinal (0.050-in. sheet)
				Aluminizing Pack[c]
Aluminizing Run				
D-115-A	3	1800	0.0027	Stress-rupture specimens, TD Nickel 978, transverse (0.090-in. chromized sheet)
D-122-A	3	1800	0.0027	Stress-rupture specimens, TD Nickel 978, longitudinal (0.090-in. chromized sheet)
D-123-A	3	1800	0.0032	Stress-rupture specimens, TD Nickel 1108, transverse (0.050-in. chromized sheet)
D-124-A	3	1800	0.0033	Stress-rupture specimens, TD Nickel 1108, longitudinal (0.050-in. chromized sheet)
			0.0034	Stress-rupture specimens, TD Nickel 918, transverse (0.030-in. chromized sheet)
			0.0035	Stress-rupture specimens, TD Nickel 918, longitudinal (0.030-in. chromized sheet)
			0.0033	Sheet tensile specimens, TD Nickel 1108, transverse (0.050-in. chromized sheet)
			0.0033	Sheet tensile specimens, TD Nickel 1108, longitudinal (0.050-in. chromized sheet)

(a) Referred to by Lot No.

(b) 20 wt % Cr (-100 mesh); 0.5 wt % NH_4Cl; balance, Al_2O_3 (-100 mesh).

(c) 20 wt % alloy (75 wt % Cr-25 wt % Al); 0.1 wt % NH_4Cl; balance, Al_2O_3 (-100 mesh).

TABLE XI. STRESS-RUPTURE DATA FOR TD NICKEL SHEET TESTED AT 2000 F (REF. 64)

Uncoated and With a Duplex Chromium-Aluminum Coating

			Uncoated			Duplex Cr-Al Coated (See Table X)		
TD Nickel Lot No.	Uncoated Thickness, in.	Section	Stress, psi	Rupture Life, hr	Elongation, per cent	Stress, psi	Rupture Life, hr	Elongation, per cent
978	0.090	Longitudinal	11,000	10.1	3	11,000	23.1	7
978	0.090	Longitudinal	11,000	10.9	6	11,000	22.7	5
978	0.090	Transverse	9,500	14.8	2	9,500	4.4	4
978	0.090	Transverse	9,500	24.0	4	9,500	41.2	3
1108	0.050	Longitudinal	13,000	61.2	5	13,000	13.9	8
1108	0.050	Longitudinal	13,000	16.8	9	13,000	18.7	6
1108	0.050	Transverse	9,000	12.5	5	9,000	7.1	3
1108	0.050	Transverse	9,000	13.0	3	9,680	13.9	3
918	0.030	Longitudinal	11,000	10.8	11	11,000	10.7	12
918	0.030	Longitudinal	11,000	3.1	10	11,000	13.7	8
918	0.030	Transverse	6,500	6.7	3	6,500	5.7	4
918	0.030	Transverse	6,500	7.1	4	6,500	6.7	3

TABLE XII. STRESS-RUPTURE DATA FOR TD NICKEL SHEET TESTED AT 2200 F (REF. 64)

Uncoated and With a Duplex Chromium-Aluminum Coating

TD Nickel Lot No.	Uncoated Thickness, in.	Section	Uncoated			Duplex Cr-Al Coated (See Table X)		
			Stress, psi	Rupture Life, hr	Elongation, per cent	Stress, psi	Rupture Life, hr	Elongation, per cent
978	0.090	Longitudinal	8,000	3.9	3	8,000	8.5	5
978	0.090	Longitudinal	8,000	3.7	4	8,000	8.7	5
978	0.090	Transverse	7,500	4.0	3	7,500	9.5	3
978	0.090	Transverse	--	--	--	7,500	9.9	3
1108	0.050	Longitudinal	8,500	81.8	3	8,500	119.8	4
1108	0.050	Longitudinal	8,500	146.0	6	8,500	54.0	5
1108	0.050	Transverse	7,000	11.2	2	7,000	4.1	4
1108	0.050	Transverse	--	--	--	7,000	2.2	4
918	0.030	Longitudinal	8,000	16.6	5	8,000	9.2	9
918	0.030	Longitudinal	8,000	13.0	7	9,060	1.6	12
918	0.030	Transverse	5,000	3.6	4	5,000	1.7	5
918	0.030	Transverse	5,000	6.9	3	5,000	2.3	5

TABLE XIII. TENSILE-TEST DATA ON TD NICKEL SHEET TESTED AT ROOM TEMPERATURE AND AT 2000 F (REF. 64)

Bare and With a Duplex Chromium-Aluminum Coating

TD Nickel Lot No.	Uncoated Thickness, in.	Section	Uncoated Yield Strength, ksi	Uncoated Ultimate Tensile Strength, ksi	Uncoated Elongation, per cent	Duplex Cr-Al Coated (See Table X) Yield Strength, ksi	Duplex Cr-Al Coated (See Table X) Ultimate Tensile Strength, ksi	Duplex Cr-Al Coated (See Table X) Elongation, per cent
				Room Temperature				
1108	0.050	Longitudinal	54.0	68.5	16.1	66.1	84.7	16.9
1108	0.050	Longitudinal	53.3	67.7	15.4	63.5	84.1	13.8
1108	0.050	Transverse	56.3	67.5	16.9	65.9	84.9	13.8
1108	0.050	Transverse	57.9	67.9	19.2	68.0	86.9	13.8
				2000 F				
1108	0.050	Longitudinal	18.2	18.4	7.7	16.8	17.9	15.4
1108	0.050	Longitudinal	18.5	18.8	6.1	17.3	18.1	12.3
1108	0.050	Transverse	16.9	17.0	5.4	17.3	17.9	6.1
1108	0.050	Transverse	16.5	17.0	4.6	15.7	17.1	4.6

TABLE XIV. PROCESSING CONDITIONS AND WEIGHT OF DEPOSIT FOR CHROMIUM-ALUMINUM COATING BY CODEPOSITION IN A ONE-STEP PACK-CEMENTATION PROCESS (REF. 64)

Pack Composition	Run Conditions Time, hr	 Temperature, F	Weight of Deposit, g/cm^2
50 wt % Alloy (97 wt % Cr-3 wt % Al), -100 mesh; 0.5 wt % NH_4Cl; balance Al_2O_3, -100 mesh	28	2300	0.0449

TABLE XV. PROCESSING CONDITIONS AND WEIGHT OF DEPOSIT FOR DUPLEX CHROMIUM-ALUMINUM COATING BY A TWO-STEP PACK-CEMENTATION PROCESS (REF. 64)

Pack Composition	Run Conditions Time, hr	 Temperature, F	Weight of Deposit, g/cm^2
Chromium-deposition step			
20 wt % Cr, -100 mesh; 0.5 wt % NH_4Cl; balance Al_2O_3, -100 mesh	~30	2300	0.0354
Aluminum-deposition step			
20 wt % alloy (75 wt % Cr-25 wt % Al); 0.1 wt % NH_4Cl; balance Al_2O_3, -100 mesh	3	1800	0.0025

Most of the chromizing work has involved the use of a pack containing chromium (90 volume per cent) and aluminum oxide (10 volume per cent) powders of -8, +30 mesh, and 0.5 weight per cent of an activator; deposition times used were generally 16 hours at 2200 F, with a system pressure of 10^{-2} torr (mm Hg). At temperatures above 2200 F, some sintering of the particles of the pack to the TD Nickel occurred.

The second step of the process, aluminizing in a vacuum pack, is being accomplished using a pack consisting of -8, +30 mesh particles of a 56 per cent chromium-44 per cent aluminum alloy and usually 0.8 weight per cent potassium fluoride as an activator; no inert refractory material is used. The process is carried out at a pressure below 10^{-2} torr. Deposition times are from 3 to 4-1/2 hours at temperatures between 1400 and 1700 F (Ref. 65).

Additional experiments are being performed to determine the optimum parameters for both the chromizing and the aluminizing steps (Ref. 65).

On the same subcontract from Du Pont (Ref. 65) TRW Inc. is also investigating the application of chromium-aluminum coatings onto TD Nickel by spraying with a slurry containing the pack-metal constituents, a binder, a halide activator, and a volatile spraying vehicle. Diffusion is accomplished in a vacuum furnace. Published results are preliminary at this time.

Aluminum-Chromium-Nickel. TRW Inc., Cleveland, Ohio, has developed a chromium-aluminum-nickel diffusion coating for TD nickel (Ref. 66). The coating is applied by a proprietary pack-cementation process in vacuum. No property data on nickel or nickel-base alloys with this coating were available to the authors.

Aluminum-Chromium-Silicon. WL-4, an aluminum-chromium-silicon coating developed by Whitfield Laboratories, Inc., Bethel, Connecticut, is applied by a pack-cementation technique (Ref. 38). Details of the coating process are proprietary.

René 41, uncoated and with the WL-4 coating, has been tested in tension at room temperature and at 1800 F after exposure to air for 1000 hours at 1800 F. Similar specimens have been tested in tension at room temperature and at 1900 F after exposure to air for 1000 hours at 1900 F. The WL-4 coating was found to limit the loss of ultimate strength as a result of exposure at both temperatures (Ref. 38).

The SAC coating of Chromalloy Division, Chromalloy Corporation, West Nyack, New York, also falls into the aluminum-chromium-silicon category (Ref. 50). The coating is applied by a proprietary pack-cementation technique (Ref. 49).

Results of impact resistance, microhardness, and stamping tests on B-1900 coated with SAC are given in Figures 18, 19, and 20. Other test data on nickel-base-alloy substrates with an SAC coating were not available to the authors.

Lycoming Division, Avco Corporation, Stratford, Connecticut, uses a different method to apply an aluminum-chromium-silicon coating (its No. 606 coating) to nickel-base alloys (Ref. 36). A chromium coating is first electroplated onto the nickel-base-alloy substrate. Next, the part is hot dipped in a molten bath of an aluminum-silicon alloy. After removal from the bath, the duplex coating is given a diffusion heat treatment in argon to form the desired coating (Ref. 36).

Lycoming's 606 coating is said to increase the resistance of nickel-base alloys to sulfidation, and to double the rupture life (stress not stated) of Inco 713 C at 1700 F (Ref. 36). No further properties of nickel-base alloys with this coating were available to the authors.

Aluminum-Cobalt. An experimental cobalt-aluminum coating, applied by a spray-and-diffuse technique, is being used on MAR-M200 and Udimet 700 in an Air Force-sponsored program to develop coated superalloy components for gas turbine-engine applications (Refs. 29, 67-69). The alloy substrates are conditioned by blasting with No. 60 aluminum oxide grit at 40 psi for 2 to 5 minutes, followed by degreasing with trichlorethylene at 180 F for 5 to 10 minutes. The slurry is prepared in batches, using the following materials (Ref. 69):

50 grams of analytical-grade cobalt powder
(Fisher Scientific Company)

200 grams of flake aluminum powder
(Metals Disintegrating Company)

680 mils of Pratt and Lambert 2012 nitrocellulose lacquer.

Thus, the metallic content of the slurry is 90 atomic per cent aluminum and 10 atomic per cent cobalt. The ingredients are mixed by ball milling in a 1-gallon porcelain crock with 1-inch-diameter porcelain balls for 8 to 16 hours. The desired viscosity is 600 to 1000 cps at 65 to 85 F, as measured by a Brookfield Viscometer Model RVT at 10 rpm and spindle number 1 (Ref. 69).

The substrate is coated with the slurry by spraying, dried in air for at least 4 hours, and given a diffusion heat treatment for 4 hours in hydrogen. For MAR-M200 substrates the temperature of the diffusion treatment is 2200 F, for Udimet 700 substrates it is 2000 F (Ref. 69).

A variation of this coating is also being used in this research (Refs. 29, 67). It is a tantalum plus cobalt-aluminum coating and involves the deposition of a coating of tantalum on the substrate prior to the application of the cobalt-aluminum coating. The tantalum, which presumably serves as a barrier to diffusion and thereby tends to stabilize the coating, was applied by Office National D'Etudes et de Recherches Aerostatials (ONERA) of Paris, France (Refs. 67, 70). Details of the coating process are proprietary (Ref. 23). The tantalum-coated substrate is given a diffusion treatment for 4 hours at 2000 F in vacuum. The cobalt-aluminum coating is applied in the manner noted in the previous paragraphs, with the exception that the diffusion treatment for the cobalt-aluminum is carried out in vacuum rather than in air (Ref. 67).

At the time of the last (fourth) quarterly progress report (Ref. 29) on this Air Force program, oxidation-erosion, sulfidation, thermal fatigue, stress-rupture, and ballistic-impact tests were being performed on MAR-M200 substrates coated with tantalum plus cobalt-aluminum (Ref. 29). The final report on that program, which should include the results of these tests, has not yet been issued.

<u>Aluminum-Iron</u>. One type of diffused aluminum-iron coating is Solar Aluminizing S13-53C (Refs. 71, 72). The coating is applied as a proprietary slurry by spraying or dipping to a thickness of about 0.006 inch. After the slurry is dried, the part is fired at 1850 to 2050 F in an inert atmosphere to effect diffusion of the aluminum into the substrate (Ref. 71). The thickness of the resultant diffused aluminum-iron coating is 0.001 to 0.0015 inch (Ref. 71); its hardness is 50 to 70 R_C (Ref. 72). Coated parts can be water quenched from 1500 F or cyclically heated to 2300 F and cooled to ambient temperature (rates of heating and cooling not given) without

damaging the coating (Ref. 72). The coating is said to have no adverse effect on fatigue strength (Ref. 72). The resistance of the coating to impact has been termed "excellent" (Ref. 72).

No specific data regarding the properties of parts coated with Solar Aluminizing S13-53C was available to the authors (Ref. 71).

Another aluminum-iron diffusion coating for nickel-base alloys, known as Haynes Diffusion Coating No. C-9, was developed by the Stellite Division, Union Carbide Corporation, Kokomo, Indiana. It is applied by a pack-cementation technique to nickel-base-alloy substrates that have been previously buffed and vapor degreased (Ref. 73). Coating thickness is generally about 0.001 inch (Ref. 74). Details regarding the coating process are proprietary.

This coating has been found to protect TD nickel from oxidation during cyclic testing in air to 2200 F (Figure 21) (Ref. 57). It also has protected IN-100 and Inco 713 C from corrosion in a molten bath of sodium sulfate (99.5 per cent) and sodium chloride (0.5 per cent) at 1652 F (Ref. 54), although the degree of protection afforded these substrates was not as great as when Haynes Diffusion Coating C-20 (aluminum-chromium) was used (Table VII).

The results of oxidation tests at 1900 F in dry air on Hastelloy 500 (Udimet 500), uncoated and with this coating and an aluminum-nickel coating, are given in Table XVI. The data are indications of the protection offered by the coatings.

Thermal-shock tests have been performed on airfoil-shaped specimens of Haynes IN-100 (Refs. 58, 74) and Hastelloy 500 (Udimet 500) (Ref. 58), both uncoated and coated with Haynes Diffusion Coating No. C-9. The test involved spinning the specimens in groups of eight mounted on a hub at 1725 rpm under conditions of cyclic heating and cooling. Each cycle consists of 60 seconds' exposure in a furnace and 90 seconds' cooling in a water spray; specimens were examined after each 100 cycles to a maximum of 500 cycles. The test results indicate that the C-9 coating increases resistance to thermal shock from 1850 F under these conditions, as shown in Table XVII.

Stress-rupture data for as-cast Haynes 713 C, Hastelloy 500, and Haynes IN-100 with this aluminum-iron coating and in the uncoated condition are given in Table XVIII (Ref. 58). The data indicate that the coating sometimes increases stress-rupture life and/or

TABLE XVI. WEIGHT CHANGE VERSUS TIME FOR HASTELLOY 500 DURING OXIDATION TESTING IN DRY AIR AT 1900 F (REF. 58)

Uncoated and With Aluminum-Iron and Aluminum-Nickel Coatings

Time at 1900 F, hr	Weight Change, mg/cm³		
	Uncoated	Aluminum-Iron Coating(a)	Aluminum-Nickel Coating(b)
23	-1.7	+0.2	+0.4
46	-3.8	+0.4	+0.8
69	-5.1	+0.4	+0.6
92	-2.8	+0.4	+0.9

(a) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

(b) Haynes Diffusion Coating No. C-3, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

TABLE XVII. DATA FROM THERMAL-SHOCK TESTS (FROM 1850 F) ON HAYNES IN-100 AND HASTELLOY 500 (REF. 58)

Uncoated and With an Aluminum-Iron Diffusion Coating(a)

Alloy	Cycles From 1850 F After Which First Crack Was Found	
	Uncoated	With Aluminum-Iron Diffusion Coating
Haynes IN-100	450(b)	No crack observed at end of 500-hr test(b)
Hastelloy 500	150(b)	400 (one test) No crack observed at end of 500-hr test (one test)

(a) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

(b) Average of two tests.

TABLE XVIII. STRESS-RUPTURE DATA FOR THREE NICKEL-BASE ALLOYS IN THE AS-CAST CONDITION AND WITH AN ALUMINUM-IRON COATING(a) (REF. 58)

Alloy	Condition	Test Temperature, F	Stress, psi	Life, hr	Elongation, per cent
Haynes 713 C	Uncoated	1600	45,000	50.5	5.2
	Coated	1600	45,000	75.3	5.6
	Uncoated	1650	38,000	54.3	5.5
	Coated	1650	38,000	57.0	7.9
	Uncoated	1700	30,000	74.2	6.7
	Coated	1700	30,000	89.6	7.8
	Uncoated	1750	25,000	60.2	9.0
	Coated	1750	25,000	55.9	10.0
Haynes IN-100	Uncoated	1700	34,000	68.4	5.2
	Coated	1700	34,000	53.7	5.8
	Uncoated	1800	22,000	122.2	8.4
	Coated	1800	22,000	128.9	6.2
	Uncoated	1850	17,000	223.9	6.5
	Coated	1850	17,000	170.0	10.0
	Uncoated	1900	13,000	180.3	5.1
	Coated	1900	13,000	210.1	9.0
Hastelloy 500	Uncoated	1600	30,000	279.4	15.0
	Coated	1600	30,000	377.5	20.0
	Uncoated	1650	25,000	294.8	21.0
	Coated	1650	25,000	216.1	19.0
	Uncoated	1700	22,000	151.4	22.5
	Coated	1700	22,000	170.0	20.5
	Uncoated	1800	10,000	334.2	31.0
	Coated	1800	10,000	426.9	25.5

(a) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

ductility and sometimes decreases either or both properties (Ref. 58). The effect may be a true function of temperature, but additional tests would be required to determine this.

The use of this coating has been found to increase the life of Hastelloy 500 and Haynes IN-100 in rotating-beam fatigue at room temperature, as shown in Table XIX (Ref. 58).

TABLE XIX. RESULTS OF ROTATING-BEAM-FATIGUE TESTS ON HASTELLOY 500 AND HAYNES IN-100 AT ROOM TEMPERATURE (REF. 58)

Uncoated and With an Aluminum-Iron Diffusion Coating(a)

Alloy	Stress, psi	Cycles to Failure	
		Uncoated	Coated
Hastelloy 500	25,000	26,047,000	31,536,000(b)
Haynes IN-100	25,000	19,208,000	31,782,000(c)

(a) Haynes Diffusion Coating No. C-9, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.
(b) Average of four tests.
(c) Average of five tests.

Aluminum-Nickel. The aluminum-nickel coating for nickel-base alloys developed by the Stellite Division of Union Carbide Corporation is designated Haynes Diffusion Coating No. C-3. It is applied by a pack-cementation process; details of the process are proprietary.

The protective effect of this coating on Hastelloy 500 (Udimet 500) during oxidation testing at 1900 F in dry air may be seen from the data presented in Table XVI (Ref. 58), which appears on page 56.

The effect of this coating in decreasing the weight loss of IN-100 during treatment for 1 hour in a molten bath of 99.5 per cent sodium sulfate and 0.5 per cent sodium chloride at 1652 F is shown in Table VII. This table appears on page 40.

Haynes 713 C (Inco 713 C) with this aluminum-nickel coating has been tested in tension at a number of temperatures to 1800 F. Data from these tests and from tests on the uncoated alloy are given in Table XX (Ref. 58); at some elevated temperatures, a small decrease in strength and ductility were noted.

TABLE XX. SHORT-TIME TENSILE PROPERTIES OF HAYNES 713 C (REF. 58)

Uncoated and With an Aluminum-Nickel Diffusion Coating(a)

Condition	Temperature, F	Ultimate Tensile Strength, psi	Yield Strength at 0.2% Offset, psi	Elongation, per cent	Reduction of Area, per cent
Uncoated	RT	121,300	107,500	5.9	8.2
Coated	RT	123,000	100,700	8.8	9.6
Uncoated	1200	122,800	101,200	8.0	16.0
Coated	1200	134,000	102,800	7.3	9.8
Uncoated	1400	136,200	104,500	6.7	15.0
Coated	1400	123,400	101,100	6.2	6.5
Uncoated	1600	106,300	83,100	9.7	17.4
Coated	1600	108,000	66,700	10.6	12.5
Uncoated	1700	90,000	--	8.5	14.7
Coated	1700	87,300	52,600	20.8	25.6
Uncoated	1800	72,000	51,300	14.0	28.0
Coated	1800	64,300	36,000	28.6	30.2

(a) Haynes Diffusion Coating No. C-3, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

The use of this coating has been found to increase the life of Hastelloy R-235 in rotating-beam fatigue at room temperature, as shown in Table XXI (Ref. 58).

WL-11, an aluminum-nickel coating developed by Whitfield Laboratories, Inc., Bethel, Connecticut, is usually applied by a slurry spray-and-diffuse technique (Ref. 38). Details regarding the coating process are proprietary.

Astroloy, uncoated and with the WL-11 coating, has been tested in tension at room temperature and at 1800 F after exposure to air for 1000 hours at 1800 F. The WL-11 coating was found to limit significantly the loss of ultimate strength as a result of the exposure at 1800 F (Ref. 38).

TABLE XXI. RESULTS OF ROTATING-BEAM-FATIGUE TESTS ON HASTELLOY R-235 AT ROOM TEMPERATURE (REF. 58)

Uncoated and With an Aluminum-Nickel Diffusion Coating(a)

	Cycles to Failure	
Stress, psi	Uncoated	Coated
40,000	1,460,000	2,735,000
35,000	3,220,000	4,638,000
30,000	27,217,000	88,426,000(b)

(a) Haynes Diffusion Coating No. C-3, Stellite Division, Union Carbide Corporation, Kokomo, Indiana.

(b) Not a failure - test discontinued.

René 41, uncoated and with the WL-11 coating, has been tested in tension at room temperature and at 1900 F after exposure to air for 1000 hours at 1900 F. The ultimate strength of the coated material was found to be about 50 per cent greater than that of the uncoated material at both test temperatures (Ref. 38).

Aluminum-nickel coatings can also be applied by a special electroplating process, as noted on pages 84-85.

Aluminum-Silicon. An aluminum-silicon coating developed by Pratt & Whitney Aircraft Division of United Aircraft Corporation and known as PWA 47 is applied to nickel-base alloys by a spray-and-diffuse technique (Ref. 27). The substrate is prepared by blasting with aluminum oxide grit and cleaning with a solvent. The slurry, which is applied with a spray gun, consists of aluminum (90 weight per cent) and silicon (10 weight per cent) powders in a nitrocellulose lacquer (Refs. 27, 70); the final diffused coating, however, contains little silicon (Ref. 70). The diffusion treatment is carried out at the usual solution-treatment temperature for the alloy; for Waspaloy, the diffusion time is 2 to 4 hours at a temperature of 1975 F (Ref. 27). Nickel-base alloys are cooled in air following the diffusion treatment.

This coating has been found to improve the bow (bending) and thermal-fatigue properties of IN-100, MAR-M200, and B-1900 in an atmosphere simulating that within a gas-turbine engine (Ref. 75). Miniature airfoil-shaped test specimens consisting of an airfoil section about 2 inches long and 1 inch wide attached to a 1-inch-long grip section were used. A weight of sufficient size to give a

5,000 psi simulated vane-bow stress at 1850 rpm was welded to the tip of the airfoil section. The test procedure involved first spinning these specimens in the equipment shown in Figures 24 and 25 at 1850 rpm for 12 hours in the products of combustion of JP-5 fuel and air. This atmosphere was chosen to simulate the corrosive-erosive environment of the hot high-velocity gas stream within the turbine section of an engine. At the conclusion of the 12-hour test the extent of bow was measured. The samples were then thermal cycled at 1850 rpm in the same test rig and combustion-product environment; each cycle involved 15 seconds at 2100 F and 30 seconds cooling in a 45 psi blast of air. The specimens were examined periodically for thermal-fatigue cracks, which tended to occur in the critically stressed area of the trailing edge. As shown by the data in Table XXII this coating has been found to decrease the amount of bow and increase the number of cycles required to initiate the first thermal-fatigue crack in IN-100, MAR-M200, and B-1900 (Ref. 75). The data from Column (3) of Table XXII are compared graphically in Figure 26; the protective effect of the aluminum-silicon coating is obvious. This figure also contains data for the $TaAl_3$ coating discussed on page 69.

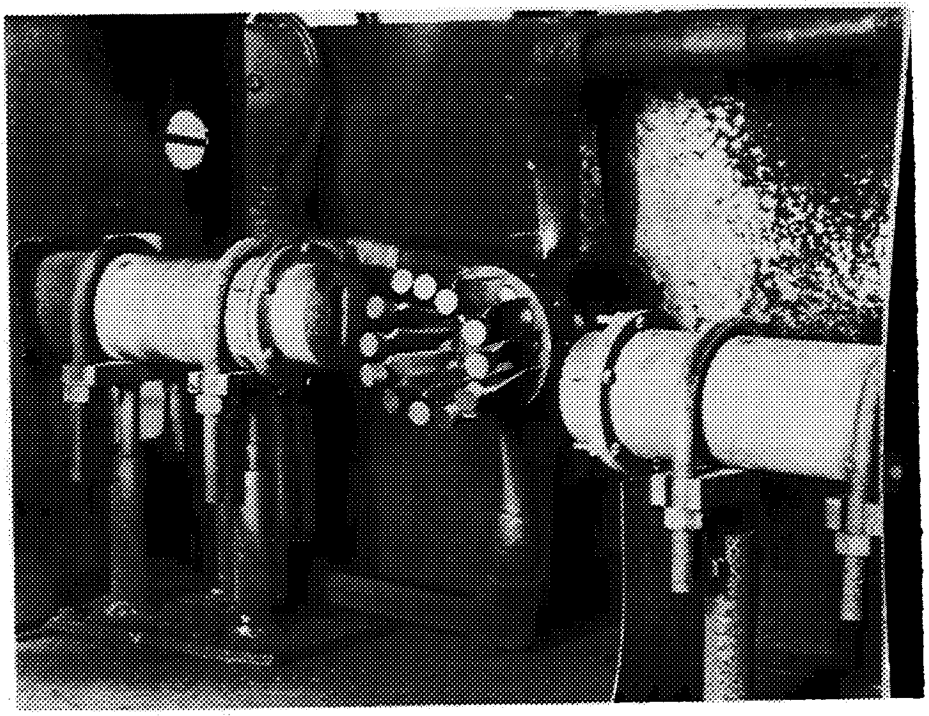

FIGURE 24. THERMAL-FATIGUE BOW RIG SHOWING POSITION OF SPECIMENS IN RELATION TO BURNER NOZZLES (REF. 75)

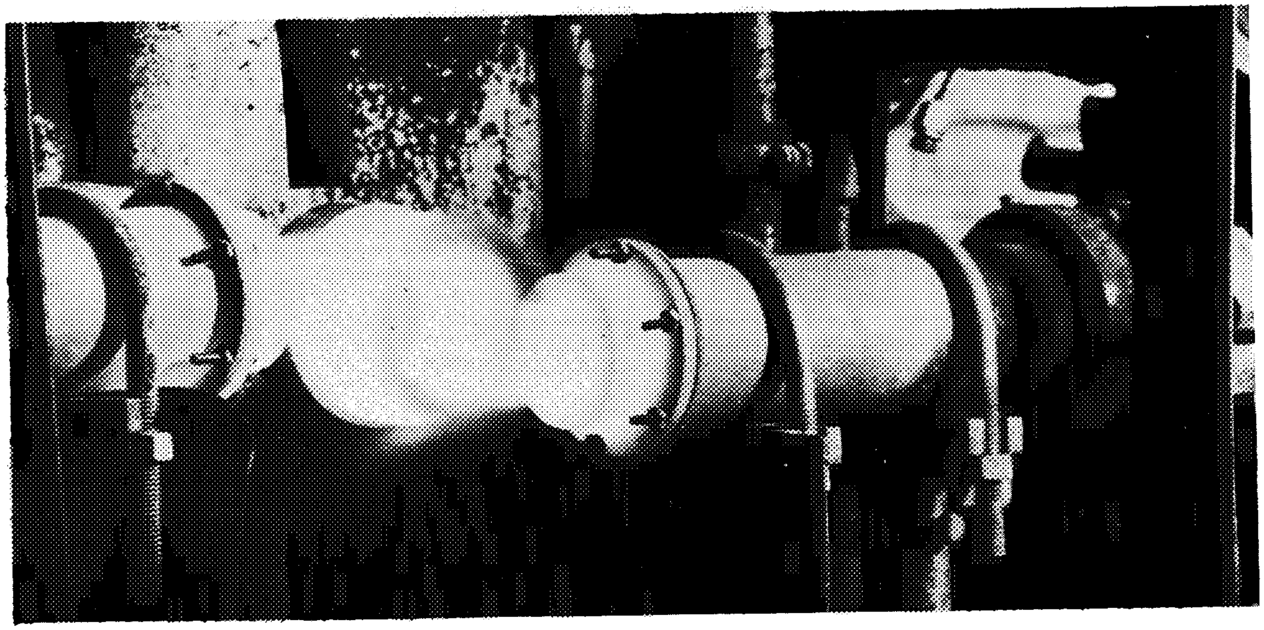

FIGURE 25. THERMAL-FATIGUE BOW RIG IN OPERATION (REF. 75)

TABLE XXII. TRAILING EDGE BOW AND THERMAL-FATIGUE DATA FOR IN-100, MAR-M200, AND B-1900 (REF. 75)

Alloy	Coating	Trailing Edge Bow(a), in.	Trailing Edge Bow in Thermal-Fatigue Bow Test(b), in.	Cycles to Crack in Thermal-Fatigue Bow Test(c)
IN-100	None	0.004-0.013	0.004-0.014	500-800
IN-100	Aluminum-silicon	0.003-0.005	0.004-0.006	200-900
MAR-M200	None	0.003-0.007	0.003-0.010	100-400
MAR-M200	Aluminum-silicon	0.001-0.002	0.002-0.006	300-1200
B-1900	None	0.002-0.010	0.004-0.015	100-1000
B-1900	Aluminum-silicon	0.002-0.003	0.003-0.004	800-1700

(a) 12 hours at 1950 F and 5,000 psi.

(b) 12 hours at 1950 F and 5,000 psi plus 400 thermal cycles (2100 F for 15 seconds hot, 30 seconds cooling).

(c) 12 hours at 1950 F and 5,000 psi prior to 2100 F thermal cycling.

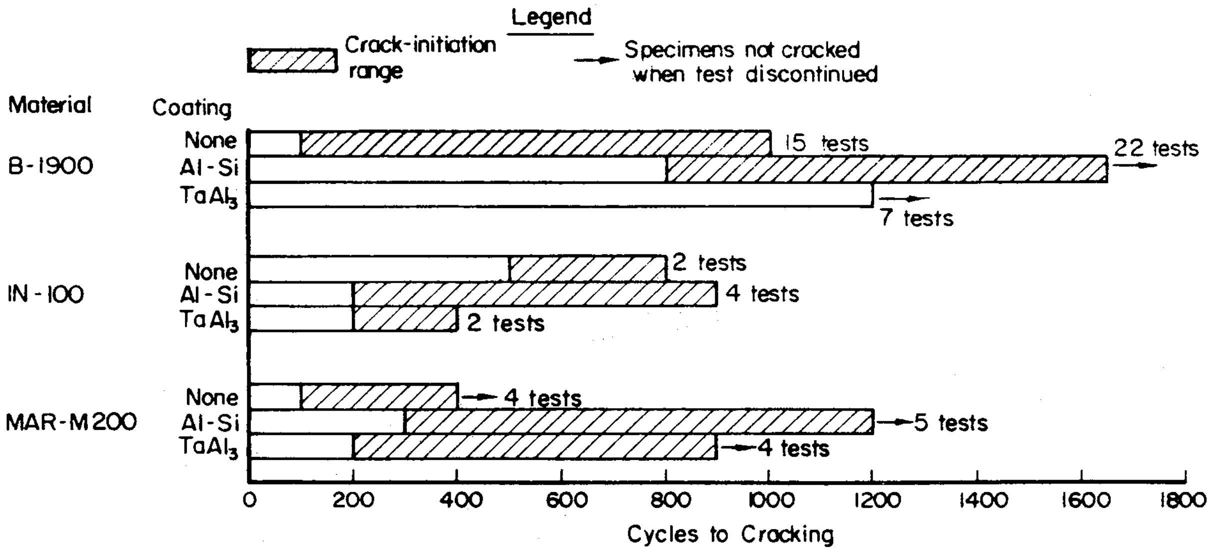

FIGURE 26. ONSET OF THERMAL-FATIGUE CRACKING FOR SPECIMENS OF B-1900, IN-100, AND MAR-M200 (REF. 75)

Specimens of B-1900 protected with this coating have also been tested in thermal fatigue by a method in which a disk-type specimen is immersed for 2 minutes in a fluidized bed at an elevated temperature and then quenched by immersing for 2 minutes in a fluidized bed held at 68 F. Transfer time between beds was about 2 seconds in each direction. The combined effect of this coating and a 1650 F/10-hour heat treatment in increasing the number of cycles required before the first crack appeared is shown in Figure 27 (Ref. 75).

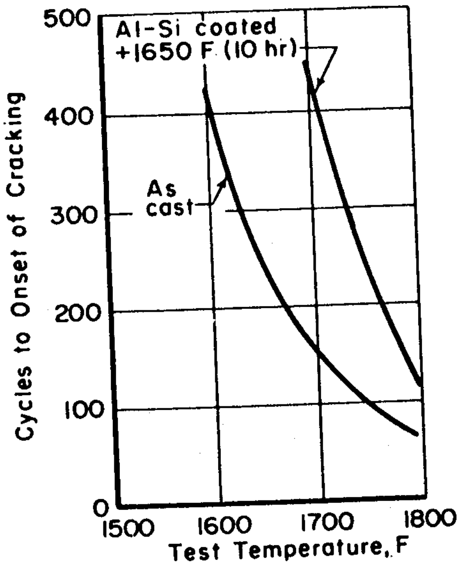

FIGURE 27. CYCLES TO ONSET OF CRACKING VERSUS TEST TEMPERATURE FOR COATED AND UNCOATED B-1900 (REF. 75)

The PWA 47 coating and Martin Metals A-11 diffused aluminum coating have been applied to MAR-M200 and Udimet 700 in a program sponsored by the U. S. Air Force to develop coated superalloy components for gas-turbine-engine applications (Refs. 29, 67-69). The stress-rupture properties of the coated alloys, as determined in the Air Force program, are given in Figures 28 and 29 (Ref. 29). Included for comparison are the stress-rupture curves and elongations at fracture for average commercial lots of the two alloys. The decrease in stress-rupture strength and elongation found for both coatings under certain conditions is not unusual for diffusion coatings.

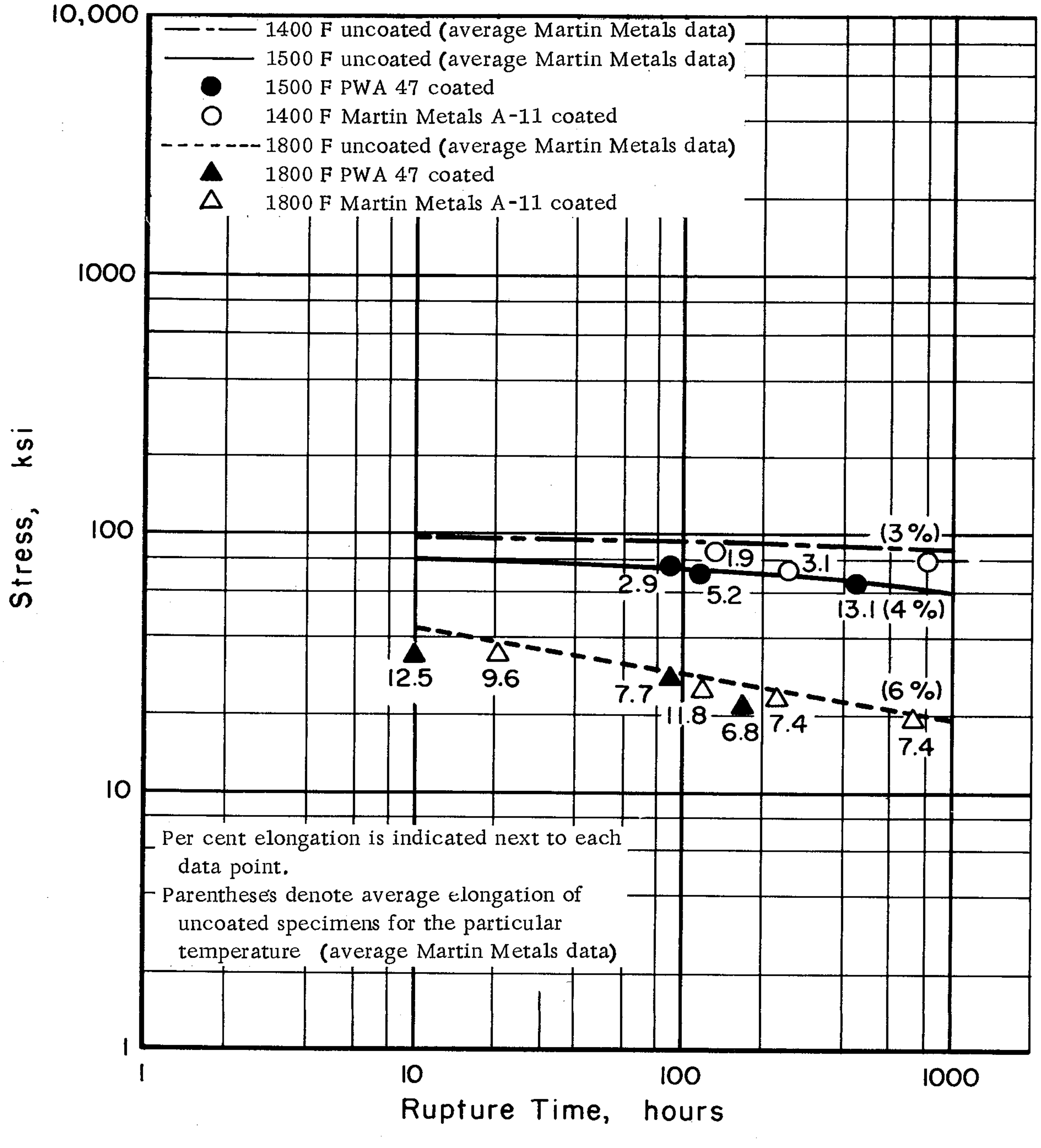

FIGURE 28. STRESS-RUPTURE PROPERTIES OF COATED AND UNCOATED MAR-M200 (REF. 29)

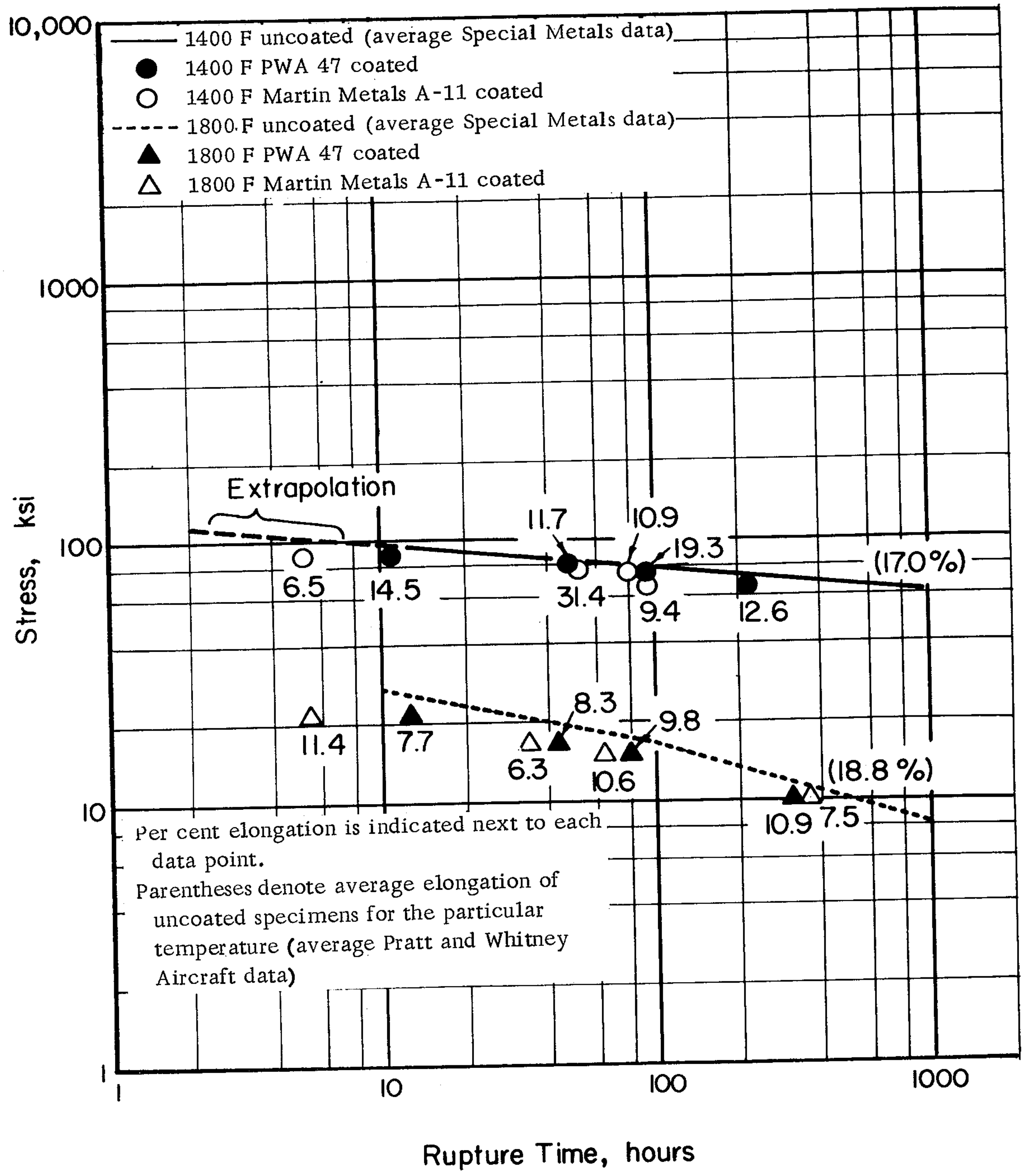

FIGURE 29. STRESS-RUPTURE PROPERTIES OF COATED AND UNCOATED UDIMET 700 (REF. 29)

Ballistic-impact tests were also performed on the coated alloys (Ref. 29). The test procedure involved the impingement of a steel-tipped lead pellet (total weight 0.7 gram) shot from a Crossman air rifle at a muzzle velocity of 170 feet per second on the coated surface. The rifle-to-target distance was not given. Tests were run at room temperature and at 1700 F; the latter temperature was achieved by heating the specimen with an oxyacetylene torch, and was measured by means of an optical pyrometer. The samples were tested in both the as-coated condition and after oxidation-erosion testing at 2000 F for 100 hours in the products of combustion of JP-5 fuel and air (Ref. 29); the equipment used for the oxidation-erosion tests is shown in Figure 24. The samples were inspected by a post-emulsion fluorescent-penetrant method after ballistic testing; results are given in Table XXIII. The data are self-explanatory.

TABLE XXIII. RESULTS OF BALLISTIC-IMPACT TESTS ON COATED MAR-M200 AND UDIMET 700 SUBSTRATES (REF. 29)

Substrate	Coating	Condition	Test Temperature, F	Results
MAR-M200	Martin Metals A-11	As received	75	Slight cracks
			1700	Slight cracks
		Erosion tested	75	Cracks
			1700	Satisfactory
	PWA 47	As received	75	Cracks
			1700	Satisfactory
		Erosion tested	75	Cracks
			1700	Slight cracks
Udimet 700	Martin Metals A-11	As received	75	Cracks
			1700	Cracks
		Erosion tested	75	Cracks
			1700	Cracks
	PWA 47	As received	75	Satisfactory
			1700	Satisfactory
		Erosion tested	75	Cracks
			1700	Satisfactory

Aluminum-Tantalum. The Air Force is sponsoring a program in which aluminum-tantalum coatings are being applied to MAR-M200 and Udimet 700 using a spray-and-diffuse technique to develop coated superalloy components for gas-turbine engine applications (Refs. 29,67-69). The same coating has also been applied to B-1900 (Ref. 75). In each case, the alloy substrate is conditioned by blasting with No. 60 aluminum oxide grit at 40 psi for 2 to 5 minutes, followed by degreasing with trichloroethylene at 180 F for 5 to 10 minutes. The slurry is prepared in batches using the following materials (Ref. 69):

452 grams of analytical-grade tantalum powder
(Fansteel Metallurgical Company)

203 grams of flake aluminum powder
(Metals Disintegrating Company)

575 mils of Pratt and Lambert No. 2012 nitrocellulose
lacquer.

The metal content of this slurry is 75 atomic per cent aluminum, 25 atomic per cent tantalum; hence, the coating has been referred to in the literature (Ref. 75) as $TaAl_3$. Very little tantalum has been found in the deposited coatings, however (Ref. 70).

The ingredients are mixed by ball milling in a 1-gallon porcelain crock with 1-inch-diameter porcelain balls for 8 to 16 hours. The desired viscosity is 600 to 1000 cps at 65 to 85 F, as measured by a Brookfield Viscometer Model RVT at 10 rpm and spindle number 1 (Ref. 69).

The resultant slurry is sprayed on the substrates and dried in air for at least 4 hours, after which the material is subjected to a diffusion heat treatment for 4 hours at 2000 F in a hydrogen atmosphere (Ref. 69).

Samples of IN-100, MAR-M200, and B-1900 coated by this process were subjected to the thermal-fatigue bow test (Ref. 75) described on pages 61-62. The only available results of these tests are given in Figure 26 (Ref. 75). These data indicate that the $TaAl_3$ coating increases the resistance of B-1900 and MAR-M200 to thermal-fatigue cracking. The data given for IN-100 substrates are too limited to warrant any definite conclusion, but indications are that the coating may adversely affect the thermal-fatigue resistance of this substrate alloy.

Beryllium With Unidentified Alloying Elements. Whitfield Laboratories, Inc., has developed a beryllium-base coating, WL-6, that is designed to provide resistance to sulfidation. The coating is applied by pack cementation; details of the process are proprietary. Data showing the specific resistance of this coating to sulfides will be published in the near future; such data were not available for this report.

Boron. Boron forms a series of borides with nickel, which includes Ni_2B, Ni_3B_2, NiB, and possibly the compound Ni_2B_3 (Ref. 76). Accordingly, nickel can be boronized, that is, a case or coating comprising one or more nickel borides can be caused to form on a nickel workpiece.

Nickel has been boronized by a vapor-plating method in a mixture of boron trichloride and hydrogen in the ratio of 1 to 4 at a temperature of 1650 F (Ref. 77). The operation was carried out in a quartz tube heated by a tubular electric-resistance furnace, and provided with appropriate equipment to admit and discharge the gas mixture. The case that formed comprised three distinct layers, while the boundary between case and core was jagged. The hardness of the outer layer was about 1700 DPH (Diamond Pyramid Hardness), that of the middle layer was about 1500 DPH, while the hardness of the inner layer was some 1150 DPH. Presumably, the composition of these layers corresponded to NiB, Ni_3B_2, and Ni_2B, respectively.

The thickness of the case was observed to be a linear function of time at temperature, indicating that the boride layers already formed did not act as barriers to the diffusion of boron to the metal. When the rate of flow of the gas mixture was increased the rate of boronizing also increased, the relationship between the two being reported as more or less quadratic. Finally, the rate of boronizing was found to be independent of the concentration of boron trichloride in the gas, within the range of concentrations investigated. This observation was taken to mean that the boron trichloride did not take a direct part in the boronizing process, but was only the initial reactant from which the compound was formed that was ultimately involved in the actual boronizing step. It was speculated that the active agent was a compound of boron and hydrogen.

In another investigation involving the vapor-plating method it has been found that nickel-base alloys, in addition to nickel itself, can be boronized (Ref. 78). The vehicle that provided the boron was a mixture of 25 parts of hydrogen to 1 part of diborane. Boride layers ranging from 20 to 60 microns in thickness could be obtained on various alloys, depending on the conditions of time and temperature. In addition to the surface boride layer, a boron-containing phase progressed intergranularly well below the surface layer. Table XXIV shows the results.

TABLE XXIV. RESULTS OF BORONIZING EXPERIMENTS ON NICKEL-BASE ALLOYS (REF. 78)

Alloy	Boronizing Conditions		Depth of Layer, microns		Microhardness, DPH
	Temperature	Time, hr	Boride	Overall	
Ni-20Cr	900 C (1650 F)	4	20-25	70-100	1780
Ni-27Cr-3Fe	950 C (1740 F)	6(a)	50-60	100-130	1890
Ni-20Cr-0.4Ti	950 C (1740 F)	6(a)	50	150-180	2010
Ni-16Cr-27Fe	950 C (1740 F)	6	20	130-150	1830

(a) Diffusion treatment of an additional 4 hours after boronizing.

Thus, it is evident that cases of extreme hardness can be applied to nickel and nickel alloys by boronizing. In addition to high hardness, it can be speculated that such cases may possibly have useful specific corrosion resistance.

Carbon. The nickel-carbon binary system features a eutectic at 2400 F and 2.22 per cent carbon, the phases involved being the terminal solid solution of carbon in nickel and free graphite. The solid solubility of carbon in nickel reaches a maximum of 0.55 per cent at the eutectic temperature and decreases to virtually nil at room temperature. The constitution diagram of the system does not show a carbide because nickel carbides are unstable and are extremely difficult to produce (Ref. 79).

Because nickel carbides are not stable, a hard case cannot be developed on nickel by a carburizing treatment, as can be done with iron. However, under some conditions, nickel will absorb carbon from its environment at elevated temperatures because of the measurable solubility of carbon in nickel at such temperatures; then, on slow cooling or on holding at intermediate temperatures, the carbon will precipitate in the metal in the form of graphite. For example, when heated 1000 hours at 1700 F in a mixture of 95 per cent graphite and 5 per cent $BaCO_3$ and then held at 1200 F for an additional 1000 hours, ordinary wrought nickel has been observed to absorb carbon and then graphitize (Ref. 80). Again, the carbon content of a nickel specimen has been increased from 0.01 per cent to 0.52 per cent on exposure to 1 atmosphere of CO for 500 hours at 1600 F, much of the carbon precipitating as graphite when the metal was cooled to room temperature (Ref. 81). In addition, long-time exposure in a mixture of 1 part CO and 1 part H_2 at temperatures of 1180 to 1750 F has caused nickel to absorb considerable amounts of carbon (Ref. 81).

The addition of copper to nickel decreases the solubility of carbon in the metal at elevated temperatures. For this reason, Monel, which contains about 30 per cent copper, is considerably more resistant to carbon absorption and graphitization. For example, Monel did not graphitize when exposed to the $BaCO_3$-graphite mixture for 1000 hours at 1700 F and was then reheated at 1200 F for 1000 hours (Ref. 80). Likewise, while Monel was observed to absorb carbon and to graphitize when exposed to CO and to a CO-H_2 mixture under the same conditions as mentioned above for nickel specimens, it did so to a much lesser extent than did the nickel (Ref. 81).

On the other hand, nickel does not absorb carbon under the conditions used to carburize steel. In particular, the time periods employed to carburize steel are much shorter than those required to cause nickel to absorb appreciable amounts of carbon. In fact, nickel alloys are often used for components of equipment in carburizing service precisely because of their resistance to the carburizing environment. For example, the wrought nickel-base alloy, Inconel 600, is frequently used for such components of carburizing and carbonitriding furnaces as retorts, muffles, and radiant tubes. Inconel 600 is also used for carburizing boxes, trays, baskets, and fixtures. The 80Ni-20Cr alloy is frequently used for woven-wire-mesh conveyor belts, with which the smaller carburizing and carbonitriding furnaces carrying relatively light loads are often equipped. Among nickel-base cast heat-resistant alloys, HW* and HX* are used for carburizing fixtures, trays, and baskets, especially where the heat-treating cycle involves oil quenching (Ref. 82).

It is to be noted that the nickel-base alloys usually employed for components in carburizing service contain substantial amounts of chromium and iron. The chromium provides the nickel base with needed high-temperature strength and oxidation resistance, while the iron acts largely as a compatible diluent that reduces the cost of the material. However, although nickel-chromium-iron alloys frequently give service in these applications extending to many thousands of hours, the presence of the chromium and the iron detracts somewhat from the essential immunity of nickel to carburizing. These alloys

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*Alloy Casting Institute designation.

can, in fact, be carburized, and to a considerable extent, though generally the time required is exceedingly long. Reducing the nickel content decreases resistance to carburizing, while the addition of silicon tends to enhance resistance.

Even for those nickel-base alloys that can be carburized, the treatment generally is not considered to be advantageous. In nickel-chromium and nickel-chromium-iron alloys, the tendency is for a case to form that contains massive chromium carbides in a chromium-depleted matrix. The effect is to promote brittleness and reduce corrosion and oxidation resistance. Moreover, in titanium- and columbium-containing alloys, carburizing tends to detract from their age-hardening capabilities by forming titanium and columbium carbides. Consequently, in heat treating nickel-base alloys atmospheres capable of carburizing them are avoided (Ref. 83).

Among the principal reasons for employing nickel alloys is to make use of their toughness and resistance to corrosion and oxidation. When the hardness and wear resistance of a carburized surface is called for, a technically superior and less costly product is usually available in the form of a carburized steel.

On the other hand, there may be special instances in which an extremely hard surface is desired on an alloy having superior high-temperature strength, such as a nickel-base high-temperature alloy. If the high-temperature strength of a relatively simple alloy is acceptable, then a satisfactory surface-hardened nickel-base material may be obtainable. A suitable base or core material might be such an alloy as Hastelloy B. As such, this alloy does not carburize. However, a hard case of chromium carbide can be applied to it by first chromizing the alloy and then carburizing it afterwards (Ref. 84).

Nitrogen. Nickel and nitrogen are quite incompatible. The solubility of nitrogen in nickel is limited and nickel-nitrogen compounds form only under special conditions. Nickel does not nitride under the conditions normally used to nitride steel (Ref. 85).

Likewise, most nickel-base alloys are highly resistant to nitriding and, in fact, the 80Ni-20Cr alloy and Inconel 600 are frequently used for retorts, workpiece containers and baskets for the nitriding of steel because of their excellent resistance to nitriding (Ref. 82). However, at higher temperatures and longer times than are generally employed in nitriding steel, these alloys will develop a thin case. For example, in an atmosphere of ammonia at 1250 F a case 0.0015 inch thick was developed on Inconel 600 when exposed for 100 hours (Ref. 86). The hardness of the case was 726 DPH (Diamond Pyramid Hardness), while that of the core was 155 DPH. Usual temperatures for nitriding steel are in the vicinity of 1000 to 1050 F.

Zirconium and Zirconium-Chromium. LTV Vought Aeronautics Division, Ling-Temco-Vought, Inc., Dallas, Texas, has achieved "promising results" in their efforts to protect TD nickel with zirconium and zirconium-chromium coatings (Ref. 22). Specific properties obtained on coated specimens are not available, however. Potassium iodide, sodium bromide, sodium fluoride, and potassium chloride were all studied as candidates for the activator in the pack. It was found that the choice of an activator is very important in that the activator influences both the quality of the coating and its ability to adhere to the substrate (Ref. 22). The activator finally chosen for the zirconium and zirconium-chromium processes was not disclosed.

ELECTROPLATING

Hundreds of components of aircraft and spacecraft are electroplated with a variety of metals and for a number of purposes. The metals most frequently used as coatings for these parts are gold, copper, nickel, chromium, silver, and zinc. The purposes served by the coatings include prevention of tarnish and corrosion, increase in wear and abrasion resistance, and control of the temperature of the component.

Gold is commonly used because of its favorable energy absorptance and emittance characteristics; in addition, gold is highly corrosion resistant (Ref. 87). Nickel plate is adherent, corrosion resistant, and can be quite hard as deposited. Hardness can be varied from about 150 to some 800 Vickers (Ref. 84). Black nickel, which usually is an alloy containing up to 48 per cent zinc along with sulfur and organic inclusions, is often applied to interior surfaces to increase heat absorption, reduce heat reflection, or promote uniformity of temperature in a component or assembly (Refs. 87, 88). Zinc, copper, and silver are often employed as substrate barrier coatings (Ref. 87). Industrial chromium plates are hard (up to 1100 Vickers), are corrosion resistant, have a low coefficient of friction, and possess excellent nongalling and nonwetting properties (Ref. 84). A summary of the characteristics of these and other electrodeposited metallic coatings is given in Table XXV.

T

		Physical and Mechanic			
Plate$^{(a)}$	Usual Basis Metals	Melting Point, F	Thermal Conductivity, Btu/hr/sq ft/°F/ft	Electrical Resistivity, microhm-cm	Reflectance (polished), per cent at 5000 A
Aluminum	Steel, iron, copper, magnesium, silver, gold, zinc, nickel	1200	122	2.8	71
Antimony	--	1166	10.2	41.7	50
Arsenic	Brass	1139	--	35	--
Bismuth	--	520	4.6	119	72$^{(b)}$
Cadmium	Steel, stainless steel, wrought iron, gray and malleable iron, copper and its alloys	610	5.3	7.5	--
Chromium	Ferrous, nonferrous metals	2939	--	14-66	High
Cobalt	Iron, steel, copper and its alloys	2736	--	--	High
Copper	Most ferrous, nonferrous metals	1981	222	3-8	44
Gold	Copper, brass, nickel, silver	1944	169	2.4	47
Indium	Silver-plated steel, lead-bearing metals	311	--	8.4	--
Iron	Ferrous metals	2795	38.7	10	0.55
Lead	Ferrous metals, copper	621	20.1	22.6	Low
Nickel	Most ferrous, nonferrous metals	2651	34.4	7.4-10.8	61
Palladium	Copper and its alloys plus underplate of silver, gold or platinum	2820	40.6	11	72$^{(b)}$

LE XXV. ELECTRODEPOSITED COATINGS (REF. 89)

roperties					
Hardness	Abrasion Resistance	Adhesion	Appearance	Cost	Thickness, mil
0-90 Vickers	Poor	--	White	Moderate	0.25+ (can also be electroformed)
ntermediate	--	Sometimes very poor	Bright white	Low	1
--	--	--	Black or gray, often antiqued	--	--
omparable to lead	--	--	White, gray (resembles nickel when polished)	Moderate to high	--
0-50 DPH	Fair	Good	Bright white	Moderate (8 to 10 times more expensive than zinc)	ASTM specifies (min): 0.15 (indoors), 0.3 (intermediate), 0.5 (outdoors)
00-1100 Vickers	Excellent	Excellent	White, mirror-like; black also available	Moderate	0.01-0.06 (decorative), 0.05-12 (hard)
50-350 Knoop	Good	--	Gray or bluish-white	Moderate	0.1-1.0 (decorative)
1-220 Vickers	Poor	Excellent	Bright or semibright red or pink	Low	0.2-2 (undercoat), 3 (functional topcoat)
5-125 for gold; 150-325 for 1%Ni alloys, 450 for 30% Ni alloys	Poor-good	Excellent	Natural bright yellow color; many other colors obtainable by alloying	High	0.002-0.01 (decorative) 0.01-2.0 (functional), 2-15 (electroforming)
oft	Poor unless diffused	Excellent when diffused	Silver-white, satin finish	--	1.1
125-400 Bhn	Very good	Very good	Matte gray	Low	125+
5 Bhn	Poor (but alloy coatings have good bearing properties	Good	Gray	Low	0.5-8 (wear) to 50 (corrosion)
140-500 DPH	Good-very good	Very good	White, either dull or bright	Moderate	0.1-1.5 (decorative), 5-20 (industrial), up to 1/4 in. (electroforming)
255-265 Knoop	--	Careful processing needed to prevent peeling	White, tends to tarnish	High	0.02-0.2

2

TAB

Plate[a]	Usual Basis Metals	Physical and Mechanical Properties				
		Melting Point, F	Thermal Conductivity, Btu/hr/sq ft/°F/ft	Electrical Resistivity, microhm-cm	Reflectance (polished), per cent at 5000 A	Hardness
Platinum	Gold, copper alloys	3223	40.2	10	58	280-29 Knoop
Rhenium	--	5732	--	21	--	250 Bhn
Rhodium	Most ferrous, nonferrous metals	3553	50.9	4.7	76	400-800 Bhn
Silver	Most ferrous, nonferrous metals	1760	244	1.6	91	50-150
Tin	Usually ferrous metals	448	36.3	11.5	54[b]	5
Zinc	Usually ferrous metals	786	64.2	5.8	55	40-50
Cobalt-Nickel	--	--	--	--	--	350-450 DPH
Copper-Tin (Bronze)	Steel, copper, brass, zinc	--	--	--	High	150-250 DPH
Copper-Zinc (Brass)	Iron, steel, aluminum, zinc	--	--	--	High	Soft
Lead-Tin	Steel, copper, brass	--	--	--	--	Soft
Tin-Nickel	Most ferrous, nonferrous metals	--	--	--	15-118	625 Vickers
Tin-Zinc	Most ferrous, nonferrous metals	--	--	--	--	Similar to ti

(a) Important characteristics and uses for each plate are given on pages 79-81.
(b) At 10,000 A.

XXV. (Continued)

brasion Resistance	Adhesion	Appearance	Cost	Thickness, mil
Poor	--	Bright gray, darker than rhodium or silver	High	Flash up to 2
--	--	Bright gray	High	--
High	Good	Mirror-bright white	Moderate	0.001-1
Good	Good	Bright white	High	0.1 (with undercoat) to 1
oor unalloyed	Good	Bright white	Moderate	0.015-0.5
Poor	Excellent	Matte gray to bright	Low	0.1-0.5 (light duty), 0.5-2 (outdoors)
Very good	Good	Gray	Moderate	0.1
Good	Excellent	Bright pink	Low	0.5
Poor	Excellent	Bright yellow at 10% tin. Yellow at 20% tin	Low	0.1
Poor	Good	White	Low	0.2
Good	Good over copper or bronze undercoat	Bright white	Moderate	0.5
Good	Good	White	Moderate	0.15

Plate	Important Characteristics and Uses
Aluminum	Corrosion resistant; good thermal- and heat-resistance properties when diffused into base metal. Infrequently used; can substitute for hot-dipped aluminum
Antimony	Tarnish resistant, pleasing appearance when polished. However, coatings too brittle for practical application
Arsenic	Infrequently used to provide decorative antique effect
Bismuth	Somewhat expensive. Infrequently used for corrosion protection
Cadmium	Pleasing appearance, provides good corrosion protection indoors on iron and steel. Less apt to darken and form corrosion products than zinc; nevertheless, zinc is more widely used because of cost. Outdoor corrosion resistance varies and should be checked by testing. Radio, television, and electronic chassis; aircraft, marine, and military outdoor use; washing equipment, steel embedded in concrete, and similar parts
Chromium	Excellent resistance to wear, abrasion, and corrosion. Low coefficient of friction and high reflectivity. Popular decorative corrosion-resistant coating on automobile exterior and interior trim, appliances, and business machines. Widely used in bearing applications and to build up worn surfaces
Cobalt	Similar to nickel, but more expensive. Infrequently used alone except for applications where high hardness is needed and on mirrors and reflectors. Frequently used in alloy electroplates to upgrade properties
Copper	Good appearance (when polished and/or lacquered) and corrosion resistance; high electrical and thermal conductivity. Undercoat to improve adhesion and protective ability of subsequent electroplates (e.g., nickel and chromium) or to prevent hydrogen embrittlement. Also used as wire coatings, stop-off coatings during heat treatment and chemical milling, lubricant during drawing, thermally conductive coatings on cooking utensils, and for electroforming
Gold	Resistant to tarnishing, chemical attack, and high-temperature oxidation. Good ductility, thermal reflectivity and electrical conductivity. Pen points, jewelry, watch and vanity cases, musical instruments, reflectors, nameplates, eyeglass frames, bracelets, trophies, novelties, electrical contacts, springs, waveguides, various electronic parts, laboratory apparatus
Indium	Tarnish resistant, malleable, and ductile. Principally used as an overlay diffusion coating on silver-plated steel bearings for high-speed aircraft engines. Also used as an alloying element to upgrade mechanical properties of alloy coatings
Iron	Can be deposited with selected properties; easily fabricated and plated over; high purity. Build-up of undersized parts, electrotypes, electroforming of molds

Plate	Important Characteristics and Uses
Lead	Resistant to many acids, hot corrosive gases, and corrosive atmospheres. Lead normally deposited by hot dipping; however, lead electroplates are used to protect chemical equipment, brine refrigerating tanks, metal gas shells, nuts and bolts, and storage battery parts
Nickel	Excellent appearance, and resistance to a wide variety of chemicals and corrosive atmospheres; can be electroplated in range of hardness (soft to hard). Decorative applications either alone or as a heavy base for thin chromium electroplates to improve corrosion resistance and mechanical properties, as in trim for automobiles, appliances, business machines, and consumer goods. Also used for electroforming, and to build up worn and mismachined parts
Palladium	Good appearance, tarnish and corrosion resistance. Ornamental and decorative applications. Can be used alone or under rhodium for radar and electronic equipment
Platinum	Good appearance, tarnish and corrosion resistance. Ornamental ("white gold") applications and as a flash decorative coating. Also to protect surfaces that must withstand unusual corrosive environments
Rhenium	Although melting point is high, begins to oxidize as low as 752 F. Poor resistance to moisture. Infrequently used but has been proposed for electronic applications such as cathode and filament emitters and heater wires
Rhodium	Brilliant white color, tarnish and corrosion resistant, good electrical conductivity. Decorative and tarnish-resistant finish for costume jewelry, insignia, emblems, musical instruments, medical and surgical parts, laboratory equipment, and optical goods. Also electrical contacts, reflectors and mirrors
Silver	Excellent appearance when suitably prepared and protected, high load-carrying capacity and electrical conductivity, good resistance to many chemicals. Decorative applications, such as tableware, hollow-ware, cigarette lighters, and musical instruments. Also industrial applications, such as bearings, surgical instruments, chemical equipment, electrical contacts, etc., where conductivity is needed
Tin	Corrosion resistant, attractive appearance, hygienic, easily soldered, soft and ductile, good bearing properties. Food and beverage containers, refrigerator evaporators, food and dairy equipment, hardware, appliance and electronic parts, copper wire, and bearings
Zinc	Easily and rapidly applied, high corrosion resistance. Electrogalvanized sheet is widely used in fabricating appliance and automotive parts. Also, for finishing small parts such as pipe couplings, bolts, nuts, rivets, washers, nails, hinges, hangers, hooks and buckles. Larger parts include electrical conduit pipe, silo and tie rods, screening, telephone exchange equipment, and iron and steel castings

Plate	Important Characteristics and Uses
Cobalt-Nickel	Wide range of magnetic properties. Magnetic recording, permanent magnet coating on memory drums in digital computers. Electroforming
Copper-Tin (Bronze)	Red-bronze coatings (lacquered to prevent tarnishing) used on inexpensive jewelry, door plates, hardware, trophies, handbag frames; as undercoat for nickel and chromium; as stop-off coating in selective nitriding of steel; also used on bearing surfaces. Speculum coatings (40-50% tin) used for tableware and household fixtures; not recommended for outdoor use
Copper-Zinc (Brass)	Rich appearance much like solid brass. Little resistance to outdoor atmospheres; may require lacquer coating to resist indoor tarnishing. Decorative applications such as lamps and trays; low-cost trim, interior automotive hardware, tubular furniture, household goods, toys, casket hardware, novelties. Also used to promote adhesion of rubber to steel
Lead-Tin	Harder and more protective than lead; good friction and bearing properties. Heavy duty bearing where corrosion protection is needed, and as an aid in soldering
Tin-Nickel	Good decorative properties (resembles chromium); high resistance to tarnishing, common reagents, and marine corrosion; good bearing and oil retention properties; solderable. Plating baths have outstanding throwing properties. Cooking utensils, analytical weights and surgical instruments, numerous watch parts, chemical pumps, valves and flow control devices
Tin-Zinc	Good corrosion resistance, excellent solderability, tendency to fingermark (can be prevented by lacquering). Radio and television parts, cable connectors, relay assemblies, galvanic protection of steel parts contacting aluminum

Electroplates can be deposited on a vast number of metals and alloys. For aerospace applications requiring electroplating, the most common basis metals are aluminum and its alloys, magnesium and magnesium alloys, low-alloy steels, and (less frequently) stainless steels and nickel-base alloys (Refs. 87, 90). Generally, the stainless steels and nickel alloys either are not used in applications where plating is suitable or required, or they do not need to be plated because they inherently possess the corrosion resistance or other attribute that plating would endeavor to supply.

Most of the plates discussed above and listed in Table XXV can be electrodeposited on nickel and nickel-base alloys. In general, each metal can be plated from several baths of different types and compositions. Each bath has its own realm of applicability, and its own set of operating conditions and requirements regarding the preparation of the basis metal. Thus, the technology of electroplating is vast and complex and a detailed treatment of the subject would be beyond the scope of this report. For more complete information on electroplating, the attention of the reader is invited to References 91 and 92.

It is appropriate, on the other hand, to present information here on procedures for preparing nickel and nickel alloys prior to plating. In general, all metallic materials require careful preparation, principally cleaning, before electroplating. However, the procedures vary both with the basis metal and the plate. In fact, it is largely in the preparation step that plating processes are influenced by the basis material.

In the preparation of nickel and its alloys for electroplating, cleaning is carried out by standard procedures (Ref. 91). The distinguishing feature of nickel and nickel alloys, however, is that they tend to be passive, that is, to possess oxygen-containing films on their surfaces. These films interfere with the adherence of electroplates and, consequently, must be removed prior to electroplating. The film-removal step is known as activation, and follows cleaning in the processing sequence.

Examples of activating procedures used for some nickel-base alloys before nickel plating are given in Table XXVI, while procedures for activating nickel alloys prior to chromium plating are shown in Table XXVII. A method used in Great Britain to activate Nimonic alloys before chromium plating is to etch 1 minute at room temperature in a bath composed of 250 to 333 g/l hydrated ferric

TABLE XXVI. PROCEDURES FOR ACTIVATING NICKEL AND NICKEL ALLOYS PRIOR TO NICKEL PLATING (REF. 93)

Alloy	Bath	Temperature, C	Polarity	Current Density, amp/dm^2	Time, min	Subsequent Treatment
Electrolytic nickel Nickel 200 Duranickel 301	25 wt % H_2SO_4, 22 oz/gal, 66 Bé sulfuric acid, chemical lead electrodes	21-27	Anodic Anodic Cathodic	2 22 22	10 2 1/30-1/20	Rinse and nickel plate
Monel 400 Monel K-500 Monel R-405 Monel 501	Same as above	21-27	Anodic	22	5 min after passivity	Rinse and nickel plate
D nickel Electrolytic nickel	Low-pH Watts' nickel or chloride nickel bath	54-60	Anodic Cathodic	1-2 Normal	10	Nickel plate
Nickel 211 Nimonic 75 Nimonic 80A Inconel 600	Wood's nickel bath ($NiCl_2 \cdot 6H_2O$), 240 g/l; HCl, 80 g/l	21-27	Anodic Cathodic	3 3	2 6	Nickel plate without rinsing
Nickel 211	Same as above	21-27	Anodic Cathodic	3 3	1/4 6	Nickel plate without rinsing
Inconel 722 Inconel X-750	50 wt % HF	21-27	Immerse	0	1/6	Rinse and plate

chloride and 155 to 170 ml/l hydrochloric acid (1.16 specific gravity) (Ref. 90). The work is rinsed and transferred immediately to the plating bath. For nickel-chromium plating of this type of alloy, electrolytic pickling in a solution of 240 g/l nickel chloride and 86 ml/l hydrochloric acid has been recommended. The parts are made anodic for 2 minutes at 30 amp/sq ft and then made cathodic for 6 minutes at the same current density, before being transferred directly to the nickel plating bath without rinsing (Ref. 94).

TABLE XXVII. PROCEDURES FOR ACTIVATING NICKEL-BASE ALLOYS FOR CHROMIUM PLATING (REF. 93)

Alloy	Activating Procedure
Inconel X-750 Hastelloy C	(1) Immerse in 6N HCl at room temperature for 1 minute (2) Immerse in Wood's nickel bath(a) (3) Reverse etch at 5.5 amp/dm^2 for 20 seconds (4) Strike plate at 5.5 amp/dm^2 for 2 minutes (5) Cold-water rinse (6) Immerse in chromium bath for 30 seconds (7) Plate(b)
Hastelloy B	(1) Immerse in 6N HCl at room temperature for 1 minute (2) Cold-water rinse (3) Immerse in 15N HNO_3 at 71 C for 30 seconds (4) Cold-water rinse (5) Immerse in chromium bath for 30 seconds (6) Plate(b)

(a) Wood's nickel bath: 240 g/l $NiCl_2 \cdot 6H_2O$; 80 g/l HCl, 20 to 30 C.

(b) Chromium plating bath: 250 g/l CrO_3; 2.5 g/l H_2SO_4, 55 C. Plating current density: 30 amp/dm^2.

In addition to the aqueous electroplating processes discussed above, a unique process for applying nickel-aluminum alloy coatings by a combination of aqueous and nonaqueous electroplating methods was disclosed in 1960 (Ref. 95). The objective was to provide a highly oxidation-resistant coating protective to temperatures of 1800 F and above. The coating is said to have proved effective on steel, nickel, and molybdenum. The coating procedure was developed by D. E. Couch and J. H. Connor of the National Bureau of Standards.

The most satisfactory procedure that they developed consisted in electrodepositing nickel from a Watts type of bath, i.e., a mixed chloride-sulfate bath, and then electroplating aluminum on the nickel layer at a current density of 2 to 15 amp/dm^2. The aluminum was deposited from a fused salt solution containing a mixture of potassium chloride, sodium chloride, and cryolite (sodium aluminum fluoride) at 1290 F. At this temperature, aluminum diffuses into the nickel layer, as it deposits, thus forming the alloy.

ELECTROLESS PLATING

Electroless plating made its debut in 1946 with the announcement of a process invented by Brenner and Riddell for plating nickel by making use of a spontaneous autocatalytic chemical reaction. Since then the technology of this type of plating process has advanced, and it is now possible to deposit nine different metals singly or in combination. The metals that have been plated are: cobalt, chromium, copper, gold, iron, nickel, palladium, silver, and vanadium (Ref. 96).

In the meantime, the term "electroless plating", which was coined by the original inventors as a label for their process, has become widely used. In fact, it is frequently used to designate other chemical plating processes in addition to the autocatalytic process. Thus, the term is sometimes applied to the salt-water gold process, contact plating processes, and immersion or replacement plating (Ref. 97).

True electroless plating differs significantly from all other chemical plating processes. Not only does electroless plating require no electric current, it is the only chemical plating process that does not depend on the presence of a couple between galvanically dissimilar metals. Thus, no external connection is made to an active metal, as in the salt-water gold process; nor does the work contact any active metal in solution, as in contact plating; nor does the substrate dissolve, as in immersion plating (Ref. 97).

In electroless plating, a catalytic substrate is immersed in an aqueous plating solution, whereupon the reaction begins spontaneously and metal is deposited only on the substrate surface. The deposited metal also catalyzes the reaction, causing it to continue autocatalytically. The essential ingredients of the plating solution are the metal ion and a reductant. Only certain metals have the required reduction potential and autocatalytic properties. In addition to those listed earlier they are ruthenium, rhodium, osmium, iridium, and platinum (Ref. 96). In the plating reaction, the metal ion is reduced to the corresponding metal by receiving the required electrons, which become available upon the oxidation of the reducing agent. Such reductants as hypophosphite ion, borohydride ion, hydrazines, formaldehyde, and boranes have been employed (Ref. 96).

Another important constituent of the plating solution is a compound, frequently the salt of an organic acid, which acts both as a buffer and as a complexing agent for the metallic ion. Buffering is important because the pH of the bath has a strong influence on its operation and the pH tends to change during bath operation. The function of complexing is to help control the availability of the metal ion so as to encourage plating only on the substrate, and discourage the formation of sludges and the spontaneous decomposition of the bath (Ref. 97).

To date, the electroless plating of nickel has become a thoroughly commercial process, while the plating of other metals by this method is not so well advanced. Compositions of solutions for depositing electroless nickel are given in Table XXVIII. The deposits are not pure nickel but are actually alloys that usually contain 4 to 8 per cent phosphorus. The presence of the phosphorus makes the deposits harder, more corrosion resistant, and less magnetic than electrodeposited nickel. The hardness of electroless nickel as deposited is about 500 Vickers; when the plate is heated for an hour at 750 F its hardness increases to some 700 Vickers. The deposits are reported to be brittle, but adherent and low in internal stress; they are smooth, semibright, and have a good appearance (Ref. 98).

Other features of electroless plating, which apply particularly to electroless nickel plating, include the fact that the solutions have perfect throwing power and can deposit uniform thick coatings on articles of complex shape. Also, the deposits are more pore-free than electroplates. Again, nonconductive surfaces can be easily sensitized to permit electroless plating (Ref. 96).

Electroless nickel plate has been used on certain aircraft parts because of its hardness, wear resistance, and low level of internal stress. The heat-treated deposit has been preferred for its superiority in these respects to the as-deposited plate (Ref. 99). In addition, heat-treated electroless nickel deposits are often used as undercoatings beneath gold electroplates on satellite and other aerospace components formed from magnesium and aluminum (Ref. 87).

TABLE XXVIII. TYPICAL COMPOSITIONS OF BATHS FOR DEPOSITING ELECTROLESS NICKEL (REF. 98)

All Baths are Operated at 90 to 100 C, Except Bath 8, 65 to 75 C and Bath 6, 80 C

	Acid Baths						Ammoniacal Baths	
	Hydroxy Acid Baths			Fluoride Baths				
	1(a)	2	3	4	5	6	7	8
Constituents of Baths, g/l	Hydrac	Citrate Acetate	Lactate-Propionate(b)	Sol-ov'ev	Gutzeit, 2, 694, 019	Dow, British 830, 597	Citrate	Pyro-phosphate
Nickel chloride, $NiCl_2 \cdot 6H_2O$	30	30	26	30(c)	21	10(d)	30	25(c)
Sodium hypophosphite, $NaH_2PO_2 \cdot H_2O$	10	10	24	30	24	20	10	25
Sodium citrate, $Na_3C_6H_5O_7 \cdot 5\text{-}1/2H_2O$	--	15	--	--	--	10	100	--
Sodium pyrophosphate, $Na_4P_2O_7$	--	--	--	--	--	--	--	50
Hydroxyacetic acid	35	--	--	--	--	--	--	--
Lactic acid	--	--	27	--	--	--	--	--
Propionic acid	--	--	2. 2	--	--	--	--	--
Succinic acid	--	--	--	--	7	--	--	--
Sodium acetate	--	5	--	--	--	--	--	--
Ammonium chloride	--	--	--	--	--	--	50	--
Ammonium fluoride	--	--	--	15	5(e)	(f)	--	--
Lead ion	--	--	0. 002	--	--	--	--	--
Alkali for neutralizing	NaOH	NaOH	NaOH	?	--	--	NH_4OH	NH_4OH
pH	4-6	4-6	4. 6	6-7	6	6. 5	8-10	10-11
Rate of deposition, μ/hr	15	7	20	30	15	30	8	15

(a) This bath is referred to as "Hydrac", which is a contraction of "hydroxyacetic".
(b) Bath patented by General American Transportation Co.
(c) $NiSO_4 \cdot 6H_2O$
(d) Basic nickel carbonate, $2NiCO_3 3Ni(OH)_2 \cdot 4H_2O$.
(e) Sodium fluoride.
(f) 70% HF solution, 5 ml/l; KF, 10 g/l; NH_4HF_2, 25 g/l.

So far as is known, the electroless plating process is not used to any great extent to coat nickel and nickel-base alloys. On the other hand, electroless plates can be deposited on these materials; in fact, reference is made in Military Specification MIL-C-26074A to nickel, among other metals, as a substrate on which to deposit electroless nickel-phosphorus coatings (Ref. 100). The coating is deposited on nickel and its alloys after they have been cleaned and activated in the same manner as is used for electroplating; for best results, it is reported that the final acid dip should preferably contain fluoride (Ref. 97). On nickel, the coating is probably used primarily to impart resistance to wear.

HARD FACING

Hard facing is the process of applying a layer, edge, or point of metal to a metallic part by a welding method or comparable technique (Ref. 101). The principal objective of hard facing is to increase the resistance of the part to various types of abrasion, wear, or erosion as well as to corrosion, high-temperature oxidation, or thermal shock, or to combinations of these factors (Ref. 102).

Several classes of hard-facing materials have been developed. A ferrous-based group, alloyed with small amounts of such elements as chromium, manganese, tungsten, molybdenum, and carbon, displays excellent shock resistance. Increased wear resistance is built into iron-base hard-facing alloys by larger additions of such elements as chromium, molybdenum, and boron. These more highly alloyed materials also possess improved resistance to corrosion and heat (Refs. 101, 102). Iron alloys, containing sufficient nickel or manganese to make them austenitic as deposited, offer good impact and abrasion resistance at room and elevated temperatures (Ref. 103).

Cobalt-base alloys, containing principally chromium, tungsten, and carbon, offer outstanding resistance to wear, abrasion and erosion, along with excellent resistance to corrosion and high-temperature oxidation. These alloys also retain high hardness at elevated temperatures (Ref. 103).

Nickel-base hard-facing alloys contain principally chromium, boron, silicon, and carbon. These alloys have excellent heat and corrosion resistance, with fair resistance to abrasion and impact. They display quite high hardness at elevated temperatures (Ref. 103).

Another type of hard-facing material is composed of tungsten carbide particles in a matrix such as cobalt. This material combines extremely high hardness with excellent resistance to impact and corrosion (Ref. 103). The compositions of some representative hard-facing materials are given in Table XXIX.

TABLE XXIX. NOMINAL COMPOSITIONS OF SOME HARD-FACING ALLOYS

Type	Composition, weight per cent										Reference
	C	Cr	Mo	W	B	Si	Fe	Ni	Co	Other	
Iron base	0.55	2.5	0.4	--	--	0.5	Bal	--	--	--	102
Ditto	0.3	6.0	0.8	--	--	1.1	Bal	--	--	--	102
"	3.0	17.0	16.0	--	--	--	Bal	--	6.25	1.9V	102
Cobalt base	2.5	30.0	--	12.0	--	--	--	--	Bal	--	102
Ditto	1.3	29.0	--	8.0	--	--	--	--	Bal	--	102
"	1.1	28.0	--	4.0	--	--	--	--	Bal	--	102
Nickel base	0.65	11.5	--	--	2.50	3.75	4.25	Bal	--	--	104
Ditto	0.75	13.5	--	--	3.00	4.25	4.75	Bal	--	--	104
"	0.70	12.5	--	--	2.75	4.00	4.50	Bal	--	--	104

A common method of applying hard-facing materials is by means of the oxyacetylene torch. The method allows good control of the operation and produces a smooth deposit (Ref. 101). All the hard-facing materials, including tungsten carbide, can be applied with the oxyacetylene technique (Ref. 103). In this method of application, the alloys are generally used in the form of bare rods or flux-coated electrodes; tungsten carbide usually is supplied in the form of a powder encased in a thin tube for ease of application (Ref. 103).

The metal-arc welding process, using either bare rods or flux-coated electrodes, is another means for applying hard facings. This process is well adapted to the production of thin, smooth layers on thick base metals (Ref. 101). The atomic-hydrogen arc welding process, as well as the tungsten-arc inert-gas (TIG) method, can also be used to apply hard-facing materials (Refs. 101, 105). These techniques are analogous to the oxyacetylene method except that higher deposition rates are obtainable because of the higher output of thermal energy generated by the arc (Ref. 101).

Other methods of application include metallizing, plasma-arc deposition, and flame or detonation plating. Metallizing guns are available that use wire or that take powder as the feed. The melting heat is usually supplied by an oxyacetylene flame, while compressed air atomizes the molten metal and projects it onto the surface of the base metal. The coating tends to be somewhat porous and, unless given a subsequent fusion treatment, is only mechanically bonded to the substrate (Ref. 101).

In the plasma-arc process, an inert gas flows through an arc established between a tungsten electrode and the water-cooled copper nozzle of the welding gun. As the gas passes through the arc it is ionized and becomes a plasma. The process of ionization raises the temperature of the gas to levels as high as 20,000 F and higher causing it to issue from the gun in a stream or jet at supersonic velocities. Powders introduced into the gas stream are melted and sprayed onto the work surface. A plasma-arc spray gun is illustrated schematically in Figure 30.

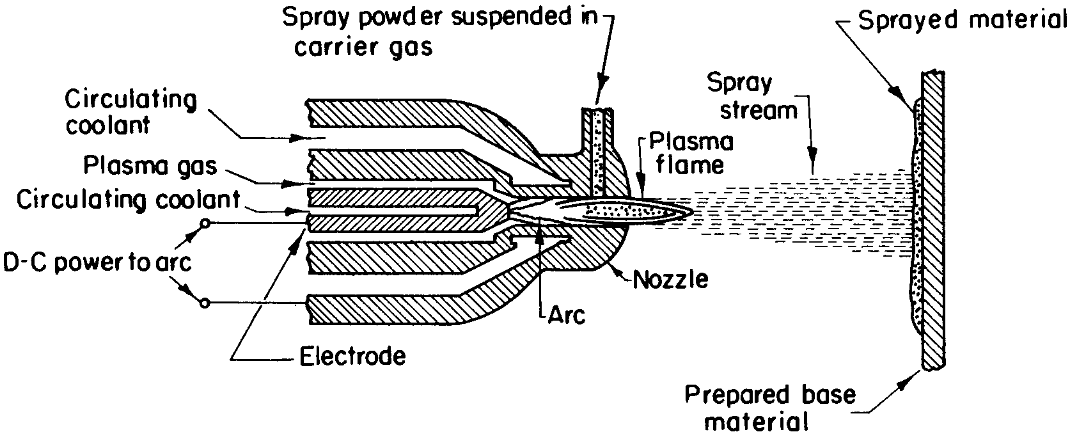


FIGURE 30. SCHEMATIC DIAGRAM OF A PLASMA-ARC SPRAY GUN (REF. 106)

The flame-plating process makes use of controlled detonation. An oxyacetylene mixture is exploded in a length of tubing that is closed at one end, heating and driving a "charge" of hard-facing powder at the work. As the powder is accelerated toward the work it melts and is blasted into the surface of the workpiece forming a dense, adherent, well-bonded coating. After detonation, the tube is purged with nitrogen, reloaded, and exploded. This cycle is repeated several times a second. The flame-plating process has been used successfully to coat Waspaloy and Udimet 700 with tungsten carbide particles, the matrix being Nichrome (Ref. 105). A schematic diagram of the process is shown in Figure 31.

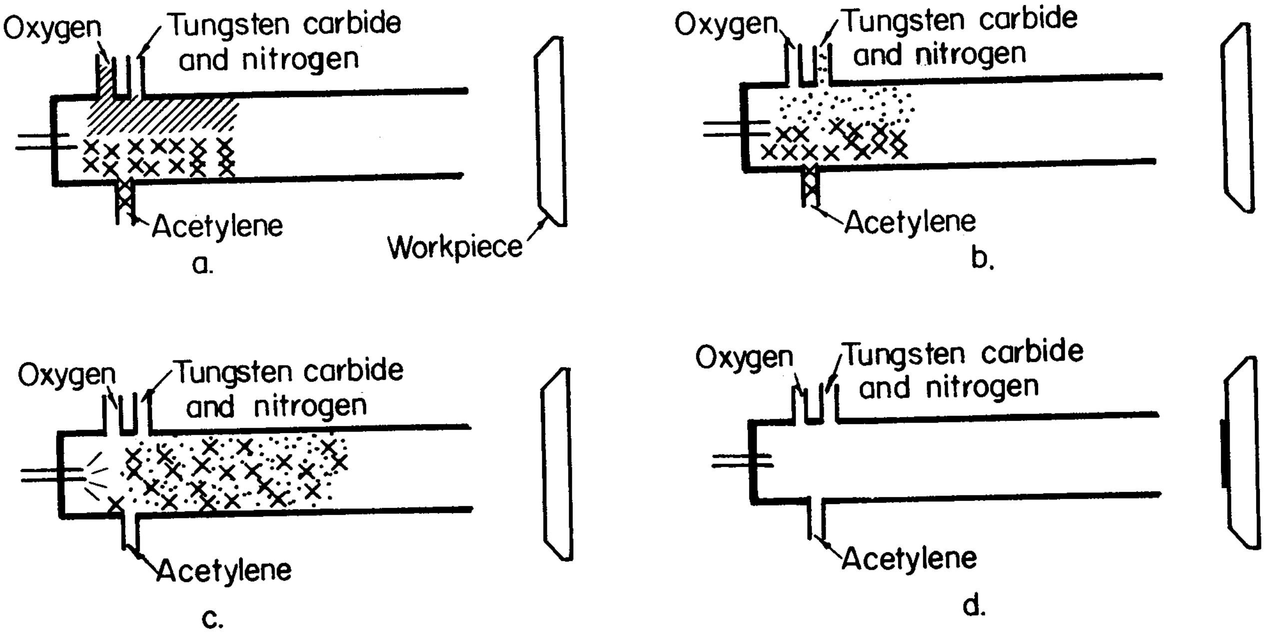


FIGURE 31. FLAME-PLATING PROCESS IN DIAGRAMMATIC FORM (REF. 107)

Note: The workpiece is held in the jaws of a chuck mounted on a saddle that can be traversed along a bed at right angles to the line of fire. This enables the work to be turned so that the whole of the area to be provided with a hard surface can be covered step by step.

(a) Oxygen and acetylene are mixed in the breech chamber on the left.
(b) A carefully measured quantity of tungsten carbide is dispersed into the breech.
(c) The gas mixture is detonated by a spark, raising the temperature of the powder and projecting it down the barrel of the gun at over 2,500 ft/sec.
(d) The plasticized tungsten carbide embeds itself in the surface of the workpiece and becomes welded to it.

Nickel-base alloys are not often hard faced. However, on occasion hard facings are applied to wrought nickel, Monel 400, and Inconel 600 as well as a number of the nickel-base superalloys. Nickel and Monel 400 are readily hard faced with cobalt-base and nickel-base hard-facing alloys and also with tungsten carbide. The oxyacetylene torch and the tungsten-arc inert-gas methods are the preferred techniques for applying hard-facing materials to these alloys (Ref. 102).

One reason for hard facing nickel-base alloys is to improve their resistance to steam erosion. To this end, valve components made of Inconel 600 are hard faced with a cobalt-base alloy corresponding to the sixth alloy listed in Table XXIX. The method of application is by means of the oxyacetylene torch (Ref. 108).

Another purpose in hard facing nickel alloys is to enhance their resistance to erosion-corrosion. An example is the hard facing of the flights on a Duranickel extrusion screw with a nickel-base alloy having a composition corresponding to the last alloy listed in Table XXIX. As shown in Figure 32, the gas torch was used to apply the hard facing. Duranickel is an age-hardenable alloy composed of 4.5 per cent aluminum with the balance being nickel. The alloy is hard surfaced in the age-hardened condition and, therefore, the torch flame must be so manipulated that the Duranickel surface does not exceed age-hardening temperatures. This is done by playing the torch in such a way that most of the heat enters the hard-facing alloy and little goes into the base metal. If surfacing were attempted on the metal in the annealed condition, the heat absorbed during the operation would age harden the surface, producing high stresses between this area and the annealed interior, which would lead to cracking (Ref. 109).

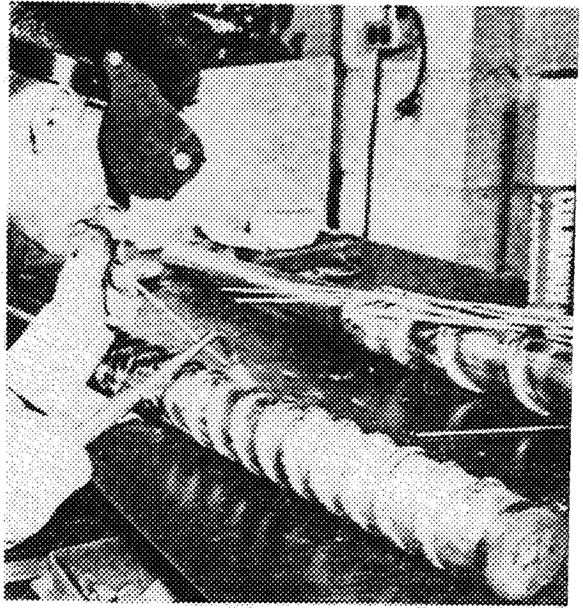


FIGURE 32. DURANICKEL EXTRUSION SCREW BEING HARD SURFACED WITH A NICKEL-BASE ALLOY (REF. 109)

Again, Monel K-500 piston rods for high-pressure oxygen compressors have been flame plated with tungsten carbide plus 9 per cent cobalt to improve their wear resistance (Ref. 110). The coating applied was 0.010 inch thick, ground and lapped to a finish of 5 to 10 microinches rms. The rods operated through glass-filled Teflon packing glands, and their average life was increased ten times by the coating treatment.

SURFACE TREATMENT FOR PURPOSES OF LUBRICATION

During the past few years, an increasing number of applications has emerged in which lubrication is required under conditions wherein oils and greases are unsuitable. Such conditions may feature temperatures high enough to cause organic lubricants to decompose; most oils and greases suffer considerable deterioration at temperatures of 500 or 600 F and above. Again, the environment may be unacceptably corrosive to organic lubricants, or the pressure may be so low as to result in serious losses due to volatilization (Ref. 111).

Furnace conveyor belts have been cited as examples of an application requiring lubrication at high temperatures and often in reactive atmospheres. As another illustration, lubricants for the control surface pivots on suspersonic aircraft are subjected to high temperatures developed by aerodynamic heating. Similar components on spacecraft are subject to the vacuum of space when the craft is on its mission and to extremely hot oxidizing conditions during reentry into the atmosphere. In addition, lubricants may be called on to be radiation resistant as well as to perform successfully in the presence of strongly reactive liquids and gases (Ref. 111).

Thus, under conditions that cause oils and greases to deteriorate, some other material must be used as the lubricating film between metal surfaces in sliding contact with each other, when reduction in frictional forces and resistance to seizing and galling are required. Films with lubricating qualities can be formed from oxides, halides, sulfides, and certain metals. During the past several years, the Lewis Research Center of NASA has been particularly active in research on lubricating materials designed to function in environments destructive to organic lubricants. In the paragraphs that follow, a number of investigations in this field, all undertaken by NASA-Lewis, are summarized.

In an investigation begun in the mid-1950's, it was observed that pure lead monoxide bonded to one of the two contacting members provided effective dry-film lubrication at elevated temperatures (Ref. 112). The method of applying the coatings generally used in this research was to dust a thin layer (about 1/16 inch thick) of the powdered oxide onto the surface of the metal, heat to a sufficiently high temperature to melt the oxide and cause it to form a uniform film on the surface, then cool in still air to room temperature. The coefficient of friction between cast Inconel X-750 and M-1 tool steel in sliding contact was maintained well below 0.2 at temperatures up to 800 F when the Inconel X-750 was coated with lead monoxide. The same coating was equally effective to a temperature of 1250 F when both members were cast Inconel X-750; however, the wear rate was quite high under these conditions.

It was also learned that small additions of iron oxide promoted adherence to the substrate, while the inclusion of silica in the formulation inhibited oxidation of the lead monoxide. With coatings of this type, there was a strong tendency for the coefficient of friction to decrease as the temperature increased into the range of 800 to 1000 F, and as sliding velocity increased from 6 to 10,000 feet per minute. Coefficients of friction as low as 0.05 to 0.08 were obtained at 1000 F and 2400 feet per minute sliding velocity (Ref. 113).

In another investigation, several ceramic coatings, diffusion-bonded fluoride coatings, and ceramic-bonded fluoride coatings were evaluated for use as dry-film lubricants at temperatures up to 1500 F (Ref. 114). Of the coatings investigated, the most effective overall lubricating qualities were obtained from a ceramic-bonded calcium fluoride coating applied to a cast Inconel X-750 substrate. The optimum formulation was 75 per cent calcium fluoride and 25 per cent ceramic binder, the composition of the latter being 60 per cent cobalt oxide plus 20 per cent barium oxide plus 20 per cent boric oxide. This coating gave coefficients of friction of 0.26 at 75 F, 0.20 at 500 F, and 0.15 at 1500 F under the prevailing test conditions. The addition of a thin overlay of pure calcium fluoride to the ceramic-bonded calcium fluoride coating reduced the coefficient of friction at 1500 F to 0.06; however, the overlay had no effect in the temperature range of 75 to 1350 F.

Further research on ceramic-bonded calcium fluoride coatings indicated that preoxidizing the Inconel X-750 substrate, before spraying on and firing the coating, improved coating wear life without detriment to the coefficient of friction (Ref. 115). Preoxidation together with the use of a burnished and sintered pure calcium fluoride overlay further increased coating life, but at some sacrifice of the coefficient of friction. The thickness of the overlay was observed to be important, preferably being not more than 0.0005 inch. Variation in sliding velocity between 430 and 5000 feet per minute had little effect at 1000 F or 1500 F with preoxidized and overlayed material. However, at 75 F the friction coefficient decreased from 0.32 to 0.19 as the sliding velocity was increased from 430 to 5200 feet per minute. Finally, it was observed that the substrate played an important role. For example, cast Inconel 600 sliding against coated Inconel X-750 was lubricated effectively to 1500 F, but at 1600 F the coating blistered. On the other hand, good lubrication and coating adherence were obtained for René 41 sliding against coated René 41 at temperatures up to 1900 F.

In later research, it was found that the reliability of ceramic-bonded calcium fluoride coatings was strongly influenced by the details of the coating application procedure (Ref. 116). Best results were obtained with prolonged wet pebble milling of the fluoride-ceramic mix, the use of a very finely atomized mist when spraying the coating on the substrate, and careful control of firing time and temperature. Additions of 1 to 3 per cent molybdic oxide to the coating formulation were beneficial. At temperatures from 1000 to 1600 F, the use of a rubbed overlay composed of 62 per cent barium fluoride and 38 per cent calcium fluoride (the binary eutectic), instead of pure calcium fluoride, resulted in considerably improved performance. Much better lubrication was obtained at 1000 F and lower temperatures when the material was run-in first at 1500 F.

Molybdenum disulfide and tungsten disulfide have been found to be effective high-temperature lubricants when used in powdered form. Both compounds were observed to give low friction coefficients in an argon atmosphere at temperatures up to 1500 F (Ref. 111). However, they appear to be somewhat limited in an air environment, molybdenum disulfide being effective up to about 800 F and tungsten disulfide up to about 1000 F.

Still another type of material that is capable of performing as a dry-film lubricant is a metal, and research on metallic film lubricants has been in progress at NASA-Lewis (Ref. 117). Thin gold films were vapor deposited on nickel, nickel-chromium, and nickel-rhenium surfaces. Their lubricating characteristics and durability were studied by running them against niobium in an ultrahigh vacuum of approximately 10^{-11} millimeter of mercury (torr). A successful lubricant of this type might find ready application in hardware designed for use in outer space. The results of the study confirmed the importance of a strong bond between the film and the substrate. Film-endurance life during friction experiments was increased when the substrate was electron bombarded and thermally etched prior to vapor deposition on the etched surface at an elevated temperature of 800 F, under which conditions a diffusion-type bond was obtained between the film and the substrate.

For additional information on dry-film lubrication, attention is called to Reference 118.

MECHANICAL TREATMENTS

Mechanical surface treatments are used either to (1) improve the finish of the surface of an article, (2) establish a compressive state of stress there, or (3) cold work the material so as to induce recrystallization in localized areas during subsequent solution treatment. Burnishing and planishing are used for improving surface finish, while explosive hardening, peening, and planishing are used to cold work the metal and/or to develop residual compressive stresses at the surface. Such work and stresses may serve to increase fatigue strength, increase surface hardness, and reduce the occurrence of cracking in welds.

BURNISHING

In burnishing, a smooth, glassy, mirror-like finish is developed on the surfaces of metal parts by frictional contact either between the parts themselves or between the parts and other pieces of material known in the trade as the medium. The operation is usually carried out in rotating barrels, shaker containers, or vibrating tubs (Refs. 119, 120).

In true burnishing, the surfaces of the workpieces are compressed and kneaded, minute high spots being pushed down, and other irregularities, such as tiny pits and scratches, being smoothed out and blended by lateral flow of the metal (Ref. 121). No abrasion or other form of metal removal takes place. By eliminating superficial irregularities a high luster is developed. In fact, the process is sometimes called lustering or coloring.

Sometimes the work is polished and burnished at the same time. Because polishing involves metal removal by abrasive action, the operation is often referred to as abrasive burnishing. The degree of metal removal in this operation can be varied from small amounts to almost none. Abrasive burnishing generally does not develop as high a luster as that produced by true burnishing.

Burnishing operations can be performed dry; however, it is usual practice to carry them out in water. The use of water has several advantages. For example, water tends to act as a cushion reducing the chance that parts will be damaged from battering as they tumble and slide in the container. In addition, water offers lubricating qualities, aids in controlling the fluidity of the mass, and tends to keep dirt away from the metallic surfaces by holding it in suspension. A particularly important function of the water is to act as the solvent and vehicle for additives introduced to increase lubrication, provide cleaning action, control pH, or help to brighten the work.

One of the most satisfactory burnishing mediums consists of hardened steel balls of various sizes, which are usually mixed with other hardened steel shapes specially designed for the purpose. The sizes and shapes of the pieces comprising the medium are selected to insure that contact will be made with all the places on the workpiece requiring burnishing, without lodging in holes and recesses, or causing the mass to jam together and become rigid (Ref. 120). Common nonspherical shapes used in burnishing mediums are ball-cones, grains, disks, diagonals, ovals, and pins.

Other materials used for burnishing mediums include leather, glass beads, corn cobs, sawdust, and zinc slugs. Mediums used for abrasive burnishing include glazed natural stones (such as granite, flint, or agate), shapes made of fused alumina, and glass pellets (Ref. 120).

The additives that are introduced by way of the water used in the operation and serve to supplement the action of the medium are known in the trade as compounds. Tallow-base soap is a favored burnishing compound because it produces long lasting suds that increase luster, provide cleaning action, and act as a cushion. Borax is sometimes added with the soap to aid in cleaning and to control pH (Ref. 120). Many compounds of complex chemistry, developed for particular applications, are in use today. Synthetic wetting agents, together with water softeners when needed, are also added to the water (Ref. 122). In general, the make-up of the burnishing compound is varied depending on the metal being burnished and on the size, shape, and surface condition of the part.

Nickel and nickel alloys can be burnished in a manner similar to that of other metals and alloys. Turbine blades constitute an example of a nickel-base-alloy part that is often finished by burnishing (Ref. 123). In establishing conditions for burnishing nickel alloys, account must be taken of the fact that most of them work harden very rapidly (Ref. 122).

EXPLOSIVE HARDENING

The high-intensity shock waves that can be generated by explosives are capable of deforming metallic materials and are, in fact, being used commercially to form a considerable variety of metallic components. The explosive-forming operation is usually carried out with the metal workpiece at room temperature. Under these circumstances, the effect of the process on mechanical properties is quite similar to that of other cold-forming operations. Thus, metals and alloys that work harden when cold formed will also harden when explosively formed. Nickel and nickel alloys are in this category.

It is also possible to harden work-hardenable metals and alloys by the passage of explosive shock waves without deforming the material appreciably. The hardness level reached and the depth of the hardening effect depend on the metallurgical characteristics and the initial properties of the material as well as on the nature of the impulse delivered to the material. The latter factor is subject to considerable control. When hardening rather than forming is the primary objective, the operation is known as explosive hardening as distinguished from explosive forming.

The major industrial use of explosive hardening thus far has been with Hadfield's manganese steel, which is an austenitic steel containing some 13 per cent manganese and 1.25 per cent carbon. This type of steel finds extensive use in applications where parts are subject to impacting and heavy abrasion. Examples include railroad frogs, jaws for rock crushers, grinding mill liners, and dipper teeth for power shovels. Such applications involve severe battering and deformation of the parts with the result that they work harden and, as a consequence, more effectively resist further deformation and wear. In many applications, however, the work hardening is only a surface effect and, in most applications, a significant increase in the life of the part could be obtained if it were placed in service already deeply hardened. Conventional methods for deeply hardening this type of steel are expensive and often unsuccessful in realizing the full potential of the material. Du Pont has been a leader in the field and has worked out procedures for deep hardening austenitic manganese steels by detonating an explosive charge in contact with the metal. In a favored method of explosive hardening, the explosive is used in the form of thin plastic sheets that are placed in contact with the surface to be hardened (Ref. 124).

In some cases, only a small area of the component is to be hardened; in other instances, the area may be quite extensive. Again, in some applications, the part is impacted one, two, or even three times to develop the desired hardness and minimize the chance of spalling (Ref. 124).

Generally, when an explosive charge is detonated in contact with a work-hardenable metal the yield strength and the tensile strength of the metal are increased while the ductility is decreased. These effects are dependent on the magnitude and the duration of the explosive impulse. The data in Table XXX show how the tensile properties and hardness of wrought nickel are affected by the pressure of the impulse (Ref. 125).

TABLE XXX. EFFECT OF IMPULSE PRESSURE ON THE MECHANICAL PROPERTIES OF NICKEL (REF. 125)

Pressure, psi	Vickers Hardness, 1-kg load	Yield Strength, psi	Tensile Strength, psi	Elongation in 1 Inch, per cent
0	125	34,000	60,500	37
1.5×10^6	183	59,000	63,500	13
3.9×10^6	208	85,000	87,000	10
6.8×10^6	263	130,000	132,000	7
Cold rolled 27 per cent	210	83,000	89,000	7
Cold rolled 81 per cent	275	91,500	96,000	--

Some properties of cold-rolled nickel are included in the table for purposes of comparison. The data suggest that a higher ratio of ductility to strength can be obtained by explosive hardening than by cold rolling. Thus, it may be surmised that work hardening by means of explosive shock waves differs in some important respects from work hardening by rolling.

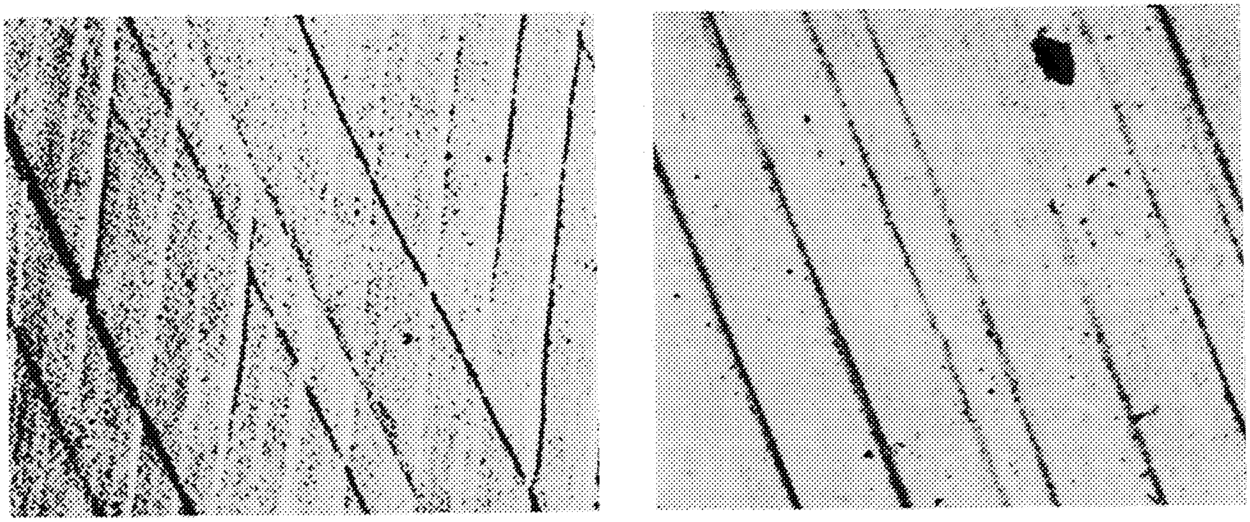
That such is the case for wrought nickel is borne out by an investigation of microstructural effects produced in this metal by explosive shock loading (Ref. 125). For example, both explosive shock loading and static loading produce slip lines in nickel. However, while the slip-band spacing developed by static loading decreases with increasing strain in the expected manner, the average slip-band spacing obtained in shock loading is essentially independent of pressure. The results shown in Table XXXI are illustrative (Ref. 125).

TABLE XXXI. AVERAGE SLIP-BAND SPACING IN NICKEL (REF. 125)

Type of Deformation	True Strain, in./in.	Vickers Hardness	Average Slip-Band Spacing, cm
Low-speed compression (static deformation)	0.01	128	5.0×10^{-4}
	0.05	145	2.2×10^{-4}
	0.13	180	1.7×10^{-4}
	0.47	260	1.0×10^{-4}
Shock compression (dynamic deformation)	0.07 (100 kbars)	183	3.0×10^{-4}
	0.14 (265 kbars)	208	2.4×10^{-4}
	0.18 (360 kbars)	242	2.3×10^{-4}
	0.22 (460 kbars)	263	2.9×10^{-4}

Additional information was revealed from electron micrographs of the slip systems developed on the surface of specimens polished and lightly etched before hardening. Figure 33a shows the slip line developed in nickel by slowly compressing the metal to a Vickers hardness of 180. Two slip systems are evident, the height of the slip step in the primary system being nearly constant. Figure 33b shows the structure produced by hardening with a 100 kilobar (1.5×10^6 psi) shock wave to a Vickers hardness of 183. There is no multiple slip in this system. There are indications, however, that the slip bands consist of elementary steps and that fragmentation of the slip lines has occurred; fragmentation is indicative of cross slip. Figure 34a shows the slip-line structure in a nickel specimen statically deformed to a hardness of 260 Vickers. Multiple slip and cross slip are evident. Figure 34b shows the slip lines produced by a 460 kilobar (6.8×10^6 psi) shock wave that increased the hardness to 263 VHN. Slip-band fragmentation has taken place and the height of the slip steps appears to be greater than in the previous photomicrographs.

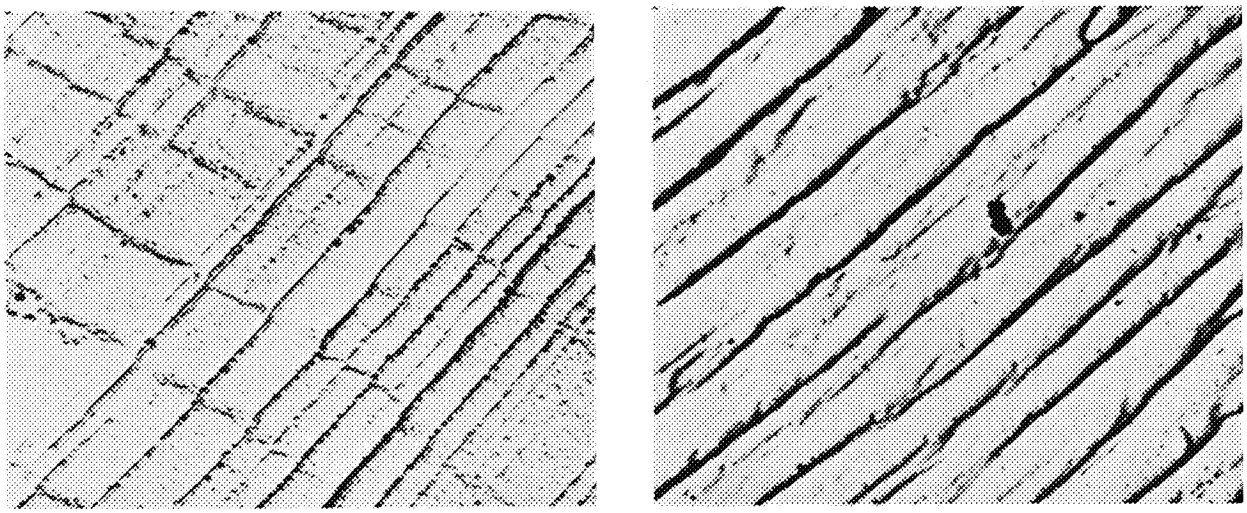
It was concluded that the chief difference between nickel hardened by slow compression and by explosive shock loading is in the finer slip-band spacing of the statically hardened material. For low deformation speeds, extensive grain rotation is possible at high strains. This situation favors slip on secondary systems and leads to an



10,000X
a. Static Deformation VHN 180

10,000X
b. Shock Deformation VHN 183

FIGURE 33. ELECTRON MICROGRAPHS OF SLIP LINES IN NICKEL (REF. 125)



10,000X
a. Static Deformation VHN 260

10,000X
b. Shock Deformation VHN 263

FIGURE 34. ELECTRON MICROGRAPHS OF SLIP LINES IN NICKEL (REF. 125)

increasing slip-band density with increasing strain. With shock loading, there is no opportunity for grain rotation and slip on secondary systems is less likely. In general, the slip bands formed by static deformation are sharper and less fragmented than those produced by shock loading. When compared on a basis of equal hardness the explosively hardened sample has a lower density of slip bands, greater step height, and more cross slip.

In addition to nickel, the effect of explosive impacting on the mechanical properties of a number of nickel-base alloys has come under study. In one investigation, bare high-explosive charges were used against 1/2 x 1 x 12-in. test pieces (Ref. 126). The shock was described as being oblique and the estimated peak pressure was 200 kilobars (3.0×10^6 psi). The pressure pulse duration was estimated to be approximately 12 microseconds. The composition and condition of the nickel alloys investigated are given in Table XXXII.

The thickness of the specimens was measured before and after firing, and it was found that the material (originally 1/2 inch thick) had experienced a slight amount of compression. The Inconel 600 showed the least dimensional change, namely, 0.004 inch; while the wrought nickel (Nickel 200) had been compressed 0.010 inch, which was the greatest dimensional change among the nickel-base alloys.

Microhardness surveys consisting of at least 12 individual measurements were made across the thickness of the specimens. The hardness was reported to be quite uniform and, therefore, only the average values were recorded, as shown in Table XXXIII. Tensile and impact properties measured before and after firing are tabulated in Table XXXIV. It can be seen that tensile and yield strengths increase, while ductility and toughness decrease as a result of the explosive impaction.

These studies clearly demonstrate that nickel and nickel-base alloys can be hardened by the passage of explosive shock waves. However, the process as applied to these alloys is not known to be in commercial use. A possible application for the process is to increase the strength of weldments in certain cases where a postweld heat treatment, such as an age-hardening treatment, cannot be done, or where the weldment cannot be planished or otherwise cold worked for strengthening purposes. However, strengthening by explosive hardening is subject to a number of the limitations of other work-hardening processes. For example, the effects diminish and finally disappear as the service temperature approaches and exceeds the stress-relieving temperature range.

TABLE XXXIV. TENSILE AND CHARPY V-NOTCH PROPERTIES OF NICKEL-BASE ALLOYS BEFORE AND AFTER FIRING TESTS (REF. 126)

Material	Tensile Strength, 1000 psi		Yield Strength 0.2% Offset, 1000 psi		Elongation in 1 Inch, per cent		Reduction of Area, per cent		Charpy V-Notch Value, foot-pounds	
	Before	After	Before	After	Before	After	Before	After	Before	After
Nickel 200	71.1	93.1	--	--	40	13	78.5	69	240+(a)	151
Monel 403	81.5	105.2	45.3	102.8	38	17.5	78.1	77.5	240+(a)	192
Monel R-405	93.2	117.8	51.8	114.8	37	15	67.8	65	240+(a)	84.5
Monel 400	84.55	110.3	--	--	37	11.5	74	66	240+(a)	157.5
Inconel 600	103.4	123.1	--	114.9	36	17	67	64.5	215.5	101.5
Hastelloy B	122.5	143.0	51.9	116.6	59.5	34.5	66	62	159	75.5
Hastelloy C	116.0	137.2	52.1	117.5	49.5	28	53	46.7	95	31
Hastelloy W	117.8	135.8	50.8	108.5	56	40	62.8	59.5	165	75.5

(a) These specimens were not broken in the test; the pendulum of the machine was stopped by the specimen.

PEENING

General Discussion. Generically, peening refers to the cold forming of metals by mechanically working them with hammer blows. Thus, in this elementary sense, it is an operation that a metalsmith frequently uses in shaping a metal artifact. However, the craftsman also knows that by peening in a certain way he can often improve the article's resistance to cracking and breaking, especially under conditions of repeated or cyclical loading. To effect this improvement in performance, the smith strikes many light, quick blows against the metal that is essentially at room temperature, the blows being too light to bring about an appreciable change of shape in the article. Under these conditions the craftsman is working only the surface of the metal (Ref. 127).

In fact, peening is most often thought of today as the mechanical working of the surface of a metal by hammer blows or by the impingement of shot. Carried out in this manner, peening causes plastic flow in the surface of the work, tending to increase surface area; however, this action is resisted and restrained by the unworked interior. As a result of the restraint, compressive stresses tend to build up in the peened surface layers of the object, while counterbalancing tensile stresses tend to develop in the interior (Refs. 128, 129). It is largely the capability of peening to develop residual compressive stresses at the surface of a metal that makes it a useful metallurgical operation.

Hammer peening is carried out manually by means of hand hammers or mechanically with pneumatic and other power hammers or with equipment, such as swaging machinery, that acts in a similar manner (Ref. 130). Mechanical hammer peening is often not convenient to apply; manual hammer peening is more adaptable and usually cheaper. However, hammer peening in general is difficult to control. Shot peening is much more amenable to control; it is a flexible operation and is quite widely used in a variety of applications.

Shot peening is usually done in a cabinet in order to confine the shot, contain dust, and aid in the collection of the shot for reuse. The work to be peened is introduced and supported mechanically. Various materials are used for shot; cast iron and steel shot are generally used to peen workpieces made of steel. For nonferrous work in which contamination of the surface with iron from the shot is unacceptable, other materials are used; shot composed of glass beads is a frequent choice. Usual shot sizes range from 1/64 to 3/32 inch in diameter. The shot may be driven against the work by an air blast

TABLE XXXII. COMPOSITION AND CONDITION OF EXPLOSIVELY HARDENED NICKEL-BASE ALLOYS (REF. 126)

Designation	Nominal Composition, weight per cent									Condition Before Test
	C	Co	Cr	Cu	Fe	Mn	Mo	Ni	Other	
Nickel 200	0.08	--	--	0.04	0.50	0.23	--	Bal	--	Vacuum annealed 2-1/2 hr at 1250 F, F.C.
Monel 403	0.0725	--	--	39.52	0.11	1.77	--	Bal	--	Hot finished
Monel R-405	0.135	--	--	30.91	1.91	1.09	--	Bal	0.050S	Hot finished
Monel 400	0.15	--	--	29.90	2.0	1.04	--	Bal	--	Vacuum annealed 3 hr at 1400 F, F.C.
Inconel 600	0.05	--	15.64	--	6.82	0.20	--	Bal	--	Vacuum annealed 3 hr at 1600 F, F.C.
Hastelloy B	0.012	0.17	0.04	--	4.66	0.24	27.40	Bal	0.24V	Solution annealed
Hastelloy C	0.07	2.21	14.64	--	5.50	0.57	16.78	Bal	0.25V, 3.75W	Solution annealed
Hastelloy W	0.022	0.55	5.49	--	5.26	0.68	25.30	Bal	--	Solution annealed

TABLE XXXIII. DIAMOND PYRAMID HARDNESS OF NICKEL ALLOYS BEFORE AND AFTER FIRING TESTS (REF. 126)

Material	Hardness Before Test	Hardness After Test
Nickel 200	101[(a)]	223[(a)]
Monel 403	151	234[(a)]
Monel R-405	181	273[(a)]
Monel 400	139[(a)]	241[(a)]
Inconel 600	168[(a)]	278[(a)]
Hastelloy B	199	308
Hastelloy C	204	316
Hastelloy W	191	304[(a)]

(a) 5-kg load used, all other measurements made with a 10-kg load.

or by the centrifugal force generated by a rapidly rotating, radially vaned wheel into whose hub region the shot is fed for distribution to the work by means of the vanes.

The important factors in shot peening are the magnitude of the compressive stresses developed in the surface layers of the metal, the depth of the effect, and the coverage or uniformity with which the peening operation is carried out over the surface of the metal. Stress and depth are strongly influenced by the characteristics of the material being peened. In addition, stress, depth, and coverage are influenced by such variables as the velocity, size, hardness, and shape of the shot; the amount of shot striking the work; the shape and size of the area covered by the shot stream; and the angle with which the shot impinges on the work. These variables are subject to control. Optimum conditions and settings for any given application are determined by experimentation. Further information on shot-peening procedures is given in References 127-129 as well as in numerous other publications.

Applications for which peening has been used include the following:

(1) Improvement of fatigue strength. The residual compressive stresses built up at the surface are considered largely responsible for the beneficial effect of peening on fatigue properties. This is the major application for shot peening.

(2) Correction of distortion in weldments and in unwelded components. In some cases, this may be brought about through considerable plastic flow of the metal; more commonly, distortion is corrected by the change in the internal stress state of the metal produced by the peening operation.

(3) Prevention of cracking in weldments. The objective is to counteract the tensile stresses developed in the joint by the shrinkage that occurs on cooling after welding.

(4) Straightening components. Again, this may be done by plastically deforming the metal (essentially cold forming) or by altering the internal stress state of the component.

(5) Improvement in resistance to stress-corrosion cracking. The objective is to overcome the surface tensile stresses necessary for stress-corrosion cracking by introducing compressive stresses through the peening operation. For this kind of application, shot peening is the method usually employed.

(6) Closing up of porosity in castings and weldments.

(7) Forming of parts. Shot peening is quite well adapted to mild bending and contouring operations on large expanses of relatively thin gage material. The changes in curvature result from the action of the internal stress system produced by the peening.

(8) Promotion of recrystallization. It is often possible to introduce enough cold work into welds and weld-heat-affected zones, as well as into wrought components, by peening to cause the metal to recrystallize when subsequently heated to an annealing temperature. Recrystallization of welds may result in improved ductility and toughness.

Nickel and Nickel-Base Alloys. Peening is not commonly employed with nickel and its alloys and the literature on the subject is meager. On the other hand, these materials do not appear to have characteristics that prevent the use of peening.

Basically, metals and alloys that harden when cold worked are affected by peening, in that they are capable of retaining residual stresses and, therefore, give the peening operation an opportunity to produce the all-important residual compressive stresses at the surface. Nickel and nickel-base alloys are in this category. In other materials, such as lead, stress relief or annealing occurs simultaneously with working at room temperature and, hence, no appreciable internal stresses are retained.

Springs and other items made from nickel-base alloys, which are exposed to fatigue conditions, could be shot peened if desired. However, many of the more complex nickel-base alloys work harden with great rapidity and, therefore, the depth of the effect produced in these alloys would be expected to be quite shallow.

The effect of shot peening diminishes and finally disappears as service temperatures increase. At and above stress-relieving temperatures the effect is lost. Thus, there is no advantage in shot peening parts destined for high-temperature applications in an effort to increase their fatigue strength. On the other hand, some benefit to fatigue resistance may be derived from shot peening when the service temperature is below that at which appreciable stress relief takes place.

No basic factor is known to exist that would militate against the use of shot peening to straighten nickel and nickel-alloy parts, or correct distortion in weldments of these materials, or yet to form them by methods similar to those used for aluminum and other metallic materials. Of course, because the characteristics of the material being peened exert a strong influence, peening operations would require suitable modification to adapt them to the particular materials under consideration.

It has been shown that hammer peening can be used to prevent cracking of repair welds made in René 41 and in Astroloy on subsequent reheat treatment (Ref. 131). These and a number of other age-hardenable nickel-base alloys are susceptible to cracking, especially along the fusion line, when repair welded and reheat treated. The material studied ranged in thickness from 0.030 to 0.075 inch, while the inert-gas-shield tungsten-arc method was the welding process that was used. Hammer peening was reportedly very successful, and it can be speculated that shot peening could also have been employed.

PLANISHING

Planishing is defined as the production of a smooth surface finish on metal by a rapid succession of blows delivered by highly polished dies or specially designed polished hammers, or by rolling in a planishing mill (Ref. 119). Any readily workable metal or alloy, such as wrought nickel and most nickel-base alloys, can be planished.

Frequently, planishing equipment is used not only for surface finishing but also for certain metal-forming operations. A common operation applied to sheet metal, which can often be carried out with a planishing hammer, in the process of dishing. Annealed Inconel X-750 sheet has been dished by means of a planishing hammer with great success (Ref. 132).

Several types of planishing mills have been developed. One type of roll planishing mill, used to planish fusion seam welds, consists of a heavy base that supports a deep-throated superstructure. The mill is shown in Figure 35. The superstructure is composed of twin horizontal members at the ends of which the planishing rolls are mounted. The rolls are so positioned that they can be forced together. The upper roll is power driven and serves to drag the work through the rolls, while the lower roll is an idler. The relationship of the rolls to the work is shown in Figure 36. Loads up to 50 tons can be applied by the rolls (Ref. 133).

Roll planishing has been found to be a useful mechanical treatment for fusion welds, and especially butt welds. The compressing or upsetting action that takes place in planishing forces protrusions and high spots of weld metal down into the weld. By this process the weld is smoothed and its contour is blended into that of the parent metal. In this way, planishing helps to streamline structures, and improve their aerodynamic characteristics and their appearance.

Another application for roll planishing is in producing seam-welded tubular and other components to close tolerances. Here, advantage is taken of the fact that, as the planishing operation reduces the thickness of a weld, it spreads the weld bead laterally at the same time. In the case of a tubular or conical component, for example, the part is made slightly undersize from sheet that has been roll formed and joined by means of a longitudinal butt weld. The weld seam is then run through the planisher one or more times to widen the weld just enough to bring the component up to the desired diameter.

Since planishing is actually a type of cold-forming operation, a roll-planished weld is effectively cold worked. The degree of working depends on the amount of deformation taking place during the planishing process. With nickel and nickel-base alloys that work harden readily, the degree of working during roll planishing can be such as to effect a considerable increase in the strength of the joint (Ref. 134).

Likewise, by means of roll planishing, it may be possible to impart sufficient cold work to a weld for it to recrystallize when subsequently heated to an annealing or solution-treating temperature. In this way, the weld takes on the grain structure of a wrought material and becomes more homogeneous. As a result, its mechanical properties tend to more nearly match those of the wrought parent metal than is the case when the weld is not worked. Fusion butt welds made in sheet-metal components formed from Inconel 600 and

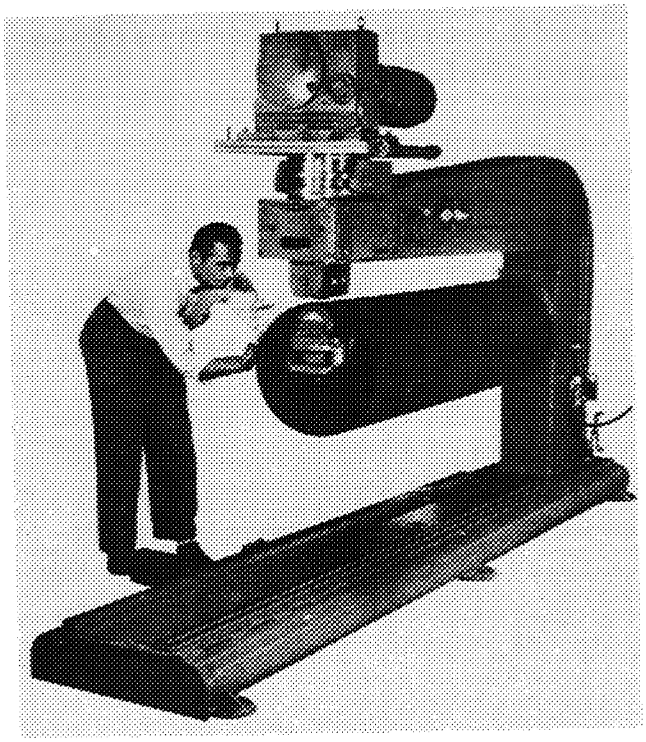


FIGURE 35. MACHINE FOR ROLL PLANISHING RESISTANCE-WELDED SEAMS

Courtesy of Airline Welding and Engineering Company, Gardena, California.

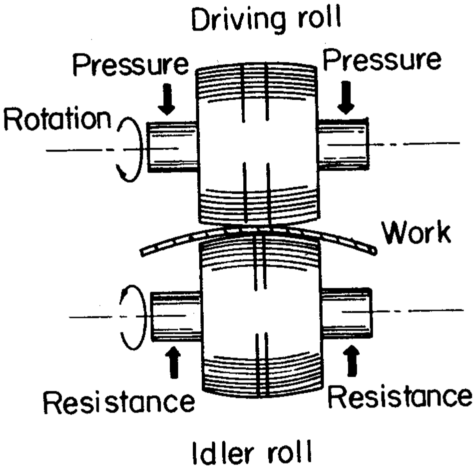


FIGURE 36. RELATIONSHIP OF THE PLANISHING ROLLS TO THE WORK (REF. 134)

718 have been roll planished so they would recrystallize on subsequent heating to solution-treating temperatures (Ref. 135).

RECOMMENDATIONS

The primary problem in regard to surface treatments for nickel and nickel-base alloys is the unavailability of a coating to protect nickel-base alloys from sulfidation at the service temperatures of advanced gas turbines operating in regions where salt air is present. As a consequence of the lack of such a coating, a number of failures have occurred in operational military aircraft in Viet Nam and elsewhere, and overhaul and service schedules, which are very difficult to maintain in times of war, are necessary. For instance, one of the major uses of the limited supply of fresh water on aircraft carriers is the washing of salt condensates from the engines of jet aircraft prior to each mission.

Although research to develop improved coatings for such engines is presently in progress, it is recommended that additional research be initiated because of the importance of the problem.

It should be noted that the need for coatings for such applications will probably be a recurring problem; while coatings are being sought to satisfy the requirements of today's engines, designers are already working on even more advanced engines. For instance, it is understood that turbine-inlet temperatures of 2000 to 2200 F and turbine-vane temperatures of 2200 to 2400 F are envisioned in the supersonic transport (SST).

A second recommendation is that research be conducted on the boronizing of nickel-base alloys. As noted earlier, most of the research in this area to date has been done in the Soviet Union. Possible advantages of such coatings in addition to hardness should be investigated. The resistance of boronized nickel-base alloys to attack by specific corrosive environments would be one area worthy of study.

APPENDIX

LIST OF PERSONAL CONTACTS

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This document has also been reviewed and approved for technical accuracy.

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