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THE MAKING OF NICKEL AND NICKEL-ALLOY SHAPES BY CASTING, POWDER METALLURGY, ELECTROFORMING, CHEMICAL VAPOR DEPOSITION, AND METAL SPRAYING

By J. G. Kura, V. D. Barth, W. H. Safranek, E. T. Hall, H. McCurdy, and H. O. McIntire

Prepared Under the Supervision of the
Research Branch, Redstone Scientific Information Center
Directorate of Research and Development
U. S. Army Missile Command
Redstone Arsenal, Alabama

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ABSTRACT

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The state of the art of making nickel and nickel-alloy shapes by methods other than machining from wrought materials is reviewed. The methods covered are casting, powder metallurgy, electroforming, chemical vapor deposition, and metal spraying. Of these methods, casting and electroforming are the most extensively used for making final shapes of nickel or nickel alloys. Powder-metallurgy methods are being used to make pure nickel strip for special applications. Special alloys such as nickel dispersion strengthened with thorium oxide, Monel, and others are made from powders in the forms of strip and extrusions. Nickel-alloy shapes are readily made by powder-metallurgy methods, and it is expected that the recently increased availability of prealloyed powders may result in more use of this versatile process. Chemical vapor deposition has found little application in making nickel shapes since it has little to recommend it over electroforming. Metal spraying has been essentially limited to application of wear-resistant and/or corrosion- and oxidation-resistant coatings to other metals.

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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 making of nickel and nickel-alloy shapes by casting, powder metallurgy, and other processes is reviewed. In addition to casting and powder metallurgy, consideration was given to production of shapes by electroforming, chemical vapor deposition, and metal spraying. The report brings together information from a wide variety of sources for the purpose of making it available to those who can use it in industry, in the military, and in the aerospace field.

TABLE OF CONTENTS

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

TABLE OF CONTENTS
(Continued)

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS
(Continued)

LIST OF ILLUSTRATIONS
(Continued)

LIST OF ILLUSTRATIONS
(Continued)

LIST OF TABLES

LIST OF TABLES
(Continued)

LIST OF TABLES (Continued)

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SUMMARY

CASTINGS

Nickel-base-alloy castings are used in a wide variety of applications requiring corrosion resistance in liquid media, or oxidation resistance combined with good strength properties at elevated temperatures. In severe high-temperature applications, notably jet-engine vanes and blades, castings have proven to be superior to forgings because some of the best alloys are too difficult to forge and because the cast structure sometimes is more creep resistant than that of wrought alloys of the same composition.

To attain the maximum in strength properties at high temperatures, it is necessary to include in the alloys elements such as titanium, aluminum, and very small amounts of boron and zirconium. These elements are readily oxidized and lost from the melt, or combine with other elements that may be present as impurities, unless high-purity melting stock is used and precautions are taken to avoid contamination and oxidation of the melt. This group of alloys is almost exclusively melted and cast in vacuum; argon atmosphere is sometimes used in lieu of vacuum.

A large number of nickel-base alloys have been developed since the introduction of the jet engine in attempts to obtain the best high-temperature properties possible. Of these alloys, IN-100 and MAR-M200 have been developed most recently, and they appear to be the most creep resistant at temperatures over the range 1500 to 1900 F. The following stress-rupture values appear to be typical for both alloys:

100-Hour Stress Rupture, 10^3 psi				
1500 F	1600 F	1700 F	1800 F	1900 F
71	51	38	25	17

Compositions of these two alloys are:

	IN-100	MAR-M200
Chromium	8-11	8-10
Cobalt	13-17	9-11
Molybdenum	2-4	--
Tungsten	--	11.5-13.5
Titanium	4.5-5.5	1.75-2.25
Aluminum	5-6	4.75-5.25
Carbon	0.15-0.20	0.12-0.17
Manganese	0.5 maximum	0.2 maximum
Silicon	0.5 maximum	0.2 maximum
Iron	1.0 maximum	1.5 maximum
Boron	0.01-0.02	0.01-0.02
Zirconium	0.03-0.09	0.03-0.08
Vanadium	0.7-0.9	--
Columbium	--	0.75-1.25
Nickel	Balance	Balance

In addition to strength, other properties of concern for high-temperature applications include oxidation resistance, thermal-shock resistance, and impact strength and ductility. Therefore, the compositions having the highest stress-rupture values may not always be the best choice for a particular application. For increased oxidation resistance it may be necessary to use alloys containing more chromium than may be desirable for optimum strength.

Vacuum melting is usually done in induction-heated furnaces having capacities of 20 to 500 pounds. A double chamber with a vacuum lock permits casting in vacuum without opening the furnace to the atmosphere between heats. The furnace crucibles are usually preformed of alumina, magnesia, or zirconia.

Casting in a vacuum requires molds of a high degree of refractoriness and freedom from any volatile materials. Thus investment molds fired at 1850-1950 F are used exclusively for this purpose. The shell-ceramic type of investment mold has virtually replaced the older full-flask type. The shell molds require less material, can be fired quickly without cracking, and are easier to handle. Also, they permit greater flexibility in controlling the cooling rate of the casting. Shell molds are made by coating a wax or plastic replica of the parts desired, together with the runners and sprue formers, with a slurry of fine refractory containing a binder. The coating is built up to a thickness of about 1/4 inch by alternately dipping and drying. Following the final drying period the molds are fired to melt out the replica and eliminate volatile components.

Grain size of the castings is important and can be controlled by adjusting the mold temperature, the pouring temperature of the melt, and by incorporating nucleating materials into the mold surface. The finish on investment castings is in the range 80 to 125 rms microinches. Dimensional reproducibility is generally about ±0.005 in./in.

Nickel-base alloys that do not contain highly reactive elements are usually melted and cast in air. There is a wide variety of these alloys, some of which are: the Monels, Inconels, Hastelloys, and the Illium series of alloys. The principal alloying elements in these nickel-base alloys are chromium, molybdenum, tungsten, copper, and silicon in various amounts. The alloys are not outstanding in creep resistance but usually are designed either for high-corrosion resistance in aqueous media or for oxidation resistance at high temperatures. Some of the alloys such as Inconel X and NA22H have good resistance to oxidation and have useful strengths at temperatures as high as 2200 F. Presence of sulfur in the atmosphere, under reducing conditions, lowers the permissible service temperature of nickel-base alloys.

In general the melting practice for the alloys that can be melted and cast in air is similar to the practice for stainless steel. Basic-lined direct-arc, indirect-arc, or induction furnaces are common. The alloys are cast into investment molds, Shaw molds, or green or dry sand molds, depending upon the size of the castings, the surface finish, and the dimensional accuracy required. Investment molds, the most expensive, have the highest precision; green-sand molds, the lowest in cost, provide the least precision. Shaw molds consist of high-grade silica bonded with hydrolyzed ethyl silicate. They provide a good surface finish and high dimensional accuracy, and are somewhat less costly than investment molds.

POWDER METALLURGY

Nickel powder is readily obtained by a number of methods including the decomposition of nickel carbonyl, electrolysis, and reduction of nickel oxide. Recently, reduction of aqueous solutions of nickel ammonium sulfate with hydrogen has become the dominant method of making nickel powder. Nickel-alloy powders can be made by coreduction, coprecipitation, or atomization of molten alloys.

Because of the relatively high cost of nickel in comparison with iron and copper, structural parts have not often been made of nickel powder. Production of nickel strip by direct rolling of powder was begun recently in Canada for making coins and for certain electronic applications. Nickel strip is made from powder in the United States for electronic applications. The powder-metallurgy product is reported to have greater purity and better properties for electronic components than conventional strip. Nickel-iron, nickel-cobalt, and nickel-copper alloys are readily produced by rolling mixed elemental powders, and are available in strip form.

In the past year or two, nickel dispersion strengthened with about 2 wt % of thorium oxide (TD nickel) has become available commercially in the form of sheet and extruded shapes. This material has good high-temperature strength, but lacks adequate oxidation resistance in the temperature range of its strength superiority.

Attempts to produce nickel-base, high-temperature alloys such as the Inconel 713 C composition by atomizing molten alloys, followed by canning and hot extrusion, have indicated some promise for providing unique properties.

If property requirements warrant the use of nickel-powder structural parts, they can be made by virtually the same practices employed for making iron parts, i. e., cold-die pressing in steel or carbide dies, followed by sintering. If high density is required, repressing followed by resintering is in order. Sintering atmosphere is less critical for nickel than it is for iron, so either dissociated ammonia or hydrogen is adequate. Sintering temperature would be in the range of 2150-2350 F.

Methods of consolidation such as slip casting, hydrostatic compaction, or hot-gas-pressure pressing may be used with nickel or nickel-alloy powders, if there is a technical and economic justification for doing so. Continuous cyclic pressing appears to be a promising method for compacting nickel-base alloys into bars or plates to be sintered and then hot and cold worked by forging or rolling.

ELECTROFORMING

Nickel is the most commonly used material for the electroforming of structural parts. A wide variety of complex shapes, ranging in dimensions from fractions of an inch up to several feet, are made by this process. Essentially, the process is one of electroplating nickel onto a preformed mandrel, then removing the mandrel by stripping, melting, or dissoluting in acid. The mandrels may be made from a variety of materials, such as wax, plaster, low-melting alloys, rubber, aluminum, or zinc. Nonconductive materials must first be given a conductive coating, such as graphite or chemically reduced silver.

Dimensional accuracy of electroforms is excellent; 0.002 to 0.005-inch total variation is relatively easy to obtain on most parts. Surface finish of 8 to 16 rms microinches is fairly common for the mandrel side of electroformed shapes. For thin-walled objects up to 0.1 inch thick the exterior surface finish is commonly 16 to 32 rms microinches. Because of the lack of throwing power of the electrolyte, wall thickness may vary depending on the complexity of the part. A number of methods are available for keeping this variation under control; 5 to 20 per cent variation is normal.

Rates of electroforming usually range from 0.002 to 0.005 in./hr. A new, high-concentration nickel sulfamate bath permits electroforming at 0.01 to 0.02 in./hr.

Electrodeposition conditions influence the properties of the electroforms. Factors that need to be controlled are: the kind of nickel salt and its concentration; the concentration of chloride or other anion employed; the pH of the solution; bath temperature; impurity concentrations; cathode-current density; and amount of agitation. Organic additives for avoiding pitting, reducing stress, or refining the grain structure also are important.

The sulfamate bath is the most popular because the deposits are strong, yet exhibit less residual stress than those produced from other baths and, therefore, are less subject to distortion. Ultimate tensile strengths of 90,000 to 120,000 psi, yield strengths of 60,000 to 70,000 psi, and elongations of 6 to 14 per cent are usual for the sulfamate baths. A residual stress of 5,000 to 10,000 psi is customary for high-strength nickel electroformed in sulfamate-chloride solutions containing no stress reducer. Nickel electroformed from the nickel-chloride bath is highly stressed, usually 40,000 to 50,000 psi, which may cause distortion. Certain organic additives and increased bath temperature tend to reduce the stress. Certain impurities tend to increase stress. These include trivalent and hexavalent chromium, divalent and quadrivalent tin and nitrate ions. Many other elements increase the stress but to lesser degrees.

Nickel tubing containing controlled porosity for passing gases and other media has been made by incorporating graphite into the deposit.

A variety of nickel alloys including nickel-cobalt, nickel-iron, nickel-manganese, and nickel-tungsten have been electroformed. Nickel-20 chromium alloy has been produced experimentally in the forms of sheet and tubing by electrodepositing alternate layers of nickel and chromium, followed by an homogenizing treatment.

Depending on the configuration and number of parts desired, electroforming can be a cost-saving method. Usually electroforming is the most promising when a relatively small number of components is required, say 5 to 200.

CHEMICAL VAPOR DEPOSITION

Production of structural parts by deposition of nickel from nickel carbonyl vapor is technically feasible. Molds, forming dies, patterns, and tools have been made by this process. However, the process is not being used to any significant extent for these applications at the present time. Chemical vapor deposition of nickel is suitable for making wave guides, nozzles, ducts, etc., that would be difficult to machine or fabricate in some other manner. In general, however, it is doubtful that this process has any advantages over electroforming. One distinct disadvantage is that nickel carbonyl is extremely toxic.

METAL SPRAYING

Although nickel and most nickel-base alloys can be sprayed successfully there has been no significant application of this process in making free-standing parts. Nickel and nickel-alloy parts can be made so readily by forging, machining, or casting that there has been no real incentive to make them by metal spraying.

Certain nickel alloys can be sprayed to provide coatings that are abrasion, corrosion, or oxidation resistant on a variety of low-alloy steels or cast irons. Sprayed alloy coatings, which are subsequently fused for improved density and adherence, include nickel-chromium, nickel-aluminum, nickel-silicon-boron and nickel-chromium-silicon-boron alloys. A variety of compositional ranges are employed depending on the specific application. In a typical spray gun the alloy powder is aspirated, usually with air, into a stream of a fuel gas. The heat from the burning fuel gas melts the particles of the alloy and projects them at high velocity against the surface being coated. Hydrogen or acetylene are the usual fuel gases employed. Coatings of this type are usually subsequently fused by the controlled application of an oxy-acetylene flame to the coated surface. Some of the nickel-base alloys intended mainly for wear resistance are as hard as 60-63 R_C. Others intended for corrosion or oxidation resistance more than for wear resistance may be as soft as 20 R_C. Usually the fused coatings are 0.015 to 0.050 inch thick. If smooth surfaces are required the coatings usually are wet ground. Finishes as smooth as 2 rms microinches can be obtained by lapping.

Nickel aluminide has been sprayed to form nonporous coatings that are resistant to gaseous atmospheres up to 1760 C. A composite particle is formed by chemically depositing nickel onto aluminum particles. When fused and sprayed there is an exothermic reaction with the formation of the nickel aluminide. The nickel aluminide is self-bonding to properly prepared surfaces, and no subsequent fusion treatment is required.

Recently plasma torches have been developed that generate temperatures in excess of 15,000 F. Plasma-sprayed nickel-base alloys can have good to excellent bonding and reasonably low porosity. Also, flame plating, which utilizes a series of rapid detonations to project hot-alloy particles onto the workpiece, can be used for spraying nickel-aluminum alloys. The particles strike the workpiece at very high velocities. On impact the particles are further heated by release of kinetic energy. Because of the intermittent nature of the detonations the surface of the workpiece can be held to temperatures of a few hundred degrees, thus avoiding metallurgical changes in the substrate. Flame-plated coatings have good adherence and are reasonably free from porosity. They must be sealed, however, for best resistance to corrosion.

INTRODUCTION

Nickel-base alloys are widely used in the forms of sheet, strip, bars, forgings, and castings. Many of the alloys are particularly corrosion resistant in aqueous media, such as salt water, alkalies, and nonoxidizing acids; others have especially good oxidation resistance at very high temperatures. Some of these latter alloys combine high oxidation resistance with exceptional strength at high temperatures, thus making them among the most widely used materials for jet-engine service at temperatures up to about 1850 F.

The wide use of nickel-base alloys, the variety of methods employed in making them into useful hardware, and the constant evolution of processing procedures suggested that a state-of-the-art study on methods of making nickel-alloy parts would be worthwhile. The study included casting, powder metallurgy, electroforming, chemical vapor deposition, and metal spraying. The sources of information included the open literature, unpublished reports on Government contracts, and personal contacts with leading authorities in the respective fields.

CASTING

The nickel-base casting alloys are numerous. They are noted for their resistance to corrosion in many environments other than those that contain sulfur. Many of them are oxidation resistant and have superior strengths at high temperatures.

The alloys and the probable patentee or developer of each alloy are identified. Typical tensile properties, stress-rupture properties, and other mechanical properties are presented. Each alloy or group of alloys is characterized with respect to how the alloy is melted, cast, heat treated, and further processed.

Because the vacuum-cast alloys are so important to the aerospace program, this type of melting practice is described in some detail. Molding practice and dimensional tolerances of castings are covered with greater detailed attention to investment molds because they are used in vacuum casting. Information on specific alloys is presented in three groupings: (1) domestic vacuum-cast alloys, (2) domestic air-melted alloys, and (3) British alloys.

VACUUM-MELTING PRACTICE

Since the advent of the turbosupercharger in 1918, rapid advancements were made in the development of wrought-bucket alloys to withstand higher service temperature, but the advancement started to level off in about 1950 (Ref. 1). The reason for the leveling off was the lack of adequate ductility as alloying was continued to increase the strength.

Some of the more promising alloys at that time were the nickel-base alloys containing titanium and aluminum that promoted beneficial precipitation hardening. These reactive elements made it difficult to process the alloys by conventional melting techniques. Vacuum melting or careful remelting of vacuum-refined stock under an inert atmosphere soon was found to improve the cleanliness of the alloy and thus improve the ductility and forgeability. In fact, oxide and nitride inclusions no longer were problems that restricted the level to which titanium and aluminum could be present in the alloy. However, with the increase in high-temperature strength from increased alloy content, forgeability was impaired and casting became an alternative method of production.

In the early history of vacuum melting obtaining consistent reproducibility of properties was a serious problem. Research by Universal Cyclops in 1957 and subsequently by the University of Michigan revealed that boron (0.005 to 0.01 per cent) and zirconium (0.05 to 0.1 per cent) had very marked beneficial effects on the stress-rupture life and ductility. Also it was discovered that boron could be introduced accidentally from a magnesia crucible and zirconium from a zirconia crucible. An excessive amount of these two elements had a deleterious effect on the stress-rupture properties and caused hot shortness by forming a low-melting phase at the grain boundaries (Ref. 2). With proper control of these elements, reproducibility of properties was greatly improved in castings produced from vacuum-melted heats.

About twice the time is required to prepare a vacuum-refined heat from virgin metal as it does to remelt a vacuum master alloy. Therefore, economy favors remelting of bar stock from vacuum-refined heats that may be as large as 10,000 pounds (Ref. 3). In addition to a shorter melting and casting cycle, remelting and casting under vacuum offers the following advantages (Ref. 4):

(1) Composition of the melt is under more precise control from heat to heat (for example, 30 small heats prepared from a master heat of Udimet 500 had the same composition within the analytical error of the laboratory) (Ref. 5).

(2) Neither adjustment of composition nor additions for deoxidation are necessary.

(3) Surface of the melt is clear of oxide film. Temperature measurements with an optical pyrometer are more accurate and permit better control of the melt temperature.

(4) Absence of oxide film on the melt results in cleaner castings internally and externally.

(5) Molds can be filled completely at a lower pouring temperature because there is no atmosphere to create back

pressure in the mold cavity (misrun is one of the main causes for rejection of investment castings poured in air (Ref. 6).

(6) The lower pouring temperatures that are permissible result in shorter casting cycles, still greater consistency in composition because of reduced reactivity with the crucible, and the production of castings having finer grains.

Induction furnaces currently have casting capacities up to 500 pounds (Ref. 3). However, most investment castings weigh less than 20 pounds per mold. The general practice is to size the furnace shell and induction coil to melt a charge to pour a single mold. A small furnace gives closer control over the composition of the melt, attains a lower pressure without the need for large, expensive vacuum pumps, requires less extensive leak monitoring, and the small mold can be processed in less time (Ref. 5).

For melts under 100 pounds, the crucible is generally prefired high-purity alumina, magnesia, or zirconia (Ref. 3). Rammed linings cannot be fired in place to a density that will not release refractory particles into the melt (Ref. 7). Such particles do not decompose and may be entrained in the melt during pouring. Although alumina crucibles have been reported to have a life of 140 heats in a vacuum-induction furnace (Ref. 7), others have reported a life of 49 to 60 heats in an indirect-arc, vacuum furnace (Ref. 8), and still others prefer magnesia or zirconia for vacuum-induction melting (Ref. 3). A silica crucible may introduce up to 0.05 per cent silicon into the melt (Ref. 7). The life of a magnesia crucible may be short because its high thermal coefficient of expansion is likely to make it crack. If the operation of the furnace is nearly continuous, the crucible life is extended. Zirconia is strong and refractory, but may introduce zirconium into the melt. The moisture problem with a newly lined furnace is handled by making one or more wash heats under vacuum (Ref. 9). Low pouring temperatures may build up a skull, which deteriorates the crucible. Repair of crucibles should be made with patch material containing a minimum of moisture.

Furnace design has evolved the two-chamber vacuum-induction system such as the one shown in Figure 1. In the two-chamber system, the mold chamber is separated by a vacuum lock from the melt chamber and the crucible is under vacuum for the extent of its life (Ref. 3). In the one-chamber system, the crucible is exposed to air each time that the mold is replaced. The power source is generally 3 to 10 kilocycles with an applied potential of not more than 400 volts to minimize arcing between the turns of the induction coil. The furnace shell is a double wall that serves as a water jacket. The interior wall is stainless steel and the outer wall is mild steel. An oil-vapor-booster pump backed with a mechanical pump can maintain a pressure of 5 to 15 microns during melting. A vapor-diffusion pump between the melt chamber and the vapor-booster pump increases the pumping speed and can maintain a level of a micron or less. Measurement of the melt temperature is made with an optical pyrometer through a glass port that can be cleaned without interrupting the vacuum in the melt chamber.

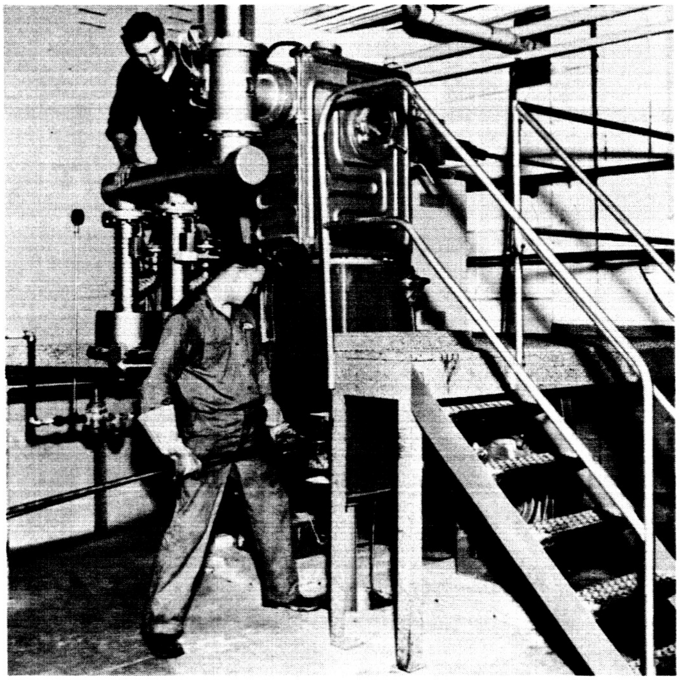


FIGURE 1. DOUBLE-CHAMBER, 30-POUND-CAPACITY VACUUM-INDUCTION FURNACE SHOWING LOADING OF MOLD CHAMBER

Courtesy of Misco Precision Casting Company.

Although the criterion for an acceptable leak rate is the quality of the casting, usually a rise in pressure of 60 μ/cfm of chamber space is acceptable for a cold furnace, or 600 to 1200 μ/cfm for a furnace containing a molten charge (Ref. 3).

The effect of leak rate on properties is illustrated by the data plotted in Figure 2 for wrought Udimet 500 melted at a pressure of

1 micron in a 1000-pound furnace (Refs. 10, 11). Stress-rupture life and ductility and tensile ductility suffered as the leak rate increased. In Figure 3 it is evident that at least part of the deterioration in stress-rupture life is a result of buildup of oxygen in the melt as the leak rate increased. Presumably the similar results could be expected with Udimet 500 castings and other casting alloys containing titanium and aluminum. Thus it is essential that the vacuum furnace be subjected to a good.maintenance program (Ref. 12). At the end of each working period, inspection should be made of the mechanical and electrical connections and vacuum seals, and accumulated dust should be removed.

A typical cycle for vacuum casting is as follows (Ref. 3). The crucible receives its charge through a vacuum lock. The charge consists of clean, vacuum-melted bars that may range from 3/4 to 2-3/4 inches in diameter (Ref. 13). The larger diameter bars have the smallest surface area per given weight and thus are apt to produce the least amount of oxide scum upon meltdown. For a charge of 25 pounds or less, the power input is 50 kilowatts and melting proceeds under a vacuum of less than 15 microns. An investment mold, preheated to 1500 to 1900 F, is placed in the mold chamber. When this chamber has been pumped down to a pressure of less than 100 microns, the vacuum lock to the melt chamber is opened and the mold is positioned for the pour. The pump for the melt chamber quickly reduces the pressure of the system to below 15 microns. After the pour, the mold is returned to its chamber to cool while the crucible is recharged through the vacuum lock on the charge chamber. While the power is on for melting the charge, the mold chamber is back filled with air. At atmospheric pressure, the mold chamber is opened and the filled mold is replaced with an empty one. In a typical production cycle, the melting rate is 2 to 2-1/2 pounds per minute, and up to a 30-pound charge is melted and cast every 5 to 15 minutes (Refs. 3, 14).

The rate of pour must be rapid to avoid misruns. Recommended pouring rates range from less than 1 second per melt or per casting (Refs. 7, 14) to 2 seconds (Ref. 3). Superheat of as little as 80 F did not adversely affect filling of turbine-blade molds provided that the vacuum pressure was 1 micron or less (Ref. 7). Reasoning would suggest that the minimum pouring rate should be dependent upon the chilling capacity of the mold material, the temperature of the mold, the superheat of the melt, the size of the section being cast, and the design of the gating system.

A mass-production method is used by Kolcast Industries to produce vacuum-melted castings (Ref. 15). Prefired investment molds

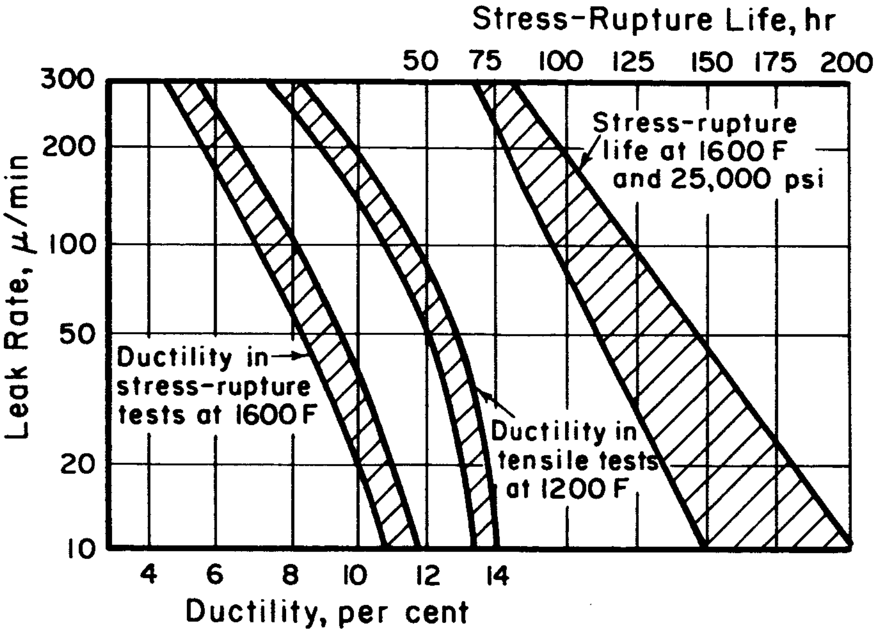


FIGURE 2. INFLUENCE OF LEAK RATE OF FURNACE ON HIGH-TEMPERATURE PROPERTIES OF WROUGHT UDIMET 500 (REF. 10)

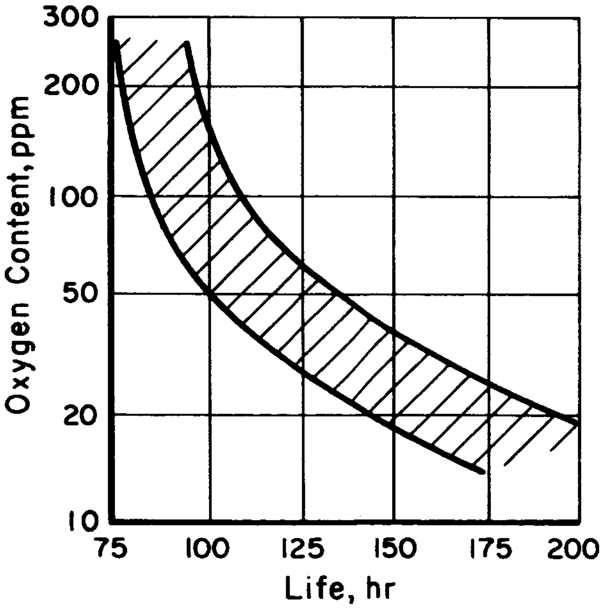


FIGURE 3. EFFECT OF OXYGEN CONTENT ON STRESS-RUPTURE LIFE OF WROUGHT UDIMET 500 AT 1600 F AND 25,000 PSI (REF. 10)

are loaded in a 46-1/2-foot chamber that is heated to 1250 F and pumped to a pressure of 10 microns. In 10 to 30 minutes, this chamber is opened to the melt chamber and a continuous chain conveyor transports the 60 (maximum) molds to the pouring station. A 350-pound induction furnace transfers metal to a preheated ladle that is used to pour the molds. The furnace crucible is charged through a vacuum lock. Filled molds are transported by a conveyor belt to a vacuum-locked cooling chamber. Production rate is said to be 4 times that of a conventional process.

Although the indirect-arc furnace costs less than an induction-melting furnace, the latter type is now more popular for vacuum melting. The indirect-arc furnace has the following disadvantages: lower melting rate, carbon pickup indirectly from the carbon electrodes, poorer control of the mold temperature, and poorer control of the melt temperature because the melt is heated only by radiation through the top surface and because there is no stirring action as in induction melting (Ref. 16). In practice, the indirect-arc furnace is pumped down to 200 microns, back filled with argon to a pressure of 50 mm of mercury, pumped down to 500 microns, and back filled to 50 mm (Ref. 12). The arc is struck and successive bars are fed into the crucible and melted. When the desired melt temperature has been slightly exceeded, the power is shut off and the furnace is pumped to about 50 microns during the couple of minutes that the melt is cooling and equilibrating to the desired temperature. Pouring is accomplished by tilting the entire furnace 180 degrees since the mold is fastened above the crucible. Often the argon pressure is increased to about 30 mm of Hg in the furnace just before or during the pour to decrease any tendency for metal-mold reaction (Ref. 16). After 3 to 5 minutes, the casting has solidified; the furnace is then righted, back filled with air, opened, and the mold removed. A typical cycle for a 20-pound charge is about 30 minutes (Ref. 12). In air melting, the mold is not attached to the furnace until after the meltdown.

In the early 1950's, remelting under an argon blanket was considered to be good practice and desirable because it was more economical than vacuum melting. However, there was always the question of how protective the blanket really was because the stress-rupture life of nickel-base alloys containing titanium and aluminum could be expected to be only about 80 per cent of the life of vacuum-melted castings (Ref. 17). Furthermore, the data (Ref. 17) in Table I show that at that time the price of vacuum-melted castings was only one-half more than air-melted castings and only slightly higher than castings from melts prepared under argon.

TABLE I. COST COMPARISON FOR INVESTMENT CASTING IN AIR, ARGON, AND VACUUM[a]

Item	Air	Argon	Vacuum
Mold cost	$35.00	$ 35.00	$ 35.00
Metal cost per mold	16.00	40.00	40.00
Melting cost per mold	2.60	5.20	9.70
Cleaning, cutoff, inspection	18.00	18.00	18.00
Total cost without return credit	$71.60	$ 98.20	$102.70
Minus metal returns	6.24	6.24	6.24
Total cost with return credit	$65.36	$ 91.96	$ 96.46
Margin	19.61	27.59	28.94
Total price	$84.97	$119.55	$125.40
Selling price per casting	$15.17	$ 21.35	$ 22.39
Increase in price over air melting, per cent	--	40.3	47.7

(a) Based on $60,000, the vacuum-induction furnace amortized on a one-shift operation in a 2-year time period when pouring 10 flask molds per hour with eight 1/2-pound castings per mold, 50 per cent yield per mold, 30 per cent rejects, $3/pound premium for vacuum-melted stock for remelting, allowing 30 per cent for margin, and including overhead.

AIR-MELTING PRACTICE

DOMESTIC ALLOYS

For those nickel-base alloys that can be melted in air, the general practice is similar to that for melting stainless steel. Large heats may be prepared in a basic-lined, direct-arc furnace under a reducing slag. An acid lining is seldom used because of the high chromium loss that results and the difficulty in controlling the silicon content of the heat (Ref. 18). Excessive superheat leads to excessive gassing of the melt. Calcium silicide or calcium-manganese silicon are used to deoxidize the melt in the ladle. If magnesium is used for deoxidation, 0.10 per cent is added in the furnace after the surface of the bath has been cleaned of slag (Ref. 19). Final adjustment of composition is done by making additions to the ladle. Sand and shell molds are commonly used. Ladle pouring of a series of investment molds is practiced in some foundries (Ref. 20).

A slag cover is not used when melting in an induction furnace or indirect-arc furnace (Ref. 21). Small furnaces of these types are commonly used to produce investment castings. The charge is usually stock from a master alloy, but may consist of clean, select scrap. To minimize formation of slag from the basic furnace lining, meltdown is rapid and superheat is kept low. The melt is deoxidized by the addition of calcium silicide or calcium-manganese silicon. For small heats for investment casting, pouring is accomplished by rapidly inverting the furnace that has the investment mold clamped above the crucible. The design of the furnace usually is such that air pressure can be applied to the interior to apply force during filling of the mold. For large heats, the metal is transferred to a ladle for pouring the molds.

BRITISH ALLOYS

The Nimocast alloys may be melted in air in an induction furnace or an indirect-arc furnace that has a basic lining, such as magnesia, chrome-magnesite, zircon, or zirconia (Ref. 22). An acid lining results in excessive loss of titanium and aluminum as well as pickup of silicon. As a guide, the loss in a basic-lined induction furnace, when melting a charge of an alloy such as Nimocast 90 in 11 to 18 minutes, is about 0.1 per cent titanium and 0.3 per cent aluminum. More than 50 per cent foundry returns in the charge may make control of composition difficult. Losses are minimized by rapid melting, minimum holding time in the molten condition, and minimum superheat of the melt consistent with pouring at a temperature that will not result in misruns. Low superheat also minimizes shrinkage porosity.

To minimize oxide inclusions in the castings, the practice is to plunge or stir 0.03 per cent calcium as a calcium-silicon alloy into the melt shortly before pouring. The residual calcium content must be kept below 0.02 per cent because of its adverse effect on stress-rupture properties (Ref. 22). Complete freedom from oxide inclusions would require melting and casting under vacuum, or remelting vacuum-melted stock under argon. One British company announced in 1961 that the Nimocast and G. 64 type of alloys are being vacuum cast as a means for eliminating oxide inclusions and maintaining high metallurgical standards (Ref. 8).

MOLDING PRACTICE

CERAMIC MOLDS

Methods. Investment and Shaw molds are ceramic molds that are fired to a degree that frees them of all volatile constituents. Because of its greater applicability to nickel-base alloys, emphasis is placed here on the investment molding process.

There are many variations in details in the production of investment molds (Refs. 23-25, 32). However, investment molding starts with a wax or plastic pattern produced by injecting these materials into an accurately machined metal die. The cost of an aluminum die may range from $50 to $350 for a complicated casting (Ref. 33). Plastic patterns are generally more expensive than wax patterns because they must be produced in a steel die that is more expensive than the brass or aluminum die that is satisfactory for producing wax patterns. The use of plastic gives better dimensional control in patterns having massive sections (Ref. 30). A complicated steel die may cost up to $5000. Of the two types of pattern materials, waxes are more widely in use (Ref. 34).

Wax patterns are assembled to wax gates, runners, and sprue by "welding" with a knife heated by electrical resistance. Plastic patterns are assembled by cementing. An assembly is invested by dipping it into a primary coating slurry consisting of a fine grade of refractory material and ethyl silicate, sodium silicate, or silica sol for bonding. While wet, the assembly is stuccoed with coarser refractory material (35 to 80 mesh), then oven dried.

To make a shell mold, dipping, stuccoing (also called sanding), and drying are repeated until the mold wall builds up to a thickness of about 1/4 inch. The final thickness can be greater if a still stronger mold is required. The use of coarser grain (10 to 35 mesh) after the first stucco coat also strengthens the shell (Ref. 23).

To make a flask mold, the precoated assembly is suspended in a stainless steel container and a coarse slurry is poured around it. Vibration and vacuum are used to eliminate air bubbles that may be present, especially those at or near the surfaces of the pattern.

Both types of mold must be dried thoroughly before dewaxing. Insufficient drying prior to dewaxing may result in mold cracks or castings with rough areas or oversize sections (Ref. 24). Whereas the flask mold is heated slowly to a low temperature to melt out the pattern; the shell mold can be placed in a hot furnace to flash melt the pattern. After the pattern is eliminated, the mold is fired at temperatures ranging from 1500 to 1900 F. The molds may be cooled and used cold or reheated for use at any desired temperature.

Shell molds came into prominent use in 1958. They are less expensive than flask molds because they require only about one-tenth of the material that is needed to make flask molds and are processed in less time (Refs. 25-27). Mechanized production can make 60 complete shells per hour that require only 10 to 15 minutes for dewaxing and firing (Ref. 26). Shell molds also have less tendency to crack during processing (Ref. 12). In addition, shell molds increase the range of casting weight to 400 pounds (Ref. 28) and lengths up to 24 inches (Ref. 27). Apparently the limit on size is set by the available facilities and is not restricted by the investment process per se. From a metallurgical viewpoint, the shell mold is preferable to the flask mold because the casting cools faster in a shell mold with the result that the as-cast grain size is smaller.

Because investment molds are fired at a high temperature and are free of volatile materials, they are the only type considered to be suitable for use in casting under vacuum. To have the mold at the proper temperature at the time of pouring, the mold can be preheated to a higher temperature to allow for the degree of cooling that takes place during the several-minute time lapse between setting it in place and pouring. If slower cooling of the casting is permissible, the shell mold can be backed in a flask with refractory grog during firing. Such a mold will have a drop of only 25 F when held for 10 minutes in a vacuum chamber (Ref. 3).

Vacuum casting accentuates metal-mold reaction. A precoat with alumina or zirconia is recommended because of greater inertness than one of silica (Ref. 14). When stuccoed with alumina, zirconia, or thoria, castings cool much faster because of the higher specific heats of these materials (Ref. 29). If silica gel happens to penetrate between the precoat and the pattern, reaction with the melt during casting can cause violent blows (Ref. 16). Inadequate firing of the mold can leave a residue of carbonaceous wax that can also result in blows (Ref. 7). Mold cavities should be cleaned of loose granules by blowing or by vacuum suction before use to eliminate this source of inclusions.

The grain size in castings is controlled by the type of mold material, as noted earlier, and by the mold temperature and pouring temperature (Ref. 14). Pouring temperature is the most potent of all of these factors (Ref. 16). In addition to a low pouring temperature, low mold temperature, and mold materials of high specific heat, small grain size can be obtained by incorporating suitable metal oxides in the dip coat (Refs. 14, 29, 30). A patent (Ref. 31) reveals that the dip slurry contains 1 to 5 per cent of the oxide of iron, cobalt, nickel, or manganese, depending upon the composition of the alloy being cast. Prior to pouring, either trichloroethylene or heptane is injected into the cavity of the hot mold to reduce the oxide to metal. The fine-grained structure in the casting is achieved by the promotion of rapid solidification. An example of the effectiveness of this mold treatment on the grain size is shown by the turbine blade in Figure 4.

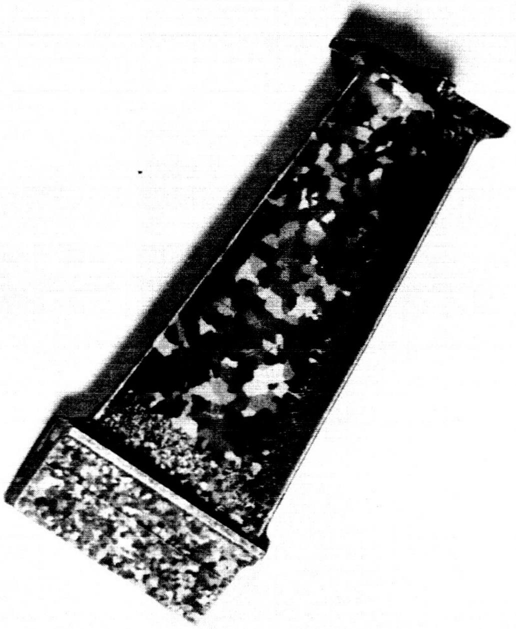


FIGURE 4. CONTROL OF GRAINS TO PREDETERMINED SIZES IN PRESELECTED AREAS OF A NICKEL-BASE TURBINE BLADE

Courtesy of Haynes Stellite Company.

In a recently developed casting technique, an increase of 75 to 100 F in operating-temperature capability was obtained in jet-engine vanes of MAR-M200 alloy along with a 200 to 300 per cent increase in ductility and a marked improvement in resistance to thermal shock (Refs. 35, 36). The improvements result from a columnar grain structure in which the grain boundaries are aligned parallel to the axis of the applied stress. Carefully controlled temperature gradients during casting produce the directional solidification that results in the columnar grains. Currently it is possible to produce castings up to 10 inches long with preferentially oriented columnar grains. The casting technique is said to be applicable to nickel-base superalloys other than MAR-M200.

Although it is not an investment process, the Shaw process is capable of producing precision castings (Ref. 37). Molds produced by the Shaw process are fired to a red heat and thus would be suitably inert for casting in a vacuum furnace. To date it has been used for casting only air-melted alloys. No reference exists on its use for casting nickel-base alloys.

In the Shaw process, a slurry consisting of refractory aggregate, hydrolized ethyl silicate, and a gelling agent is poured over the pattern. A flexible gel forms quickly under controllable conditions. Stripping of the mold is possible with very little draft on the pattern. After stripping, the mold is ignited with a torch. This causes the formation of a microcrazed surface that makes the mold, after firing to a red heat, immune to thermal shock. The molds may be poured hot or cold. Shaw shell molds can be used for castings not exceeding about 20 pounds in weight. The solid block mold or composite mold (backing consists of about 90 per cent carbon dioxide-bonded aggregate for greater economy) is used for larger castings.

There is no standard answer or rule-of-thumb approach to the question of how many parts must be produced to make it economical to use investment castings. The only way to determine the break-even point is to obtain quotations for various quantities of parts produced by investment casting and by fabrication, and to plot the data as illustrated by the examples in Figures 5 and 6 (Ref. 38). Figure 5 is representative of a large, complex part of an alloy that is difficult to work. Figure 6 is representative of a smaller part that requires considerable machining or numerous setups for brazing or welding.

Finish and Tolerance. The finish on investment castings is in the range of 80 to 125 rms microinches (Ref. 33). Some suggested specifications for minimum dimensional tolerances are as follows (Ref. 39):

General dimensions:	±0.005 in./in. In some cases, ±0.003 inch is possible on selected dimensions or on dimensions under 0.250 inch
Radii:	±1/64 inch for small radii. For large radii, ±1/64 inch for each 2 inches of radius

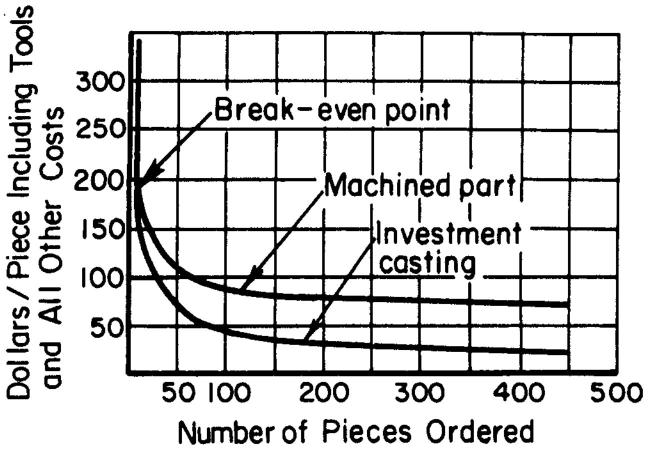


FIGURE 5. COST FOR MACHINED PART VERSUS INVESTMENT CASTING

This curve might be applicable to a typical large complicated part requiring extensive machining, many machine setups, or a material that is extremely difficult to work. The investment-casting curve includes a cost for secondary machining operations.

Machined Part:	
Tooling	$350
Machining cost	150 each (1-10 pieces) 100 each (11-50 pieces) 75 each (51-250 pieces) 73 each (251-450 pieces)
Material cost	3 each
Investment Casting:	
Tooling	$1200
Part price	50 each (1-10 pieces) 35 each (11-50 pieces) 25 each (51-100 pieces) 22 each (101-500 pieces)
Machining cost	$ 15 each (1-10 pieces) 12 each (11-50 pieces) 9 each (51-100 pieces) 7 each (101-500 pieces)

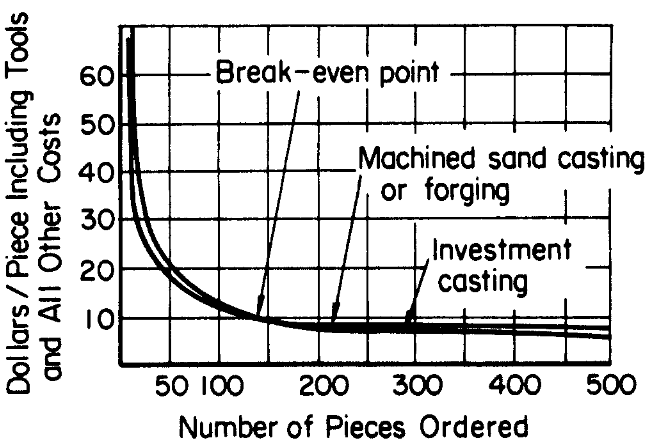


FIGURE 6. RELATIVE COST FOR MACHINED CASTING, FORGING, AND INVESTMENT CASTING

Curves similar to those shown above might be plotted for many fairly small sized sand castings or forgings requiring considerable machining or a number of setups. This set of curves might also be typical of an assembly of several parts that might require a very minimum of machining, but also require fixturing for brazing, welding, etc.

Machined Part or Assembly of Parts:	
Patterns/tooling	$350.00
Casting or part cost	3.50 each (1-50 pieces) 1.50 each (51-100 pieces) 1.35 each (101-250 pieces) 1.25 each (251-500 pieces)
Machining/assembly, etc.	6.00 each (1-25 pieces) 4.50 each (26-100 pieces) 4.00 each (101-250 pieces) 3.80 each (251-500 pieces)
Investment Casting:	
Tooling	$800.00
Part cost	6.00 each (1-50 pieces) 3.50 each (51-100 pieces) 2.85 each (101-250 pieces) 2.75 each (251-500 pieces) 2.65 each (501-)
Machining costs	3.00 each (1-50 pieces) 1.80 each (51-100 pieces) 1.70 each (101-250 pieces) 1.60 each (251-500 pieces) 1.55 each (501-)

Straightness: ±0.020 inch for up to 2 inches, ±0.030 inch for 2 to 4 inches, ±0.040 inch for 4 to 6 inches, ±0.060 inch for over 6 inches.

The finish from a Shaw mold is 80 to 120 rms microinches (Ref. 37). It is claimed that an average tolerance of ±0.003 inch per inch can be held consistently on dimensions up to 3 inches, and an overall tolerance of ±0.005 inch is generally possible for lengths of 3 to 6 inches.

SAND MOLDS

Methods. Sand molds are made by a number of methods and are restricted to the production of castings from air-melted heats. The molding methods are green sand, dry sand, carbon dioxide-cured sand, and resin shell. There are numerous variations in formulation and molding procedure for each method. Only basic steps are described here.

Green molding sand is a mixture of sand grains (usually silica), 5 to 10 per cent clay, and 3 to 5 per cent water. The mixture is rammed around a pattern placed in a flask. A mold is made in two halves, called the cope (upper half) and drag (lower half). The mold is ready for pouring immediately upon assembly of the cope and drag. There is no limit on the size of casting that can be molded in green sand; the sand is reusable. However, long, thin projections of the mold are not strong enough to withstand handling of the mold or the force of the molten metal during pouring. Such projections must be made of dry sand cores. In casting nickel-base alloys a reaction may occur with the moisture in the green sand mold. This problem is alleviated by drying the face of the mold just prior to use.

Dry sand molding is similar to green sand molding except that binders such as pitch or oil are used instead of clay. After ramming, the mold is stripped from the core box and hardened by baking at 350 to 500 F. Molds are usually cooled to room temperature for pouring.

The carbon dioxide molding process employs sodium silicate as the binder. After ramming and before stripping, carbon dioxide gas is blown through the mold. The reaction with the silicate, which hardens the mold, is accomplished very quickly. The characteristics of the mold are generally similar to those of a dry sand mold without the need for prolonged baking to develop the bond. Restraint to shrinkage of the casting is greater than in green or dry sand molds.

Shell molding by the Croning process (also called C-process) should not be confused with the shell investment molding process. In the Croning process, a mixture of sand grains and 3 to 8 per cent thermosetting plastic is dropped onto a metal pattern heated to a moderate temperature. In a timed sequence, the pattern is inverted to allow the loose mixture to drop away from the shell that has formed against the pattern. After an additional dwell period of 1 or 2 minutes, the cured shell, which is 1/4 to 3/8 inch thick, is stripped from the pattern. Mold halves are cemented or clamped together and may be poured immediately. Pattern cost is high in this process. Finish and dimensional tolerance can be quite good. The characteristic of ready collapsibility results in negligible restraint to shrinkage of the casting, but also restricts the use of shell molds for the less massive castings. However, production of castings weighing several hundred pounds is possible if the sand mold is backed with sand or shot.

Finish and Tolerance. Greater dimensional precision and higher cost per casting can be expected from the resin-bonded shell process, and the poorest precision and lowest cost from the green-sand-molding process. In general, the molding processes rank as follows (Ref. 40):

Resin-bonded shell - highest precision, highest cost
CO_2 cured
Dry sand
Green sand - lowest precision, lowest cost.

COMPARISON OF DIMENSIONAL TOLERANCES AND SURFACE FINISHES

Dimensional accuracy of a casting is dependent upon the accuracy of the pattern after allowance is made for the patternmaker's shrinkage for the specific alloy being cast. Reproducibility of dimensions is related to the reproducibility of all the steps in the molding and pouring practice for the specific shape being cast. The data in Table II are presented to provide a comparison of the dimensional tolerances and surface finishes that characterize the various molding processes (Refs. 33, 37, 41, 42).

TABLE II. DIMENSIONAL TOLERANCE AND FINISH BY VARIOUS MOLDING PROCESSES

Molding Process	Tolerance[(a)], ±in.		Finish, rms microinches	Finish Stock for Machining, in.[(b)]
	In 1 Inch	In 6 Inches		
Green sand	0.015-0.030 (normal) 0.005 (optimum)	0.020-0.040	250-1000 (normal) 100-250 (optimum)	0.030-0.250
Dry sand	0.005 (optimum)	0.020-0.040	100-250 (optimum)	0.030-0.250
CO_2 cured	0.010-0.020	0.015-0.030	100-250 (optimum)	0.020-0.125
Resin-bonded shell	0.005-0.010	0.013-0.026	50-250	0.010-0.060
Shaw	0.003 (optimum)	0.005-0.010 (optimum)	80-120	--
Investment	0.005-0.010 (normal) 0.003 (optimum)	0.010-0.020	80-125 (normal) 10-85 (optimum)	0.010-0.060

(a) If dimension is across a parting line, add 0.005 to 0.015 inch to the tolerance.

(b) Add to the dimensional tolerance.

COMPOSITIONS AND PROPERTIES OF ALLOYS

DOMESTIC VACUUM-CAST ALLOYS

Table III lists the composition and probable developer of each of the domestic vacuum-cast alloys, Actually several of the alloys are also melted and cast under argon. Those melted and cast under argon are identified in the discussion of each alloy.

Table IV presents typical tensile and stress-rupture properties. Other pertinent properties and characteristics are presented in the discussion of the individual alloys.

Inconel 713 C and 713 LC. Specification AMS 5391A requires the following properties for Inconel 713 C in the as-cast condition (Ref. 43):

(1) Stress-rupture properties: 30-hour minimum life and 5 per cent minimum elongation when stressed at 22,000 psi at 1800 F

(2) Tensile properties at room temperature: 110,000 psi minimum tensile strength, 100,000 psi minimum yield

TABLE III. COMPOSITION OF DOMESTIC VACUUM-CAST, NICKEL-BASE SUPERALLOYS

Per Cent.

Alloy	C	Mn	Si	Cr	Ni	Co	Mo	Ti	Al	Fe	B	Other	Probable Developer	Reference
Inconel 713 C(a)	0.08/0.20	0.25*(b)	0.50*	12.0/14.0	Bal	1.00*	3.8/5.2	0.50/1.0	5.5/6.5	2.5*	0.005/0.015	0.015*S, 1.8/2.8Cb, 0.50*Cu, 0.05/0.15Zr, 0.015*V	INCO	43,46,47
Inconel 713 C	0.03/0.07	0.25*	0.50*	11.0/13.0	Bal	--	3.8/5.2	0.4/1.0	5.5/6.5	0.50*	0.005/0.015	1.5/2.5Cb, 0.05/0.15Zr, 0.50*Cu, 0.015*S,	Ditto	48
Inconel 717 C	0.12	--	--	11.0	Bal	8.5	4.2	0.8	6.1	--	0.012	2.0Cb, 0.10Zr	"	49
Inconel 718 C(a)	0.10*	0.25*	0.50*	17/21	50/55	--	2.0/4.0	0.4/1.2	0.4/1.0	Bal	--	0.03*S, 4.4/5.4Cb, 0.5*Cu, 1.75*Ti+Al	"	46
Inconel 700	0.10	--	--	15	Bal	28	3.0	2.0	3.0	0.5	--	--	"	50
Inconel 702	0.04	0.10	0.25	15.5	79.0	--	--	--	3.0	0.5	--	--	"	51
IN-100(PDRL-100, EPK-24)(a)	0.15/0.20	0.50*	0.50*	8/11	Bal	13/17	2.0/4.0	4.5/5.5	5.0/6.0	1.0*	0.01/0.02	0.015*S, 0.03/0.09Zr, 0.70/0.90V	"	46
MAR-M200(SM-200)(a)	0.12/0.17	0.20*	0.20*	8.0/10.0	Bal	9.0/11.0	--	1.75/2.25	4.75/5.25	1.50*	0.01/0.02	11.5/13.5W, 0.75/1.25Cb, 0.03/0.08Zr, 0.015*S, 0.10*Cu	Martin Metals	46,52
Udimet 500(a)	0.10*	0.20*	0.30*	16/20	Bal	16/20	3.0/5.0	2.5/3.25	2.5/3.25	2.0*	0.003/0.010	0.015*S, 0.10*Cu	Special Metals	46,53
Udimet 600	0.1	--	--	18.0	Bal	16.0	4.0	3.1	4.0	4.0	0.04	--	Ditto	12
Udimet 700(a)	0.10*	0.15*	0.20*	14/16	Bal	17/20	4.5/5.5	2.75/3.75	3.75/4.75	4.0*	0.025/0.035	0.015*S, 0.06*Zr, 0.10*Cu	"	46
René 41(a)	0.06/0.12	0.50*	0.50*	18/20	Bal	10/12	9/10.5	3.0/3.3	1.5/1.8	5.0*	0.003*	--	General Electric	46
Astroloy	0.05	0.05	0.05	15.0	Bal	15.0	5.0	3.5	4.5	--	0.03	--	Ditto	54
I 1360 (Guy Alloy)	0.10	--	--	--	70.5	10	5	--	6	4.5	0.3	2Cb	"	2,49
DCM	0.08*	0.10*	0.15*	14.0/16.0	Bal	--	4.5/6.0	3.35/3.65	4.4/4.8	4.0/6.0	0.07/0.09	0.015*S, 0.10*Cu	"	55,56
B&B	0.08	--	--	15	45	30	5	2.5	3.0	--	0.5	--	"	49
SEL-1(a)	0.08	0.3*	0.5*	15	Bal	26	4.5	2.4	4.4	1.0*	0.015	--	"	49
SEL-15(a)	0.07	0.3*	0.5*	11	Bal	14.5	6.5	2.5	5.4	0.5*	0.015	1.5W, 0.5Cb	"	49
GMR-235	0.10/0.20	0.10*	0.30*	14/17	Bal	--	4.5/6.0	1.5/2.5	2.5/3.5	8/12	0.05/0.10	--	General Motors	57
GMR-235D	0.10/0.20	0.10*	0.30*	14/17	Bal	--	4.5/6.0	2.0/3.0	3.25/4.00	3.5/5.0	0.05/0.10	--	Ditto	57
TRW-1800(a)	0.09	--	--	13.0	Bal	--	--	0.6	6.0	--	0.07	9.0W, 1.5Cb, 0.07Zr, 1.0V	TAPCO	2,49
Waspaloy	0.10*	0.75*	0.50*	18/21	Bal	12/15	3.5/5.0	2.75/3.25	1.0/1.5	2.0*	0.005	0.12Zr	Pratt & Whitney	14,58
Nicrotung(a)	0.08/0.13	--	--	11/13	Bal	9/11	--	3.75/4.75	3.75/4.75	--	0.02/0.08	7.0/8.5W, 0.02/0.08Zr	Westinghouse Electric	59
Cosmoloy F	0.04*	0.10*	0.10*	15	Bal	--	3.8	3.4	4.7	0.20*	0.08	2.2W, 0.07Zr	Cannon-Muskegon	51,60
F-342	0.15	0.40	0.40	15	Bal	--	5.0	--	5.0	5.0	0.3	--	Allegheny Ludlum	49
HDA-8294	0.01	0.1	0.1	15	73.5	--	5.0	--	6.7	--	--	--	Haynes-Stellite	61
NASA-Basic	--	--	--	6	Bal	--	8	--	6	--	--	1Zr	NASA	62
NASA-Ti	0.125	--	--	6	Bal	--	8	1.5	6	--	--	1Zr	Ditto	62
NASA-WV	0.125	--	--	6	Bal	--	4	--	6	--	--	1Zr, 4W, 2.5V	"	62
NASA-TaWV	0.125	--	--	6	Bal	--	4	--	6	--	--	1Zr, 8Ta, 4W, 2.5V	"	62

(a) Most frequently mentioned in the technical literature published during the period 1963 to 1965.

(b) * - indicates maximum values.

TABLE IV. SHORT-TIME TENSILE AND STRESS-RUPTURE PROPERTIES OF DOMESTIC VACUUM-CAST, NICKEL-BASE ALLOYS

Average or Typical Values.

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi			Stress-Rupture Life at 15 10^3 Psi, hr
								10-Hour Life	100-Hour Life	1000-Hour Life	
Inconel 713 C (Ref. 63)	As cast	70	125	109	8	--	--	--	--	--	--
		1400	133	107	7	--	--	--	80	72	--
		1600	110	85	10	--	--	--	43	32	--
		1700	95	70	10	--	--	--	30	22	--
		1800	75	54	15	--	--	--	20	15	--
(Ref. 45)	As cast (vacuum, argon)	1500	--	--	--	--	--	--	61, 53	--	--
		1600	--	--	--	--	--	--	45, 37	--	--
		1700	--	--	--	--	--	--	20, 12	--	--
Inconel 713 LC (Ref. 48)	As cast	70	130	109	15.3	20.9	--	--	--	--	--
		1200	157	113.9	11.0	11	--	--	--	--	--
		1500	123	99	15.0	17	--	--	62	45	--
		1700	94.2	66.9	6.0	12	--	--	29	21	--
		1800	--	--	--	--	--	--	20	15	--
Inconel 717 C (Ref. 49)	As cast	1350	--	--	--	--	--	--	97	76	--
		1500	--	--	--	--	--	--	60	44	--
		1600	--	--	--	--	--	--	42	28	--
		1800	--	--	--	--	--	--	21	13	--
Inconel 718 C (Ref. 64)	2000 F/1 hr, 1800 F/1 hr, 1325 F/16 hr, air cool each time	70	155	127	31	38	--	--	--	--	--
		1200	118	103	27	52	--	--	93	--	--
		1300	113	98	22	45	--	--	70	--	--
		1350	105	90	19	37	--	--	59	--	--
		1400	--	--	--	--	--	--	42	--	--
		1500	--	--	--	--	--	--	23	--	--
Inconel 700 (Ref. 65)	As cast	1500	--	--	--	--	--	--	39.5	--	--
		1600	--	--	--	--	--	--	25.5	--	--
		1700	--	--	--	--	--	--	19	--	--
		1800	--	--	--	--	--	--	7.5	--	--
(Ref. 50)	As cast (air, argon, vacuum)	1500	--	--	2-4,	--	--	--	37, 42, 42	--	--
		1500	--	--	3-4,	--	--	--	--	--	--
		1500	--	--	21	--	--	--	--	--	--
		1500	--	--	--	--	--	--	--	--	--
Inconel 702 (Ref. 51)	As cast	1200	--	--	--	--	--	70-80	--	--	--
		1350	--	--	--	--	--	40-50	--	--	--
		1500	--	--	--	--	--	20-30	--	--	--
		1600	--	--	--	--	--	10-20	--	--	--
		1700	--	--	--	--	--	0-10	--	--	--

TABLE IV. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi			Stress-Rupture Life at 15 10^3 Psi, hr
								10-Hour Life	100-Hour Life	1000-Hour Life	
IN-100 (Refs. 30, 45, 46, 49)	As cast	70	135	115	6	--	36 R_C	--	--	--	--
		1200	140	118	5	--	--	--	--	--	--
		1350	--	--	--	--	--	88	--	--	77
		1500	135	125	4	--	--	71	--	--	51
		1600	--	--	--	--	--	52	--	--	35
		1800	76	58	4	--	--	25	--	--	15
		1900	--	--	--	--	--	17	--	--	11
MAR-M200 (Refs. 31, 46, 52)	As cast	70	135	120	7	--	40 R_C	--	--	--	--
		1200	138	125	4	--	--	--	--	--	--
		1500	131	118	3.5	--	--	--	73	54	--
		1600	123	110	3.7	--	--	--	56	39	--
		1700	100	92	4.1	--	--	--	38.5	28	--
		1800	80	68	4.5	--	--	--	26.5	19	--
		1900	60	50	5	--	--	--	18	13	--
		2000	47	40	5.5	--	--	--	10.3	8	--
		2100	--	--	--	--	--	--	5.2	--	--
Udimet 500 (Ref. 66)	2100 F/4 hr, 1975 F/4 hr, 1400 F/16 hr, air cool each time	70	103-130	95-115	7-13	7-13	--	--	--	--	--
		1200	100-122	85-105	7-15	7-16	--	--	--	--	--
		1500	85-103	75-95	10-17	7-18	--	--	50	42	--
		1600	--	--	--	--	--	--	35	--	--
		1700	--	--	13	--	--	--	22	11	--
		1800	--	--	--	--	--	--	12	--	--
Udimet 600 (Ref. 12)	(a)	1500	--	--	--	--	--	--	53	--	--
		1600	--	--	--	--	--	--	35	--	--
		1700	--	--	--	--	--	--	24	--	--
		1800	--	--	--	--	--	--	15	--	--
Udimet 700 (Refs. 30, 45, 46)	2100 F/2 hr, 1400 F/16 hr, air cool each time	70	145	123	5.0	--	--	--	--	--	--
		1200	141	118	6.0	--	--	--	--	--	--
		1500	134	111	8.0	--	--	--	--	47	--
		1600	--	--	--	--	--	--	46	32	--
		1700	--	--	--	--	--	--	30	--	--
		1800	61	--	4.5	--	--	--	20	--	--
		1900	--	--	--	--	--	--	12.5	--	--
René 41 (Refs. 13, 46, 67)	1950 F/3 hr, rapid cool	70	120	103	8	10	37 R_C	--	--	--	--
		800	105	92	8	10	--	--	--	--	--
		1200	105	90	10	10	--	--	--	--	--
		1650	--	--	--	--	--	--	--	--	--
Astroloy (Ref. 2)	2100 F/2 hr, air cool	1500	--	--	--	--	--	--	50	--	--

TABLE IV. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi			Stress-Rupture Life at 15 10^3 Psi, hr
								10-Hour Life	100-Hour Life	1000-Hour Life	
I-1360 (Refs. 49, 68)	As cast	70	122	106	1	3.5	--	--	--	--	--
		1200	124	115	1	5	--	--	--	--	--
		1350	--	--	--	--	--	--	70	52.5	--
		1500	115	82	2.5	6.5	--	--	40.5	27.3	--
		1600	--	--	--	--	--	--	26	16.5	--
		1700	68	45	10.5	17	--	--	--	--	--
		1800	--	--	--	--	--	--	9.4	4.8	--
DCM (Refs. 45, 55, 56)	2100 F/1 hr, 1950 F/2 hr, 1550 F/4 hr, air cool each time	70	141.5	120.0	5.0	9.0	--	--	--	--	--
		1200	142.0	118.0	3.0	7.5	--	--	--	--	--
		1500	--	--	--	--	--	--	68	49	--
		1600	103.0	75.0	10.0	20.0	--	--	46	33	--
		1700	--	--	--	--	--	--	32	21	--
		1800	63.0	40.0	20.0	35.0	--	--	20	12	--
		1900	43.0	25.0	--	--	--	--	12	--	--
B&B (Ref. 49)	(a)	1600	--	--	--	--	--	--	37	--	--
SEL-1 (Ref. 49)	(a)	1200	--	--	--	--	--	--	98	78	--
		1350	--	--	--	--	--	--	78	63	--
		1500	--	--	--	--	--	--	54	39	--
		1600	--	--	--	--	--	--	38	25	--
		1800	--	--	--	--	--	--	13	7	--
SEL-15 (Ref. 49)	(a)	1200	--	--	--	--	--	--	--	110	--
		1350	--	--	--	--	--	--	95	82	--
		1500	--	--	--	--	--	--	66	43	--
		1600	--	--	--	--	--	--	47	34	--
		1800	--	--	--	--	--	--	19	11	--
GMR-235 (Refs. 57, 69)	As cast air-melted master	70	112	--	5	--	36 R_C max	--	--	--	--
		1200	110	88	3.5	10	--	--	--	--	--
		1400	100	80	5	10	--	--	52	38	--
		1500	79	60	7	10.5	--	--	37	24	--
		1600	62	41	10	14	--	--	23	15	--
		1700	50	30	20	24	--	--	15	11	--
GMR-235 (Ref. 57)	As cast, vacuum melted master	70	104	92	3	6	36 R_C max	--	--	--	--
		1200	97	81	3	10	--	--	--	--	--
		1400	98	82	3	7	--	--	--	--	--
		1500	105	78	3	7	--	--	41	28.5	--
		1600	92	63	7.5	6	--	--	28.2	19.5	--
		1700	67	43	18	27	--	--	18.5	12.3	--

TABLE IV. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi			Stress-Rupture Life at 15 10^3 Psi, hr
								10-Hour Life	100-Hour Life	1000-Hour Life	
GMR-235D (Ref. 57)	As cast vacuum-melted master	70	111	102	3.5	6	36 R_C max	--	--	--	--
		1200	112	87	6	11	--	--	--	--	--
		1400	115	85	3	--	--	--	--	--	--
		1500	120	80	3	9	--	--	48	33.5	--
		1600	105	64	7	--	--	--	31.7	23	--
		1700	75	42	12	23	--	--	20.5	16.9	--
TRW-1800 (Ref. 49)	As cast	1600	--	--	--	--	--	--	46	32	--
		1700	--	--	--	--	--	--	20	11	--
Waspaloy (Refs. 30, 58, 66)	1975 F/4 hr, 1550 F/4 hr, 1400 F/16 hr, air cool each time	70	134.5	100	12	--	30 R_C	--	--	--	--
		1200	126.5	93.6	15	--	--	--	--	--	--
		1400	--	--	--	--	--	--	58	--	--
		1500	94.7	79.5	10	--	--	--	40.5	34	--
		1600	--	--	--	--	--	--	25	17	--
		1700	--	--	--	--	--	--	12	--	--
		1800	23.2	22.2	43	--	--	--	3	--	--
Nicrotung (Refs. 49, 59, 70)	As cast	70	130	120	5	--	35-45 R_C	--	--	--	--
		1200	120	110	10.5	--	--	--	--	--	--
		1400	117	106	5	--	--	--	--	--	--
		1500	--	--	--	--	--	--	65	45	--
		1600	86	76	4	--	--	--	48	31	--
		1700	--	--	--	--	--	--	32	22	--
		1800	67	52	6	--	--	--	22	13	--
		1900	--	--	--	--	--	--	15	--	--
		2000	31	24	8	--	--	--	8	--	--
Cosmoloy F (Ref. 72)	2100 F/1 hr, air cool	1700	--	--	--	--	--	--	24	--	--
		1800	--	--	--	--	--	--	14	--	--
		1900	--	--	--	--	--	--	6.8	--	--
		2000	--	--	--	--	--	--	3.5	--	--
F-342 (Ref. 49)	As cast	1500	--	--	--	--	--	--	45	29	--
		1600	--	--	--	--	--	--	30.5	18.5	--
HDA 8294 (Refs. 4, 9, 60)	(a)	1500	--	--	--	--	--	--	--	42	--
		1600	--	--	--	--	--	--	40	27	--
		1700	--	--	--	--	--	--	26	18	--
		1800	--	--	--	--	--	--	18	--	--
NASA-Basic (Refs. 62, 71)	As cast	70	104	--	7.1	24	31 R_C	--	--	--	--
		1700	--	--	--	--	--	--	--	--	1000
		1800	39.9	--	7.2	19.4	--	--	--	--	60
NASA-Ti (Refs. 62, 71)	As cast	70	106	--	0.9	3.2	--	--	--	--	--
		1800	56.2	--	4.8	5.1	--	--	--	--	350
		1850	--	--	--	--	--	--	--	--	100

TABLE IV. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi			Stress-Rupture Life at 15 10^3 Psi, hr
								10-Hour Life	100-Hour Life	1000-Hour Life	
NASA-WV	As cast	70	113	--	1.6	0.6	--	--	--	--	--
(Refs. 62, 71)		1800	66.9	--	4.1	4.7	--	--	--	--	760
		1850	--	--	--	--	--	--	--	--	300
		1900	--	--	--	--	--	--	--	--	100
NASA-TaWV	As cast	70	134.5	--	--	--	41 R_C	--	--	--	--
(Ref. 62)		1800	80.1	--	--	--	--	--	--	--	1300
		1900	55.7	--	--	--	--	--	--	--	140
		2000	49.2	--	--	--	--	--	--	--	14
		2100	34.4	--	--	--	--	--	--	--	--

(a) Details not given.

strength (0.2 per cent offset), 3 per cent minimum elongation

(3) Hardness: Rockwell C 30 to 42.

The composition of the alloy is given in Table III and typical tensile and stress-rupture properties are presented in Table IV.

Although originally intended as an air-melted master alloy that was to be remelted and cast under argon (Ref. 44), as shown in Table IV, Inconel 713 C has improved stress-rupture properties when it is vacuum cast (Ref. 45). Room temperature impact toughness is also improved by vacuum casting. Charpy V-notch specimens gave 7.0 ft-lb for bars cast in argon and 9.4 ft-lb for bars cast in vacuum (Ref. 47). No heat treatment is required to develop these properties. In addition to high stress-rupture strength up to 1800 F, the alloy has good resistance to thermal fatigue (comparable with IN-100 (Ref. 45) and good resistance to oxidation up to 2000 F (penetration rate of 0.0181 ipy for continuous exposure and 0.0190 ipy for intermittent exposure) (Ref. 47).

Inconel 713 C melts in the temperature range of 2350 to 2450 F. It has excellent fluidity when vacuum cast. Broad sections have been run that were as thin as 0.045 inch (Ref. 73). Investment castings weighing up to 100 pounds have been readily produced with intricately cored sections (Ref. 63). An example of a casting requiring coring is shown in Figure 7.

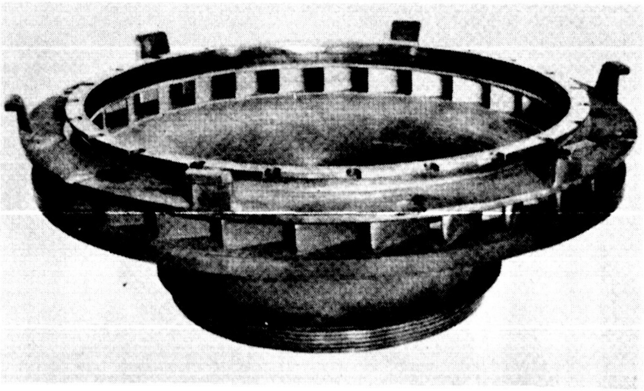


FIGURE 7. TURBINE NOZZLE INVESTMENT CAST FROM INCONEL 713 C (REF. 63)

The alloy has a moderate degree of weldability. Castings in section thicknesses up to 1/2 inch can be joined satisfactorily if restraint is kept low (Ref. 63). Successful grinding is dependent upon low heat input to the work. A wheel with low hardness and open structure is recommended (Refs. 47, 63).

For integrally cast turbine disks and buckets, such as the casting illustrated in Figure 8, the current trend is toward alloys with improved room-temperature ductility, provided that the penalty is only a small loss in high-temperature strength. Inconel 713 LC (Ref. 48), which has 0.05 per cent carbon, was developed to meet this need. Of interest is the fact that a low iron content (0.5 per cent) imparts superior mechanical properties as compared with an alloy of high iron content (1.2 per cent). The data for the Inconel 713 LC alloy in Table IV are representative of castings having low iron content.

FIGURE 8. INTEGRAL TURBINE DISK AND BUCKETS INVESTMENT CAST IN INCONEL 713 C BY AiRESEARCH DIV., GARRETT CORP. (REF. 29)

Courtesy of AiResearch, Division of Garrett Corp.

Inconel 713 LC is poured at about 3000 F, which is about 600 F superheat. This assures filling of the mold and soundness in the casting. However, the pouring temperature needs to be controlled closely because of the effect on the properties. For example, an investment mold preheated in the range of 1400 to 1900 F will produce castings with up to twice the room-temperature tensile ductility when cast with a superheat of 550 F as compared with a superheat of 250 F. Both the stress-rupture life and ductility are improved by adequate superheat in pouring, as shown by the following data (Ref. 48):

	Stress-Rupture Data			
	1700 F/30,000 psi		1800 F/22,000 psi	
Pouring Temp, F	Life, hr	Elongation, per cent	Life, hr	Elongation, per cent
2660	30.3	2.7	17.2	2.7
2760	56.8	4.0	32.3	4.0
2960	95.8	6.7	42.8	6.8

Inconel 717 C. In the as-cast condition, Inconel 717 C has stress-rupture properties similar to Inconel 713 C. It is slightly lower in chromium content, but appreciably higher in cobalt content than Inconel 713 C.

Inconel 718 C. Castings of this alloy have excellent room-temperature ductility and high strength. The alloy is recommended for applications requiring high strength and creep resistance at temperatures up to 1300 F (Ref. 46). Its weldability makes it useful in the construction of cast subassemblies such as jet-engine frames (Ref. 64).

Early master heats were air melts prepared under a slag cover and remelts were cast under argon (Ref. 74). Later it was found that heats prepared under vacuum had vastly superior properties (Ref. 75). Test bars cast in shell investment molds have a finer grain size than bars cast in flask molds. A finer grain size results in increased strength and ductility (Ref. 76). For this reason, the General Electric Company specifies that the grain size should not exceed 1/4 inch to achieve the following minimum properties (Ref. 74):

115,000 psi ultimate tensile strength
85,000 psi yield strength (0.2% offset)
5 per cent elongation
10 per cent reduction in area.

Homogenizing for 1 hour at 2000 F prior to solution annealing and aging increased the room-temperature yield strength from 110,000 psi to 122,000 psi, and the reduction in area from 18.5 per cent to 22.5 per cent (Ref. 64). A furnace-cooling aging treatment can accentuate the precipitation reaction to achieve further strengthening (Ref. 64):

	1700 F/1 Hr, Air Cool; 1325 F/16 Hr, Air Cool	1700 F/1 Hr, Air Cool; 1325 F/8 Hr, Furnace Cool in 20 Hr to 1150 F, Air Cool
Tensile strength, psi	155,100	172,700
Yield strength (0.2% offset), psi	123,200	130,300
Elongation, per cent	20.5	13.0
Reduction in area, per cent	25.0	15.0

Inconel 718 C castings have good weldability. An argon torch with backup gas is satisfactory for sections up to 1/8 inch; helium is used for thicker sections with a Hastelloy R-235 filler wire (Ref. 75). For optimum properties, welding should be followed by the solution annealing and aging heat treatment (Ref. 74).

Inconel 700. The stress-rupture data in Table IV show that the strength and ductility of Inconel 700 are improved slightly when the alloy is melted and cast under argon rather than in air. When melted under vacuum and cast under argon the strength was not changed, but the ductility was increased by a factor of six (Refs. 50, 77). The test bar data were generated from 20-pound virgin-metal charges that were melted in a stabilized zirconia crucible in an induction roll-over furnace and poured at 2800 F (270 F superheat) into silica investment molds preheated to 1600 F.

Inconel 702. This alloy is much leaner in strengthening elements than Inconel 700. As a result, its high-temperature strength is lower as evidenced by the data in Table IV for the 10-hour stress-rupture life of the cast alloy.

IN-100. This alloy was known earlier as PDRL-100 and is also known by the British designation EPK 24. Specifications call for a minimum stress-rupture life of 23 hours at 1800 F and 29,000 psi with a minimum elongation of 4 per cent (Ref. 45). Some typical average properties at elevated temperature are presented in Table IV. The alloy is usable up to 1900 F. Its resistance to thermal shock is comparable with that of Inconel 713 C.

Castings are apt to have a small amount of microporosity (Ref. 78). In certain designs where castings are subjected to extreme restraint from shrinking during solidification hot tearing may occur (Ref. 45). This characteristic is believed to be related to the low hot ductility of the alloy as revealed by the following data (Ref. 78):

Test Temperature, F	Tensile Elongation, per cent
1832	4.1
2012	1.5
2102	1.4
2192	2.0

If the design of the casting makes it susceptible to hot tearing, it may be alleviated to some extent by adjusting the boron and zirconium content to the low side of the specification (Ref. 45).

MAR-M200. Until recently, this alloy was known as SM-200. The specification requirement for this popular alloy is 23 hours minimum life at 1800 F at a stress of 29, 000 psi with a minimum elongation of 3 per cent in the as-cast condition. Under these conditions the average rupture life is about 50 hours and the average elongation is about 5.5 per cent (Ref. 52). Room-temperature tensile properties specified are 120, 000 psi ultimate strength, 100, 000 psi yield strength at 0.2 per cent offset, and 5 per cent elongation. Table IV shows the average tensile and stress-rupture values over a wide range of temperatures.

Although MAR-M200 and IN-100 have about the same chromium content, they differ markedly in their resistance to oxidation at elevated temperatures. In static air at 2000 F at about 20 hours, IN-100 showed a weight gain of 1.6 mg/cm^2 and continued to gain weight at a rate of about 0.04 mg/cm^2 per hour. After the initial 20 hours, MAR-M200 showed a weight gain of slightly under 1 mg/cm^2 with essentially no further gain in weight with further exposure time (Ref. 52).

The MAR-M200 castings normally are not heat treated. However, improved properties are obtainable in the intermediate temperature range of 1300 to 1500 F by a solution anneal and aging heat treatment such as 16 hours at 1650 F followed by 12 hours at 1550 F (Ref. 52). The properties at high service temperatures are not affected by such heat treatments.

This alloy is considered to have good casting characteristics comparable with those of other nickel-base alloys such as Inconel 713 C (Refs. 52, 79). Its castability is better than those nickel-base alloys having a higher aluminum-plus-titanium content, such as IN-100.

Pratt & Whitney Aircraft Division is currently evaluating MAR-M200 jet-engine vanes and blades produced by their newly developed casting technique (Ref. 35). Compared with the conventional castings, the directionally solidified components are claimed to have a 75 to 100 F increase in operating-temperature capability, up to 300 per cent increase in ductility, and a major improvement in resistance to thermal shock. Details of the casting process are not available. It was revealed that controlled temperature gradients during solidification produced a columnar grain structure in which the grain boundaries are aligned parallel to the axis of the applied stress. Castings prodiced by this method are designated as PWA-664 or DS-200. The technique is effective with other nickel-base alloys. Pratt & Whitney has applied for a patent on the gas-turbine hardware produced by the new process.

MAR-M200 can be welded like other precipitation-hardenable alloys of its general type. The methods that employ a protective atmosphere are the most satisfactory (Ref. 79).

Milling and broaching with high-speed tool steels is done at 15 to 20 surface feet per minute at feeds of 1 to 3 mils (Ref. 79). The feed for drilling should be 1 to 3 mils and the speed should be 15 to 25 surface feet per minute with a cobalt high-speed steel or somewhat lower with a high-speed steel such as Type M-3. Drills should have an included angle of 130 to 140 degrees and a clearance angle of 10 to 15 degrees. Mineral oil with 2.5 per cent active sulfur is a suitable coolant.

Udimet 500. Udimet 500 has high strength up to 1700 F and resists oxidation up to 1800 F (Refs. 46, 53). It is used primarily for parts such as turbine blades.

For castings in the heat-treated condition, AMS 5384 calls for a minimum tensile strength of 120,000 psi and a minimum of 7 per cent elongation when tested at 1200 F (Ref. 53). A minimum stress-rupture life of 35 hours is called for when stressed at 25,000 psi at 1700 F. Hardness is to be 30 Rockwell C or higher.

The tensile and stress-rupture properties presented in Table IV are not the most recent. They are presented because more recent data on the tensile properties over a range in temperature are not available in the literature. Presumably the data in Table IV are for castings having a fairly coarse grain; this may account for the strength and ductility appearing to be somewhat low. A small, uniform grain size is desirable in this alloy for improved response to heat treatment and improved strength and ductility. This is illustrated by the following tensile properties at 1200 F (Ref. 14):

Grain Size, in.	Ultimate Tensile Strength, 10^3 psi	Elongation, per cent	Reduction in Area, per cent
1/64	140.0	16.0	20.7
3/32	114.2	11.1	10.7

A small grain size throughout an airfoil section is obtained by using a low pouring temperature. If no controls were exercised, such as pouring temperature, mold temperature, or grain-nucleating agents incorporated in the mold, it would not be uncommon to obtain grains as large as 3/4 inch (Ref. 9).

By comparing impact properties, particularly the values at 1300 F, the General Electric Company determined whether cast turbine-blade alloys had adequate resistance to impact damage (Ref. 60). Udimet 500 castings with a grain size of 1/8 to 1/32 inch were given the normal heat treatment and then aged at 1600 F for 48 hours to approximate an airfoil during operation. Acceptable resistance to impact damage was established by the following impact values:

Test Temperature, F	Impact Toughness, ft-lb	
	Unnotched	Notched
70	15	8
1300	33	13
1600	60	17

Udimet 600 and 700. From the stress-rupture properties in Table IV, Udimet 600 is comparable with Udimet 500. Probably for this reason, Udimet 600 was not exploited as a casting alloy. No additional information is available on it.

Udimet 700 is one of the current popular casting alloys for gas-turbine blades and other parts requiring high strength up to 1800 F (Ref. 46). Its stress-rupture properties are superior to those of Udimet 500. Good risering practice must be followed to minimize the presence of microshrinkage.

In some limited cases, the General Electric Company found the following correlation of properties between test bars and turbine blades cast in the same flask (Ref. 80) (the test temperature was 1300 F for all casting):

Casting	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Stress-Rupture Life at 22 10^3 psi at 1800 F, hr
Test bar	134.5	118	4.0	10.9	43.9
Blade	114.7	112.4	2.4	6.0	28.9
Test bar	142.5	129.2	3.5	9.3	39.7
Blade	122.5	119.6	3.3	7.5	32.4

The superior ductility and strength in the tensile test and the superior stress-rupture life of the test-bar castings are probably a reflection of greater soundness in the test bars than in the blades. For example, one blade showed a stress-rupture life of 7.3 hours, and metallographic examination revealed the presence of microshrinkage.

Apparently castings with a large grain size (1/8 to 1/4 inch) have a somewhat lower fatigue endurance strength at 1500 F than castings with a fine grain size (1/32 to 1/8 inch) as indicated by the following data (Ref. 81):

Grain Size, in.	Stress, 10^3 psi	Failure, cycles
1/32 to 1/16	70.0	9,000
	65.0	9,439,000
	65.0	259,000
	62.5	30,564,000
	60.0	30,715,000 (no failure)
1/8 to 1/4	60.0	507,000
	55.0	821,000
	55.0	5,709,000
	52.5	30,153,000 (no failure)
	50.0	46,332,000 (no failure)

However, castings with a small difference in grain size are indistinguishable in the fatigue test. In a comparison of castings having a grain size of 1/32 inch and 1/16 inch, there was no significant difference in the results (Ref. 82). At a runout of 49.8 million cycles, the fatigue endurance strength at 1500 F was 32,500 psi.

Impact toughness of Udimet 700 cast turbine blades was comparable with the toughness of Udimet 500. The Udimet 700 blades had a grain size of 1/8 to 1/32 inch and were solution annealed for 2 hours at 2100 F. They were aged for 4 hours at 1600 F to simulate the condition of an airfoil during operation. In this condition, the impact values were as follows (Ref. 60):

Test Temperature, F	Impact Toughness, ft-lb	
	Unnotched	Notched
70	15	5
1300	30	10
1600	36	8

René 41. René 41 as a casting alloy is relatively new. It is recommended for structural applications requiring high strength at elevated temperatures. Specifications call for a minimum stress-rupture life of 25 hours at 1650 F at a stress of 25,000 psi. An average life of 29.7 hours is attained with an elongation of 8.8 per cent and a reduction in area of 8.7 per cent (Ref. 13). Above 1650 F, René 41 starts to oxidize at an appreciable rate.

The beneficial effect on tensile properties at 1200 F, when castings are produced with a finer grain size, is illustrated by the following data (Ref. 13):

Grain Size, in.	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent
1/16	128.0	107.2	4.5	11.9
1/8	115.0	99.0	5.4	9.1
1/8 to 3/8	102.3	96.2	3.8	7.1
3/16 to 7/16	100.6	93.7	3.5	6.3

Specification for the minimum tensile properties at 1200 F, when the casting has a maximum grain size of 1/8 inch, calls for 110 x 10^3 psi ultimate strength, 90 x 10^3 psi yield strength, 3.0 per cent elongation, and 5.0 per cent reduction in area (Ref. 13). It is said that a coarser grain size favors improvement in stress-rupture life.

René 41 has good castability and good weldability (Ref. 13). With a melting point of about 2350 F, it has excellent fluidity when poured in the range of 2650 to 2700 F. Thin sections can be run readily. Hot tearing is not a problem when casting in shell investment molds because the friability of this type of mold helps to reduce strains in the casting. The low boron content of the alloy is also helpful in minimizing hot shortness. Another reason that a shell investment mold is desirable is that it does not retard the cooling of the casting as much as a flask investment mold. Slow cooling should be avoided because precipitation may occur at the grain boundaries and cause a slight detrimental effect on the properties (Ref. 13).

Astroloy. Although originally developed as a wrought alloy, Astroloy has been investment cast satisfactorily into complex shapes (Ref. 54). Castings and sheet material of this alloy can be joined in the annealed condition by inert-arc fusion welding with Hastelloy X wire as the filler. Annealing of castings consists of heating for 2 hours at 2100 F and air cooling. In this condition, cast test bars have shown the following values for stress-rupture life and reduction in area at 1800 F under a stress of 20,000 psi: 78.7 hours and 4.7 per cent; 130.1 hours and 5.6 per cent; 31.3 hours and 5.0 per cent; and 39.7 hours and 7.0 per cent.

The alloy was considered to be suitable for use as turbine blades and hardware type of castings for fabricated structures (Refs. 2, 54). Cast blades heated for 48 hours at 1600 F after the solution-anneal heat treatment had acceptable resistance to impact damage as judged by the following data:

Test Temperature, F	Impact Toughness, ft-lb	
	Unnotched	Notched
70	35	5
1300	43	11
1600	45	14

I-1360. This alloy was developed in about 1949 and, until recently, was known as Guy alloy. It now has a slightly lower chromium content than its original version. It is used for structural parts at high temperatures.

Neither the tensile nor the stress-rupture properties were affected by induction melting under an argon blanket as compared with melting under vacuum and pouring under a partial pressure of argon (Ref. 68). This is shown by the data on the following page.

Melting Method	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	100-Hr Stress-Rupture Life	
						Stress, 10^3 psi	Elongation, per cent
Argon	70	128	105	1	4	--	--
Vacuum-argon	70	115	107	1	3	--	--
Argon	1200	128	115	1	6	--	--
Vacuum-argon	1200	120	115	1	4	--	--
Argon	1350	--	--	--	--	68	4
Vacuum-argon	1350	--	--	--	--	70	5
Argon	1500	113	78	3	7	48	8
Vacuum-argon	1500	117	85	2	6	45	8
Argon	1600	--	--	--	--	34	11
Vacuum-argon	1600	--	--	--	--	30	10
Argon	1700	70	47	11	18	21	10
Vacuum-argon	1700	65	43	10	16	20	12
Argon	1800	--	--	00	00	12	13
Vacuum-argon	1800	--	--	--	--	13	12

The data in Table IV are the average values for the above heats melted under argon and in a vacuum-argon atmosphere.

The above data probably reflect that great care was exercised during melting and pouring under argon. In contrast, the following stress-rupture data at 1500 F show that melting under vacuum can be superior to melting under argon and that melting under argon can be as inferior as melting in air (Ref. 77).

Melting Method	100-Hr Stress-Rupture Life: Stress, 10^3 psi	100-Hr Stress-Rupture Life: Elongation, per cent
Air	42	3
Argon	41	1-3
Vacuum-argon	56	7-10

The nitrogen content of the I-1360 alloy has a potent effect on the stress-rupture ductility (Ref. 77). An increase in nitrogen decreased the elongation as shown by the following data:

Nitrogen Content, per cent	100-Hr Stress-Rupture Test at 1500 F, elongation, per cent
0.01	6-7
0.02	5-6
0.03	4-4.5
0.07	2

A marked difference in resistance to oxidation was noted between heats melted under argon and those melted under vacuum and poured under a partial pressure of argon (Ref. 68). The tensile and stress-rupture properties of the two heats were very similar. The compositions also were very similar, including the gaseous elements that were 0.028 per cent nitrogen, 0.0001 per cent hydrogen, and 0.0001 per cent oxygen in both heats. However, as shown below, the vacuum-melted heat was superior in resistance to oxidation in air at 2000 F:

Exposure Method	Melting Method	Weight Gain, mg/in.^{2}: 100 Hr	200 Hr	400 Hr
Continuous	Argon	28	65	82
	Vacuum-argon	4	4	4
Cycled to room temperature	Argon	50	68	146
	Vacuum-argon	34	44	81

For reliable reproducibility of optimum properties and freedom from surface and internal inclusions, castings of I-1360 should be vacuum melted. It was claimed that castings contained gas porosity unless the vacuum melt were poured under a partial pressure of argon (Ref. 77). Although an investment mold was used at a preheat temperature of 1600 F, no details on mold material or method of manufacture of the mold were given. Presumably it was a silica mold because it has been found (Ref. 16) that metal-mold reaction between nickel-base alloys and silica is inhibited by casting under argon.

DCM. The oxidation and corrosion resistance with high strength at elevated temperatures make the DCM alloy suitable for turbine blades and jet-engine components (Ref. 55). Aside from the fact that this alloy must be vacuum melted, the most significant variable that affects its properties is the as-cast grain size (Ref. 56). Figure 9 shows the quantitative relationship that exists between the grain size and the stress-rupture life at a stress level of 15,000 psi at 1800 F. The effect is quite marked. For example, for the above conditions, if the average grain diameter is increased from 1/32 to 1/8 inch, the stress-rupture life is increased from about 200 to 400 hours.

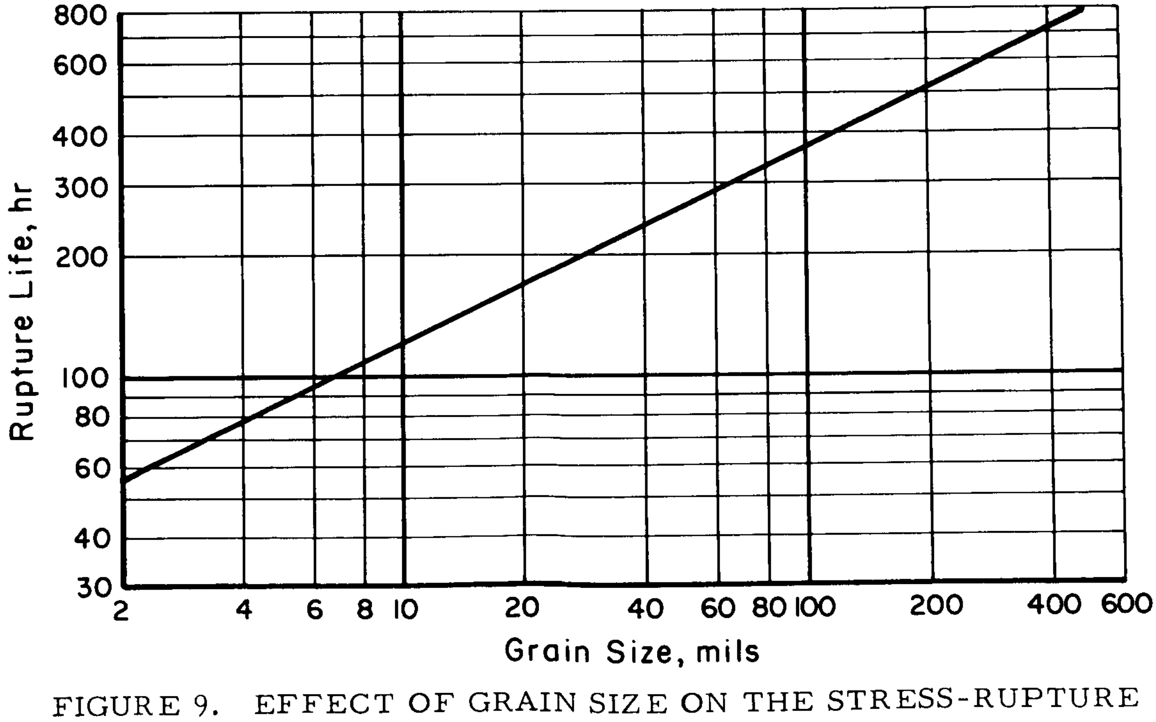


FIGURE 9. EFFECT OF GRAIN SIZE ON THE STRESS-RUPTURE LIFE OF DCM ALLOY AT 1800 F AND AT A STRESS OF 15,000 PSI (REF. 56)

In creep tests at 1800 F, the strength at 0.2 per cent plastic strain was 9000 psi (Ref. 56). The data were obtained with bars having an exceptionally fine grain size. A higher creep strength would be expected for bars having larger grain size.

Most of the turbine-blade alloys show a drop in fatigue strength as the test temperature approaches 1600 F. The DCM alloy is unusual in this respect. Its pneumatic fatigue strength in completely reversed bending is essentially constant at 35,000 psi through the temperature range of 1300 to 1700 F (Ref. 56).

In tests performed by the General Electric Company, turbine blades of the DCM alloy did not exhibit acceptable resistance to impact damage as judged by the low values presented below for impact toughness (Ref. 60).

Test Temperature, F	Impact Toughness, ft-lb	
	Unnotched	Notched
70	5	2
1300	5	3
1600	8	3

The castings evaluated had a grain size of 1/8 to 1/32 inch and were solution annealed for 1 hour at 2100 F, air cooled, and then heated for 48 hours at 1600 F to simulate temperature conditions during operation of an airfoil.

Castings are machinable in all conditions, but are usually machined best in the fully age-hardened state (Ref. 55). Rugged machine tools and low speeds should be used because this alloy is similar to annealed high-speed steels in machinability. The DCM alloy is considered to be nonweldable.

B & B. Only one reference noted that this was a casting alloy (Ref. 49). In addition to the composition given in Table III, the only additional information available is that the alloy has a 100-hour stress-rupture strength of 37,000 psi at 1600 F.

SEL-1 and 15. The limited amount of published data on these two turbine-blade alloys deals with the stress-rupture properties that are presented in Table IV. SEL-15 is the stronger of these two alloys.

GMR-235 and 235D. These two alloys are used for jet-engine parts such as turbine wheels, vanes, and blades operating at temperatures in excess of 1400 F (Ref. 57). Originally the master heat of the GMR-235 alloy was air melted and remelted and cast under argon. Later it was found that vacuum melting of the master alloy improved the properties (Refs. 4, 5, 9, 65, 77). The manganese was then reduced from a maximum of 0.25 to 0.10 per cent and the silicon from 0.60 to 0.30 per cent. Also the boron content was changed from the range of 0.025-0.07 to 0.05-0.10 per cent.

The next change for still further improvement in properties was the development of the GMR-235D alloy, which has half of the specified iron content of the GMR-235 alloy and a slight increase in the content of aluminum and titanium. The vacuum-melted master alloys of GMR-235 and 235D may both be remelted and cast under argon. The effect of the master alloy melting practice and composition of the two types of alloy on the stress-rupture life is shown below (Ref. 57):

Alloy	Master Heat-Melting Method	Remelting and Casting Method	Stress-Rupture Life at 1600 F and 28,000 Psi, hr
GMR-235	Air melted	Arc-argon	40
	Vacuum melted	Arc-argon	108
GMR-235D	Vacuum melted	Arc-argon	160

Vacuum casting of the vacuum-melted GMR-235 master alloy is said to have little effect on the properties as compared with remelting and casting under a good cover of argon (Ref. 57). However, vacuum casting as compared with air or argon melting and casting of a virgin charge (equivalent to an air-melted master) may improve the ductility markedly, as illustrated by the following data (Ref. 77):

Melting and Casting Atmosphere	100-Hr Stress-Rupture Properties at 1500 F	
	Stress, 10^3 psi	Elongation, per cent
Air	41	6-9
Argon	41	3-7
Vacuum	42	14-19

In a thermal-fatigue test, a flame heated the leading edge of cast blades and an air jet then cooled the blades. In a schedule of 300 cycles at 2000 F, 100 cycles at 2050 F, and then 100 cycles at 2100 F, 70 per cent of the conventionally cast GMR-235D blades survived, whereas the survival was 100 per cent when the GMR-235D blades were vacuum cast (Ref. 4). In this test, the blades had been dip coated with aluminum and diffusion heat treated.

The boron content has a potent effect on the properties of the GMR-235 alloy (Ref. 69). Both the stress-rupture life and ductility increase and the room-temperature ductility decreases as the boron content is increased to about 0.1 per cent. Beyond this content, the stress-rupture properties decrease and the casting approaches a state of brittleness at room temperature.

Castings of GMR-235 and 235D are normally used in the as-cast condition. Solution heat treatment will increase the stress-rupture life as shown by the following data (Ref. 4):

	Stress-Rupture Life at 1600 F and 28,000 Psi, hr			
	GMR-235		GMR-235D	
Master Heat-Melting Method	As Cast	Heat Treated	As Cast	Heat Treated
Vacuum melted	155	170	220	340
Air melted	65	140	--	--

The heat treatment consisted of a solution anneal of 3 hours at 2050 F for GMR-235 test bars and 2 hours at 2100 F for GMR-235D test bars (Ref. 57). Precipitation strengthening occurs during air cooling from the solution-anneal temperature; an aging treatment is not necessary. The increase in strength is accompanied by some loss in ductility. For larger cast sections in both of the alloys, a solution anneal at 2150 F may be necessary to obtain room-temperature properties comparable with cast test bars. To avoid intergranular oxidation, heat treatment must be conducted in a vacuum or in a protective atmosphere. The latter should be either neutral or slightly reducing.

Average tensile and stress-rupture properties are given in Table III for both alloys cast under argon. The specifications for minimum properties are as follows (Ref. 57):

			Tensile Properties		Stress-Rupture Properties		
Alloy	Master Heat-Melting Method	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Elongation, per cent	Stress, 10^3 psi	Life, hr	Elongation, per cent
GMR-235	Air melted	1500	73.5	3.0	35	75	5
GMR-235	Vacuum melted	1600	--	--	28	60	10
GMR-235D	Vacuum melted	1600	--	--	35	30	10

There is essentially no difference in the impact toughness of castings poured under argon from the GMR-235 air-melted master alloy and the GMR-235D vacuum-melted master alloy. This is shown by the following data (Ref. 57):

Test Temperature, F	Charpy Impact Strength (V Notch), ft-lb	
	GMR-235	GMR-235D
1000	8	7
1200	7	7
1350	7	6
1500	7	8
1600	8	7
1700	10	10

TRW-1800. The TRW-1800 alloy is used for jet-turbine blades. It is used in the as-cast condition. The following data on the impact toughness of cast blades were deemed to indicate adequate resistance to impact damage, even though they were somewhat inferior to Udimet-500 blades (Ref. 60):

Test Temperature, F	Impact Toughness, ft-lb	
	Unnotched	Notched
70	19	4
1300	21	5
1600	25	8

Before testing, the blade material was heated at 1600 F for 48 hours to approximate the service of an airfoil.

Waspaloy. As shown by the data in Table IV, Waspaloy loses its strength rapidly above 1500 F. Vacuum casting was applied to this alloy when it was learned that it improved the properties of the wrought alloy. As illustrated by the following data, the ductility of castings is doubled by vacuum melting as compared with melting under argon in an indirect-arc furnace (Refs. 5, 66):

Melting Condition	100-Hr Stress-Rupture Life at 1500 F		
	Stress, 10^3 psi	Elongation, per cent	Reduction in Area, per cent
Vacuum	40.5	15	16
Indirect arc, argon blanket	40	7	8

Ductility is higher and the values are more consistent when Waspaloy is vacuum cast because of the absence of oxide films and inclusions (Ref. 66).

Nicrotung. Nicrotung was developed with the objective of providing an alloy that has a 100-hour stress-rupture strength of 20,000 psi at 1800 F. The data in Table IV show that this goal was reached. No heat treatment is required. Minimum stress-rupture properties of investment castings are (Ref. 67):

Test Temperature, F	Stress, 10^3 psi	Minimum Rupture Time, hr	Minimum Elongation, per cent
1700	35	30	3
1800	25	30	4
1900	15	30	5
2000	10	30	6

Good high-temperature strength and heat and oxidation resistance make Nicrotung castings suitable for use as highly stressed parts at elevated temperatures up to 2000 F in applications such as gas turbines. Nicrotung is used for items such as specimen holders and adapters in creep-rupture tensile-testing machines. The room-temperature ductility is moderate but adequate; the fatigue strength is 37,000 psi at 1700 F and 27,000 psi at 1800 F (Ref. 59).

This alloy has good castability (Ref. 59). Optimum properties are obtainable by melting and casting either under vacuum or a good blanket of argon when the indirect-arc furnace is used (Ref. 70). Poorer and unpredictable properties result from melting and casting in air.

It is important to have the carbon, boron, and zirconium contents within the specified limits. When all three of these elements are present in the proper amount, the strength and ductility are much improved. This is illustrated by the following stress-rupture data at 1800 F and 25,000 psi (Ref. 70):

Heat No.	Content, per cent			Rupture Time, hr	Elongation, per cent
	Carbon	Boron	Zirconium		
1	--	--	--	(a)	(a)
2	--	--	0.05	2.3	2.0
3	--	0.05	0.05	24.2	2.9
4	0.10	--	--	18.8	2.4
5	0.10	0.05	--	22.5	2.0
6	0.10	--	0.05	23.9	2.2
7	--	0.05	--	13.6	2.5
8	0.10	0.05	0.05	71.8	4.6

(a) Bars were broken when removed from the mold.

Machining is done with carbide tools at low speeds and feeds (Ref. 59). Finish machining is best done by grinding.

Cosmoloy F. From the composition given in Table III, it is presumed that the high titanium and aluminum contents require that this alloy be melted and cast under vacuum. The stress-rupture properties in Table IV are for cast bars having an average grain size of 1/32 to 1/8 inch (Ref. 72).

In evaluating various cast-turbine-blade alloys for resistance to impact damage, the General Electric Company found that Cosmoloy F was comparable with Udimet 500, which has acceptable toughness. The values presented below show that the castings with the larger grain size had higher impact toughness, but only when in the unnotched condition (Ref. 60):

Test Temperature, F	Grain Size, in.	Impact Toughness, ft-lb	
		Unnotched	Notched
70	1/32-1/8	34	6
70	1/4-1/2	78	9
1300	1/32-1/8	46	9-1/2
1300	1/4-1/2	60	10
1600	1/32-1/8	41	10
1600	1/4-1/2	50	12

In the above tests, the castings were solution annealed for 1 hour at 2100 F and air cooled. Then they were aged for 48 hours at 1600 F to approximate the service of an airfoil.

F-342. This is an experimental casting alloy. Although it does not contain titanium, its high aluminum content (5 per cent) probably requires melting and casting to be done in vacuum for consistent optimum properties. Table IV presents the stress-rupture properties at 1500 and 1600 F.

HDA 8294. The Haynes Development Alloy (HDA) was announced in 1958 as a vacuum-cast alloy (Ref. 9). The surprisingly simple composition (see Table III) resulted in stress-rupture strengths (see Table IV) exceeding those for Udimet 500. The literature does not reveal why this alloy failed to become popular.

NASA Alloys. The four NASA casting alloys listed in Table III are modifications to improve the properties of the basic composition that in itself appeared to have adequate strength and oxidation resistance for turbine blades and high-temperature structural applications. As shown by the data in Table IV, the strength of the NASA alloys increases as the alloying content is increased. At 15,000 psi, the stress-rupture life of NASA-WV exceeds that of cast Udimet 700 or Nicrotung at all temperatures of interest (Ref. 71). Only the MAR-M200 alloy matches the stress-rupture life of the NASA-TaWV alloy stressed at 15,000 psi (Ref. 62). The NASA-Basic alloy is useful up to 1800 F, and the NASA-TaWV, which is the one that is most highly alloyed, is useful up to 2000 F (Ref. 62).

Except for NASA-Basic, this series of alloys has low room-temperature ductility. However, room-temperature toughness rather than ductility was used as the criterion for acceptibility (Ref. 71). This was based on a previous study that showed cast blades having an impact toughness as low as about 10 to 16 in-lb would perform satisfactorily in engines. The data presented below for three of the alloys show that they exceed 23 in-lb and, therefore, have adequate toughness (Ref. 71):

Alloy	Izod Impact Toughness, in-lb
NASA-Basic	51.4, 23.0, >62.5, <62.5
NASA-Ti	>62.5, 45.0, 40.2, 29.0, 36.5, 41.7
NASA-WV	>63.5, >62.5

In the development of these alloys, virgin melts were prepared under an argon blanket in an induction furnace (Ref. 62). The alloys froze in the temperature range of 2400 to 2500 F and were poured at 3100 F. Test bars were cast in both silica and zirconia shell investment molds (at 1600 F) with equally excellent finish. Sectioning of the castings was done with an abrasive cutoff wheel. The alloys are machinable with tungsten carbide cutting tools. Grinding with a silicon carbide wheel is the preferred method of finishing.

Of interest is the fact that the series of NASA alloys produced good properties in the as-cast condition when melted and cast under argon. Direct comparison with vacuum-cast material has not been made. Vacuum casting might be expected to improve the ductility of the alloys.

DOMESTIC AIR-MELTED ALLOYS

The compositions of the domestic air-melted alloys are presented in Table V, and typical tensile and stress-rupture data are summarized in Table VI. Further characterization of the alloys is presented in the following discussion of each alloy.

Monel Alloys. Monel, "H" Monel, and "S" Monel are nickel-base alloys containing essentially 30 per cent copper and 1.5, 3, or 4 per cent silicon. As a class they have fairly high room-temperature strength, arc resistance to galling in sliding contacts, and are resistant to sulfur-free environments and acid solutions other than strongly oxidizing ones. They are used as bearings, pumps, and valves in the chemical-, pulp-, and food-processing industries and in marine applications (Refs. 83-86).

The Monels are sand cast in the range of 2650 to 2800 F. Magnesium is the preferred deoxidizer. Solidification shrinkage is 1/4 inch per foot. All three of the alloys are used in the as-cast condition. However, "S" Monel can be softened by annealing and can be rehardened by aging (Ref. 86):

Condition	Ultimate Tensile Strength, 10^3 psi	Elongation, per cent	Hardness, BHN
As cast	120-145	4-1	275-350
1600 F/1 hr, air cool to 1200 F, water quench	100-125	15-5	175-270
1100 F/4-6 hr, air or furnace cool	120-145	4-1	300-380

TABLE V. COMPOSITION OF DOMESTIC AIR-MELTED, HEAT- OR

Per Cent

Alloy	C	Mn	Si	Cr	Ni	Co	Mo	Ti
Monel	0.35*(a)	1.5*	2.0*	--	62/68	--	--	--
"H" Monel	0.30*	1.5*	2.7/3.7	--	61/68	--	--	--
"S" Monel	0.25*	1.5*	3.5/4.5	--	60 min	--	--	--
CY-40 (Inconel)	0.40*	1.5*	3.0*	14.0/17.0	Bal	--	--	--
Inconel X	0.08*	0.3/1.0	0.50*	14.0/16.0	70 min	--	--	2.25/2.7
NA22H	0.50	1.3	1.0	27	48	--	--	--
Thetaloy (PWA-651)	0.38	2.5	--	25	Bal	12.5	3.0	--
Supertherm	0.50	--	--	26	35	15	--	--
HOM	0.40	--	--	26	36	--	--	--
Hastelloy B	0.12*	1.0*	1.0*	1.0*	Bal	2.5*	26.0/30.0	--
Hastelloy C	0.15*	1.0*	1.0*	15.5/17.5	Bal	2.5*	16.0/18.0	--
Hastelloy D	0.12	1.0	9.0	--	Bal	2.5*	--	--
Hastelloy F	--	--	--	22	47	--	7	--
Hastelloy N	--	--	--	7	70	--	17	--
Hastelloy X	0.20*	1.00*	1.00*	20.50/23.00	Bal	0.50/2.50	8.00/10.00	--
HU	0.35/0.75	2.00*	2.50*	17/21	37/41	--	0.5*(c)	--
HW	0.35/0.75	2.00*	2.50*	10/14	58/62	--	0.5*(c)	--
HX	0.35/0.75	2.00*	2.50*	15/19	64/68	--	0.5*(c)	--
Illium B	--	--	(b)	28	50	--	8.5	--
Illium G	--	--	--	22.5	56	--	6.5	--
Illium 98	--	--	--	28	55	--	8.5	--

(a) * - indicates maximum values.
(b) Four grades: 2.5/4.5Si, 4.5/5.7Si, 5.7/6.1Si, 6.1/6.3Si.
(c) Not intentionally added.

CORROSION-RESISTANT NICKEL-BASE CASTING ALLOYS

Al	Fe	Other	Probable Developer	Reference
0.50*	2.5*	26/33Cu	INCO	83
0.50*	2.5*	27/33Cu	Ditto	84
0.50*	2.5*	27/31Cu	"	85
--	11.0*	1.25Cu	"	87
0.4/1.0	5.0/9.0	0.7/1.0Cb	"	58
--	Bal	6.0W	Blaw-Knox	49
--	--	7.0W	Pratt & Whitney	49
--	Bal	5W	American Brake Shoe	88
--	Bal	--	Duraloy Co.	88
--	4.0/7.0	0.03*S, 0.20/0.6V	U. C. Stellite	89
--	4.5/7.0	3.75/5.25W, 0.20/0.6V	Ditto	58,90,91
--	1	3.0Cu	"	49
--	17	--	"	92
--	5	--	"	92
--	17.00/20.00	0.20/1.00W, 0.040*P, 0.030*S	"	93
--	--	0.04*P, 0.04*S	--	94,95
--	--	0.04*P, 0.04*S	--	94,95
--	--	0.04*P, 0.04*S	--	94,95
--	--	5.5Cu	Stainless Foundry and Engineering	96
--	--	6.5Cu	Ditto	96
--	--	5.5Cu	"	96

TABLE VI. SHORT-TIME TENSILE AND STRESS-RUPTURE PROPERTIES OF DOMESTIC AIR-MELTED, HEAT- OR CORROSION-RESISTANT NICKEL-BASE ALLOYS

Average or Typical Values.

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi	
								100-Hour Life	1000-Hour Life
Monel (Ref. 83)	As cast$^{(a)}$	70	65-90	32-40	25-45	--	125-150 BHN	--	--
"H" Monel (Ref. 84)	As cast$^{(a)}$	70	100-130	60-80	10-25	--	240-290 BHN	--	--
"S" Monel (Ref. 85)	As cast$^{(a)}$	70	110-145	80-115	1-4	--	275-350 BHN	--	--
CY-40 (Refs. 87, 97)	As cast$^{(a, b)}$	70	70-95	30-45	10-30	--	160-190 BHN	--	--
Inconel X (Refs. 58, 69)	Heat treated$^{(b, c)}$	70	105	85	4	6	29 R_C	--	--
		1500	--	--	--	--	--	28	--
NA22H (Ref. 98)	1900 F/3-5 hr Slow cool$^{(a, b)}$	70	64.5	--	3.5	2.7	--	--	--
		1600	29.5	--	26.5	37.9	--	--	--
		1800	18.0	--	32.0	48.0	--	5.2	3.6
		1900	--	--	--	--	--	3.8	2.4
		2000	--	--	--	--	--	2.8	1.7
		2100	--	--	--	--	--	2.1	0.8
		2200	--	--	--	--	--	1.0	0.5
Thetaloy (Ref. 18)	As cast$^{(a, b)}$	70	75	--	3	--	--	--	--
		1500	55	--	10	--	--	--	--
Supertherm (Ref. 100)	--$^{(c)}$	1800	--	--	--	--	--	8500	5000
		1900	--	--	--	--	--	6000	3500
		2000	--	--	--	--	--	4000	2500
		2100	--	--	--	--	--	2600	1500
		2200	--	--	--	--	--	1000	420
		2300	--	--	--	--	--	450	--
HOM	--$^{(d)}$	--	--	--	--	--	--	--	--

TABLE VI. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi: 100-Hour Life	Rupture Stress, 10^3 psi: 1000-Hour Life
Hastelloy B (Refs. 92, 99)	As cast	70(a)	90	50	10	--	93 R_B	--	--
		70(b)	85	53.5	12	15	93 R_B	--	--
		1000(b)	77	--	15	15	--	--	--
		1200(b)	65	--	15	20	--	--	--
		1500(b)	58	--	19	20	--	13.5(e)	10.5(e)
Hastelloy C (Refs. 92, 99)	As cast	70(a)	80	50	9	--	93 R_B	--	--
		70(b)	89	52	11	12	91 R_B	--	--
		1300(b)	--	--	--	--	--	38	30
		1500(b)	55	--	19	17	--	20	15
		1700(b)	--	--	--	--	--	10	6
Hastelloy D (Ref. 92)	As cast(a)	70	115	115	1	1	30-39 R_C	--	--
Hastelloy F (Ref. 92)	As cast(a)	70	73	37	20	--	83 R_B	--	--
Hastelloy N (Ref. 92)	As cast	70(a)	86.8	40.2	22.0	--	--	--	--
		70(b)	86.4	37.3	16.8	--	--	--	--
Hastelloy X (Ref. 92)	As cast	70(a)	65	42	11.0	--	89 R_B	--	--
		1200(a)	53	--	--	--	--	--	--
		1500(a)	47	--	--	--	--	--	--
		1600(a)	42	--	--	--	--	--	--
		1800(a)	27	--	--	--	--	--	--
		70(b)	67	46.5	17.1	--	87 R_B	--	--
		1200(b)	50	--	--	--	--	32	27
		1500(b)	45	--	--	--	--	15	9
		1600(b)	40	--	--	--	--	12	7.5
		1800(b)	21	--	--	--	--	6	3
HU (Ref. 95)	As cast(a)	70	70	40	9	--	170 BHN	--	--
		1400	40	--	--	--	--	15	--
		1600	19.6	--	20	--	--	8	6
		1800	10	6.2	28	--	--	4.5	2.9
HW (Ref. 95)	As cast(a)	70	68	36	4	--	185 BHN	--	--
		1400	32	23	--	--	--	10	7.8
		1600	19	15	--	--	--	6	4.5
		1800	10	8	40	--	--	3.6	2.6

TABLE VI. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi: 100-Hour Life	Rupture Stress, 10^3 psi: 1000-Hour Life
HX	As cast(a)	70	65	36	9	--	176 BHN	--	--
(Ref. 95)		1400	--	--	--	--	--	13	--
		1600	20.5	17.5	48	--	--	6.7	4
		1800	10.7	6.9	40	--	--	3.5	2.2
		2000	--	--	--	--	--	1.7	0.9
Illium B1 (Ref. 96)	As cast(a)	70	61-67	50-62	1.0 - 4.5	2.5 - 3.5	200-240 BHN	--	--
B2	As cast(a)	70	58-65	58-62	0.5 - 1.5	2.5 max	240-290 BHN	--	--
B3	As cast(a)	70	51-57	51-57	0.5 max	Nil	290-325 BHN	--	--
B4	As cast(a)	70	45-51	45-51	0.5 max	Nil	325-360 BHN	--	--
Illium G (Ref. 96)	As cast(a)	70	68	38.9	7.5	11.3	168 BHN	--	--
Illium 98 (Ref. 96)	As cast(a)	70	54	41	18	22	160 BHN	--	--

(a) Sand casting.
(b) Investment casting.
(c) Details not given.
(d) Information not found in published literature.
(e) Heat treatment: 2125 F/1 hr, 1700 F/72 hr, air cooled each time.

In this series of alloys, "S" Monel has the highest resistance to seizing in sliding applications. It retains its room-temperature hardness up to a temperature of 1100 F (Ref. 85).

Joining of these alloys is readily accomplished by soldering, brazing, and welding (Refs. 83-85). The solder must be phosphorus-free and used with an acid flux. The braze material must also be free of lead and used with a fluoride-base flux. In both methods, the flame is slightly reducing. In welding, a flux-coated electrode of Monel is used. A special weldable grade of Monel has columbium added to the basic composition. The nominal composition of the weldable grade is 0.15 C, 1.6 Si, 63 Ni, 2.5 Fe, 31 Cu, 1.5 Cb (Ref. 81).

CY-40. Recently, the Alloy Casting Institute listed CY-40 as the cast equivalent of the wrought alloy, Inconel 600 (Ref. 101). Previously the cast grade was simply called Inconel. It is used for its good strength and its inertness in the food, dairy, chemical, aeronautical, and nuclear industries. In corrosive vapors it resists attack at elevated temperatures (Ref. 95). In an oxidizing sulfurous atmosphere, the top service temperature is 1500 F. The top service temperature is 1000 F in a reducing sulfurous atmosphere, 2000 F in an oxidizing sulfur-free atmosphere, 2100 F in a reducing sulfur-free atmosphere, and 1800 F in steam (Ref. 97).

Magnesium is the preferred deoxidizer and the melt is sand or investment cast (Refs. 39, 102) in the range of 2800 to 2950 F (Ref. 92). Castability in investment molds is considered to be good with respect to shrinkage and resistance to hot tearing, and is excellent with respect to fluidity (Ref. 102).

Castings are machinable, but considerable heat is generated. Therefore, high-speed tools should be used with an adequate cooling lubricant such as a sulfur-base oil (Ref. 97). Joining is accomplished by the conventional welding, brazing, and soldering techniques.

Inconel X. The composition of Inconel X is given in Table V and the meager information on the properties is listed in Table VI. It is among the common investment-cast alloys (Ref. 39). The small amount of information on the properties identifies it as belonging in the class of superstrength alloys.

NA22H. NA22H is a heat-resistant casting alloy. Its high structural stability and good resistance to oxidation persists up to 2200 F (Refs. 88, 98). These characteristics, plus the high resistance to thermal shock and cyclic heating, make this alloy useful for furnace fixtures and parts. It is also usefully resistant to attack by carburization or molten cyanide salts. It will resist the most difficult range of acid corrosion in which both acidic and oxidizing environments are encountered.

In addition to the properties listed in Table VI, the creep properties of the NA22H alloy are as follows (Ref. 98):

Temperature, F	Limiting Stress for Minimum Creep Rate of 0.0001 Per Cent Per Hour, psi
1800	2300
1900	1500
2000	1050
2100	450
2220	250

Castings are stress relieved by soaking at 1900 F and then slowly cooled. A surface oxide will form unless an inert or reducing atmosphere is used. The oxide can be removed by scrubbing during pickling in a solution of 10 per cent nitric acid and 2 per cent hydrofluoric acid (Ref. 98).

The machining characteristics are similar to those of cast stainless steel (Ref. 98). It is important to have the tool cutting into the metal at all times to avoid work hardening of the surface.

The weldability is good (Ref. 98). Coated rod of the same composition is used for arc welding. In gas welding, the flame should contain a slight excess of acetylene to produce a slightly reducing condition around the weld pool. Thin sections need not be preheated. With heavy sections, it is desirable to use a low heat input with frequent peening to relieve stresses.

Thetaloy. Only the meager data presented in Table V have been found on this alloy in the cast form. It belongs in the class of heat-resistant alloys such as NA22H. It is sometimes identified by the designation PWA-651 (Ref. 39).

Supertherm. Supertherm can be cast by the conventional foundry techniques including the centrifugal process (Ref. 100). It is used for furnace components because of its resistance to oxidation, low creep rate, and high stress-rupture properties at temperatures above 1800 F. In some applications it has been used at a service temperature of 2300 F, but at a low stress level. The alloy receives its strength by precipitation hardening and, therefore, has ductility values below 10 per cent.

HOM. This alloy is in the same class as Supertherm. Properties for HOM castings were not found in the literature.

Hastelloy B, C, D, F, N, and X. As a class, the Hastelloys have excellent castability (Ref. 103). Castings are produced in green or dry sand, in resin-bonded shell molds, and in investment molds (Refs. 92, 104, 105). Patternmaker's shrinkage is 3/16 inch per foot for Hastelloy D and 1/4 inch per foot for the other alloys. The castings are heat and corrosion resistant. They are not hardened by heat treatment, have fair machinability, and are weldable. Composition limits are quite broad.

The Hastelloy castings normally are not heat treated. For maximum ductility, corrosion resistance, and machinability, the following treatments are recommended (Ref. 92):

Hastelloy	Heat Treatment
B	Soak at 2150 F, rapid air cool or water quench
C	Soak at 2225 F, water quench
D	Charge in a cold furnace, heat to and soak at 1850 F, furnace cool
F	Soak at 2150 F, rapid air cool or water quench
N	Soak at 2165 F, rapid air cool or water quench
X	Soak at 2150 F, rapid air cool or water quench

The solution temperature for Hastelloy F should not be exceeded because the strength and corrosion resistance may be impaired.

Hastelloy B has high-temperature strength. It may be used at up to 1400 F in an oxidizing atmosphere and at a still higher temperature in a reducing atmosphere (Ref. 92). It is resistant to nonoxidizing acids and salts. The room-temperature Izod impact strength is about 18 ft-lb for sand castings and 13 ft-lb for investment castings. Recommended methods for joining are electric-resistance welding and metal-arc welding.

Investment castings of Hastelloy B are covered by AMS-5396 (Ref. 89). The specified minimum requirements are as follows:

	Room Temperature	1500 F
Tensile strength, psi	75,000	53,000
Yield strength (0.2% Offset), psi	50,000	--
Elongation, per cent	10	10
Hardness, BHN	180-235	--

When heats are prepared by arc melting, bend tests are required. Three bars measuring at least 0.090 inch in diameter or thickness and about 2 inches in length are produced in the same mold as the production casting. At least two of the bars from a given mold must not crack when they are bent at room temperature through an angle of 30 degrees around a 0.5-inch-diameter mandrel.

The AMS 5396 states that investment castings are to be poured from a remelted master heat or directly from a master heat. A master heat is not to exceed 7000 pounds in weight. Foundry returns must not be remelted for casting unless refined as a master heat. The purchaser may permit blending of master heats to form a master heat lot. Tensile specimens cast to represent the master heat are to have a 0.25-inch reduced section and are to be cast in the same type of mold and under the same conditions as the commercial castings.

Hastelloy C has good strength up to 1500 F and is resistant to oxidizing and reducing atmospheres up to 2000 F (Ref. 92). It is resistant to solutions of practically all of the chloride compounds. Investment castings are used for parts such as nozzle guide vanes. Sand castings are used in applications such as tail pipe and exhaust cone fittings. Long exposure in the intermediate temperature range of 930 to 1450 F can result in accelerated corrosion (Ref. 92). This susceptible structural condition can be erased by subsequent solution treatment at 2225 F followed by water quenching.

Investment castings of Hastelloy C are covered by AMS 5388B (Ref. 90). The use of refined master heats and production of test specimens are the same as described for the production of Hastelloy B investment castings. Normally the precision castings are not heat treated. Specified minimum tensile properties at 1500 F are 50,000 psi ultimate strength and 10 per cent elongation. In the as-cast condition, the hardness must not exceed 21 Rockwell C. After being heated at 1475 F for 50 hours, the room-temperature hardness must not exceed 42 Rockwell C. In the bend test at room temperature, cracking must not occur in at least two specimens bent through an angle of 20 degrees around a 0.5-inch-diameter mandrel. If more than one of the three cast bars fail in this test, similar bars (0.090 inch in diameter or thickness and 2 inches in length) may be machined from castings, gates, or runners and tested. In lieu of these machined specimens, disposition of castings may be determined by showing that the carbon content of the castings conforms to the range in the specification.

Sand castings of Hastelloy C are covered by AMS 5389A (Ref. 91). Although the composition specified is the same as for investment castings, no restrictions are placed on the preparation of the heat for casting in sand molds. However, sand castings are usually solution heat treated at 2225 F for at least 30 minutes and cooled in air to improve their ductility and resistance to corrosion. In addition to the usual requirements of freedom from deleterious internal or external defects, the hardness of sand castings must not exceed 221 Brinell. The room-temperature Izod impact strength for Hastelloy C is about 20 ft-lb for sand castings and 5 ft-lb for investment castings (Ref. 192). Joining practice is the same as for Hastelloy B.

Hastelloy D resists nonoxidizing acids and salts, concentrated sulfuric acid, and hydrochloric acid under mild conditions (Ref. 92). It has excellent resistance to wear and galling against soft or hard alloys. It is not recommended for service under oxidizing conditions at elevated temperature. The alloy has very low ductility and impact strength. Joining is done with an axyacetylene torch with a slightly reducing flame.

Hastelloy F is resistant to acid or alkaline solutions and to oxidizing or reducing conditions. It has good resistance to stress-corrosion cracking in chloride solutions. Joining practice is the same as for Hastelloy B.

Hastelloy N was developed as a container material for molten fluoride salts (Ref. 92). It resists oxidation in air at up to 1800 F in continuous service and up to 1900 F in intermittent service. The room-temperature Izod impact strength is about 28 ft-lb for investment castings. Data are lacking on the strength of castings at elevated temperatures. However, data on the wrought alloy would indicate that castings retain useful strengths to a fairly high temperature. This statement also applies to Hastelloy F. The joining practice for Hastelloy N is the same as for Hastelloy B.

Hastelloy X has good strength above 1500 F and oxidation resistance up to 2200 F (Ref. 92). Because it is also resistant to carburizing atmospheres, it is used as fixtures and parts of industrial furnaces and other applications involving severe conditions at elevated temperatures. The alloy is rarely used for its resistance to attack by aqueous solutions at room temperature. Investment castings are used for parts such as turbine shroud segments and turbine vanes (Ref. 93).

Table VI presents typical tensile properties of both sand castings and investment castings of Hastelloy X. The sand castings are slightly stronger and less ductile. Only investment castings are covered by an Aeronautical Materials Specification, AMS 5390 (Ref. 93). This specification describes the use of master heats and preparation of test specimens in the same manner as the specifications for investment castings of Hastelloys B and C. Minimum requirements for tensile properties at 1500 F are 35,000 psi ultimate strength and 12 per cent elongation. In the as-cast condition hardness must not exceed 96 Rockwell B. After being heated at 1475 F for 50 hours, the room-temperature hardness of castings must not exceed 24 Rockwell C. At least two of the three bend-test specimens, which are cast to represent the heat, must not crack during bending at room temperature through an angle of 30 degrees around a 0.5-inch-diameter mandrel. If more than one specimen from a mold fails this test, disposition of the castings may be determined from additional tests on specimens machined from castings, gates, or runners. Joining practice for Hastelloy X is the same as for Hastelloy B (Ref. 92).

HU, HW, and HX. Of these three sand-cast heat-resistant alloys, the one most commonly in use is HW (Ref. 94). They all have very similar characteristics (Refs. 18, 95) and are justified when the service temperature is above 1000 F. They have good strength and ductility and resist oxidation and corrosion at elevated temperatures in various atmospheres except those containing a high sulfur content. Because they are austenitic, no brittle phases form at elevated temperatures. They are resistant to carburization and thermal fatigue, have good weldability, and are readily machined. Applications include those that require bearing a load at elevated temperatures or withstanding cyclic temperatures over a wide range.

Castability is good, and sections as thin as 3/16 inch can be run (Ref. 18). However, as a class, these heat-resistant alloys lose fluidity and solidify more rapidly than ordinary carbon steels (Ref. 106). As a result, castings are somewhat susceptible to shrinkage, hot tearing, and cold shuts. The patternmaker's shrinkage is 5/16 inch per foot for HU and 9/32 inch per foot for HW and HX (Ref. 18).

In addition to the properties reported in Table VI, the following tabulation presents the values for minimum creep stress at various temperatures (Ref. 95):

Test Temperature, F	Minimum Creep Stress for Strain of 0.0001 Per Cent Per Hour, 10^3 psi		
	HU	HW	HX
1400	8.5	6	6.4
1600	5	3	3.2
1800	2.2	1.4	1.6
2000	0.6	0.5	0.6

The following recommendations are given for machining with high-speed tools (Refs. 94, 95):

Rough cuts:	40 to 45 sfpm speed, 0.025 to 0.035-inch feed, 1/16 to 1/8 inch deep
Finish cuts:	80 to 90 sfpm speed, 0.010 to 0.015-inch feed, 1/16 to 1/8 inch deep
Drilling:	40 to 60 sfpm speed, 0.001 to 0.002-inch feed for a 1/8-inch drill to 0.015 to 0.205-inch feed for a drill 1 inch or over in diameter
Tapping:	5 to 15 sfpm speed with the use of a chip curler and breaker.

With carbide tools, these speeds may be increased by a factor of 3. With either type of tool, the tool should be in constant penetration in the casting to avoid work hardening of the surface. Sulfurized oil is recommended as the lubricant for high-speed tools and soluble oil in water for carbide tools.

Welding is done by the metal-arc and oxyacetylene processes (Refs. 94, 95). For HU castings, an AISI type 330 or 18Cr-38Ni bare electrode is used in the metal-arc processes. In gas welding, the flame is strongly reducing and a type 330 rod is used bare and without flux. For HW and HX castings, a lime-coated electrode of Inconel 132 is used for arc welding. In gas welding, a reducing flame is used with a "stainless" flux with a bare Inconel filler rod. Neither preheating nor postheating is required (Refs. 18, 92, 95). The HU alloy combines high creep strength with ductility up to 2000 F. It is the best of the three alloys in resistance to corrosion at elevated temperatures in oxidizing or reducing atmospheres containing moderate amounts of sulfur (up to 100 grains per cubic foot). Applications include salt pots, quench trays, furnace fixtures, and gas-dissociation equipment.

The HW alloy is useable up to 2050 F in a strongly oxidizing atmosphere or up to 1900 F in oxidizing or reducing gases that have a low sulfur content. The scale that forms is very adherent. Uses for this alloy are electric-resistance heating elements, cyanide pots, gas retorts, quenching fixtures, hearth plates, and furnace muffles.

The HX alloy resists corrosion up to 2100 F even when some sulfur is present in the atmosphere (Ref. 95). It has been reported, however, that HX castings decarburize rapidly in the range of 2000 to 2100 F (Ref. 18). Because it has the highest nickel content of the three alloys, it has the best resistance to carburization and best resistance to thermal fatigue. The latter property makes the alloy the most suitable for service involving side temperature cycling. Uses include quenching fixtures, carburizing and cyaniding equipment, heat-treating boxes, retorts, and furnace-burner parts.

Illium B, G, and 98. The Illium alloys were developed to provide sand castings that are resistant to sulfuric as well as nitric acid over a wide range of concentrations and exposure conditions (Ref. 96). They have limited resistance in hydrochloric acid and acid chlorides. Illium G is resistant to sulfuric acid in all concentrations at up to 150 F, and Illium 98 is resistant at up to 175 F. All of the alloys resist nitric acid at all concentrations at all temperatures.

As shown in Table V, Illium B has four modifications in composition of Illium 98 by the addition of silicon. Boron is also added to give resistance to erosion, wear, and galling in hot sulfuric acid solutions. In general, the resistance to various media is similar to Illium G except that Illium B is less resistant to strongly oxidizing media. For maximum hardness and wear resistance, the Illium B alloys are given the following aging treatments:

Alloy Grade	Temperature, F	Time, hr	Hardness, BHN
B1	1400	24	387
B2	1300	24	430
B3	1250	24	460
B4	1100	24	555

Joining is accomplished with an oxyacetylene torch with a 1400 F preheat followed by slow cooling after welding. Applications for Illium B are bearings for corrosive media, cutting blades, and pump impellers that involve corrosion and erosion.

Illium G is the cast counterpart of Illium R, which is a wrought alloy. It is resistant to sulfuric, nitric, and phosphoric acids and many acid-salt mixtures. It withstands both oxidizing and reducing agents, and acid and alkaline solutions. Resistance is good to most sulfur compounds and gases, but is poor to halogen and halogen salt solutions. Welding is done by the metal-arc or inert-gas-arc processes. Applications are for pumps and valves and process equipment where high strength is desired in severely corrosive environments.

Illium 98 is more resistant than Illium G to the same agents. It is used for pumps and valves for sulfuric acid in all concentrations. It is not resistant to halogen, halogen acids, or halogen salt solutions. Joining practice is the same as for Illium G.

All three of the Illium alloys have a patternmaker's shrinkage of 5/16 inch per foot.

BRITISH VACUUM-CAST ALLOYS

Table VII presents the compositions of the British casting alloys and gives an alternative designation of the alloys in parentheses. Table VIII summarizes the typical tensile and stress-rupture data for each alloy. Other characteristics are presented in the following discussions of the alloys.

EPK 24. This alloy is the British equivalent of the IN-100 developed in the United States. It is discussed in an earlier section of the report that deals with vacuum-cast alloys produced in the United States.

EPD 16. The EPD 16 alloy was developed recently. It is investment cast under vacuum for rotor and stator blades in aircraft gas-turbine engines (Ref. 107). It has excellent high-temperature strength as shown by the data in Table VIII. Impact toughness of unnotched specimens at room temperature is 15 to 18 ft-lb after heating for 16 hours at 1800 F (Ref. 107).

EPK 36. This alloy was developed at about the same time as EPD 16 and is also vacuum cast. As revealed by the data in Table VIII, the EPK 36 alloy has higher tensile strength at all temperatures, but is slightly inferior to the EPD 16 alloy in stress-rupture properties.

TABLE VII. COMPOSITION OF BRITISH VACUUM-CAST AND

Per Cent.

Alloy	C	Mn	Si	Cr	Ni	Co	Mo	Ti	Al
									Vacuum Cast
EPK-24(IN-100)(a)	0.15/2.0	0.50•(b)	0.50•	8/11	Bal	13/17	2.0/4.0	4.5/5.5	5.0/6.0
EPD 16	0.12	--	--	6.0	Bal	--	2.0	--	6.0
EPK 36	0.10	--	--	10.0	Bal	10.0	4.0	5.0	5.0
M22VC	0.08/0.16	--	--	5.0/6.5	Bal	--	1.5/2.4	--	5.9/6.6
G. 44	0.08	0.3	0.4	20.0	Bal	8.0	6.0	1.6	1.0
G. 55	0.15	0.2	0.3	15.0	Bal	--	4.0	2.5	2.5
G. 64(a)	0.08/0.16	0.80•	0.20/0.80	10/12	Bal	--	2.0/4.0	--	5.5/6.5
G. 67	0.12	0.5	0.5	16.0	Bal	--	3.0	1.0	6.0
G. 74(a)	0.12	--	--	16.0	Bal	10.0	--	4.0	4.0
G. 84B	0.14	--	--	10	Bal	15	2.5	5.2	5
G. 94(a)	0.06	--	--	9.0	Bal	10.0	4.0	--	6.0
G. 39	0.50/0.55	1.0	0.8	19.0/20.0	Bal	--	3.0	--	--
G. 69	0.3	0.5	0.5	20.0	Bal	10.0	3.0	0.3•	0.2
									Air Melted
Nimocast 75 (Nimonic C. 75)	0.05/0.15	0.20/1.0	0.20/1.0	18.0/22.0	Bal	--	--	0.2/0.6	0.4/1.0
Nimocast 80 (Nimonic CC)	0.03/0.10	0.20/1.0	0.20/1.0	18.0/22.0	Bal	--	--	2.20/3.00	0.8/1.16
Nimocast 90 (Nimonic CB)	0.05/0.10	0.2/0.5	0.2/0.6	18.5/20.5	Bal	15.5/18.0	--	2.2/2.6	1.0/1.4
Nimocast 242 (C. 242)	0.27/0.40	0.2/0.5	0.2/0.5	18.0/23.0	Bal	9.0/11.0	9.5/11.0	0.30	0.20
Nimocast 257 (MC. 57)	0.06/0.10	0.3	0.4	20	Bal	16	--	1.6	0.9
Nimocast 258 (MC. 58)	0.2	0.3	0.4	10	Bal	20	5	3.7	4.8
Nimocast PE10 (MC. 102)	0.02/0.06	0.1/0.5	0.1/0.4	19.0/20.5	Bal	5.0•	5.5/6.5	--	--
C. 130	0.10	1.00	1.00	20.0/23.0	Bal	1.0	9.0/10.5	2.2/2.8	0.5/0.9

(a) Most frequently mentioned in the technical literature published during the period 1963 to 1965.

(b) • - indicates maximum value.

AIR-MELTED NICKEL-BASE ALLOYS

Fe	B	Other	Probable Developer	Reference
1.0*	0.01/0.02	0.015*S, 0.03/0.09Zr, 0.70/0.90V	INCO (U.S.A.)	46
--	0.020	11.0W, 1.5Cb, 0.12Zr	Mond Nickel (Henry Wiggen)	107
--	0.015	0.12Zr	Ditto	107
1.0*	--	10.5/11.5W, 2.6/3.4Ta, 0.4/0.8Zr	Mond Nickel	110
1.0*	--	--	Jesson-Saville	49
1.0*	--	2.0W, B added	Ditto	49
5.0*	0.10/0.40	2.0/5.0W, 1.5/2.5Cb	"	46
5.0*	Added	4.0W	"	111
--	--	8.0W	"	111
--	Added	1.25W	"	78
--	Added	4.0W, 4.0Cb, Zr added	"	131,78
5.0*	--	3.0W, 1.5Cb, 1.5Ta	"	112
1.0*	--	3.0W, 3.0Cb	"	49
5.0*	--	--	Mond Nickel (Henry Wiggen)	113
5.0*	--	--	Ditto	113
2.0*	--	0.4*Cu	"	114
1.0*	--	--	Rolls Royce	113
2*	--	--	Mond Nickel (Henry Wiggen)	115
2*	--	--	Ditto	115
.0*	--	2.0/3.0W, 6.2/6.7Cb+Ta	"	116
.0	--	--	Rolls Royce	113

TABLE VIII. SHORT-TIME TENSILE AND STRESS-RUPTURE PROPERTIES OF BRITISH VACUUM-CAST, NICKEL-BASE ALLOYS

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2 % Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi	
								100-Hr Life	1000-Hr Life
EPD 16 (Refs. 49,107)	As cast[(a)]	170	112	--	5.6	10	--	--	--
		1200	116	--	2	4	--	--	--
		1400	115	--	2.5	5	--	91	74
		1500	109	--	3.5	6	--	73	56
		1600	103	--	4	6	--	56	37
		1800	75	--	6	7.5	--	26	17
EPK 36 (Refs. 49,107)	As cast[(a)]	70	125	--	7.9	24	--	--	--
		1200	145	--	8.5	12.5	--	--	--
		1400	133	--	7	5.5	--	82	69
		1500	120	--	6	4	--	71	56
		1600	106	--	7.5	6	--	52	39
		1800	78	--	6	6	--	24	15
M 22 VC (Ref. 108)	As cast[(a)]	1922	--	--	--	--	--	17.8	--
G. 44 (Ref. 49)	As cast[(a)]	1200	--	--	--	--	--	60.5	--
		1350	--	--	--	--	--	45.2	--
		1500	--	--	--	--	--	29.1	--
		1600	--	--	--	--	--	19.3	--
		1800	--	--	--	--	--	4.5	--
G. 55 (Ref. 49)	As cast[(a)]	1200	--	--	--	--	--	95	--
		1350	--	--	--	--	--	75	--
		1500	--	--	--	--	--	48.8	--
		1600	--	--	--	--	--	31.5	--
		1800	--	--	--	--	--	7.6	--
G. 64 (Refs. 46,49)	As cast[(a)]	70	111.5	110	3.3	3.5	36 R_C	--	--
		1500	--	--	--	--	--	58.6	42.5
		1600	--	--	--	--	--	42.5	28.7
		1800	--	--	--	--	--	19	13.9
G. 67 (Ref. 49)	As cast[(a)]	1500	--	--	--	--	--	64.9	44.8
		1600	--	--	--	--	--	42.5	30.7
		1800	--	--	--	--	--	18.1	12.1
G. 74 (Refs. 31,117)	As cast[(a)]	1650	--	--	--	--	--	39.6	--
		1700	--	--	--	--	--	33	--
		1800	--	--	--	--	--	22	--
G. 84B (Ref. 78)	As cast[(a)]	1600	--	--	--	--	--	53	--
		1700	--	--	--	--	--	35	--
		1800	--	--	--	--	--	24	--
		1900	--	--	--	--	--	16	--
G. 94 (Ref. 78)	As cast[(a)]	70	140	132	6	14.5	--	--	--
		1200	127	118	5	12	--	--	--
		1400	120	113	4	9	--	86	75
		1500	114	103	3	10	--	67	55
		1600	99	86	6	13	--	49	38
		1700	83	66	7.5	14.5	--	34	24
		1800	65	52	7.5	14.5	--	23	15
		1900	--	--	--	--	--	14	8
G. 39 (Refs. 49,112)	As cast[(a)]	70	73.2	--	5	5	190 BHN	--	--
		1200	61	--	9	--	--	38	29.1
		1350	57	--	12.5	--	--	24	18.2
		1500	49	--	18	--	--	15.9	12.2
		1600	40	--	24	--	--	11.7	8.8
		1800	--	--	--	--	--	6.6	4.6

TABLE VIII. (Continued)

Alloy	Condition	Test Temperature, F	Ultimate Tensile Strength, 10^3 psi	Yield Strength (0.2% Offset), 10^3 psi	Elongation, per cent	Reduction in Area, per cent	Hardness	Rupture Stress, 10^3 psi	
								100-Hr Life	1000-Hr Life
G. 69 (Ref. 49)	As cast(a)	1200	--	--	--	--	--	55	--
		1350	--	--	--	--	--	35.2	--
		1500	--	--	--	--	--	22.4	--
		1600	--	--	--	--	--	17.5	--
		1800	--	--	--	--	--	9.2	--
Nimocast 75 (Ref. 115)	As cast	70(a)	76.6	28.8	43.8	--	139 DPH	--	--
		70(b)	73.3	28.8	34.8	--	164 DPH	--	--
Nimocast 80 (Refs. 49,115)	1975 F/4 hr, 1290 F/16 hr, air cooled each time	70(b)	109.6	76.6	13.7	--	270 DPH	--	--
		70(a)	104.5	74.4	15.2	--	275 DPH	--	--
		1200(a)	--	--	--	--	--	39.2	29.1
		1350(a)	--	--	--	--	--	24.2	17.9
Nimocast 90 (Ref. 114)	Ditto	70(b)	106	79.7	12.6	--	291 DPH	--	--
		70(a)	101.5	75.2	14.1	--	291 DPH	--	--
		1200(a)	85	69	14	--	--	59.1	57.1
		1350(a)	75.5	65	7	--	--	51.5	41.5
		1500(a)	65	55	10	--	--	31.4	22.4
		1600(a)	37	28	18	--	--	18	11.2
		1800(a)	10	6	47	--	--	--	--
Nimocast 242 (Refs. 49,115)	As cast(a)	70	68.4	42.2	7.9	--	220 DPH	--	--
		1350	--	--	--	--	--	25.3	16.4
		1500	--	--	--	--	--	16.8	10.7
		1600	--	--	--	--	--	12.8	7.6
		1800	--	--	--	--	--	6.3	--
Nimocast 257 (Ref. 115)	1975 F/8 hr, 1290 F/16 hr, air cooled each time(c)	70	99	63.8	15.8	--	--	--	--
		1200	72.6	55.4	8.7	--	--	--	--
Nimocast 258 (Refs. 49,115)	As cast(a)	70	120.1	112.2	3.4	--	375 DPH	--	--
		1350	--	--	--	--	--	72.8	--
		1500	--	--	--	--	--	52.6	--
		1600	--	--	--	--	--	40.3	--
		1800	--	--	--	--	--	17.9	--
Nimocast PE10 (Refs. 49,118)	1975 F/8 hr, 1560 F/16 hr, air cooled each time(a)	1200	--	--	--	--	--	80.0	69.4
		1350	--	--	--	--	--	49.2	35.8
		1500	--	--	--	--	--	28.0	19.0
		1600	--	--	--	--	--	19.0	12.3
		1800	--	--	--	--	--	8.9	5.6
C. 130 (Ref. 113)	As cast	70(a, d)	72.6	--	5	--	--	--	--

(a) Investment cast.
(b) Sand cast.
(c) Centrifugally cast.
(d) Specified minimum properties.

The impact toughness of unnotched specimens at room temperature is 34 to 48 ft-lb in the as-cast condition and 19 to 37 ft-lb when heated for 16 hours at 1800 F (Ref. 107). These are higher values than those for the EPD 16 alloy.

M22VC. Fairly recently, it was reported (Refs. 108-110) that the M22VC alloy was in an advanced stage of development. It was said to offer outstanding stress-rupture strength at temperatures exceeding 1800 F. Castability in terms of freedom from dispersed microporosity was said to be excellent. However, the only data given in the literature are the compositions presented in Table VII, and the one value of 17.8 10^3 psi for the 100-hour stress-rupture life at 1922 F.

G. 44, G. 55, G. 64, G. 67, G. 74, G. 84B, and G. 94. All of these alloys are melted and cast under vacuum. They were developed for turbine blades and parts requiring high strength and resistance to corrosion at elevated temperatures. As shown by the stress-rupture properties in Table VIII, the general trend is an increase in strength with increase in the numerical designation of the alloys in this series. No heat treatment is required.

Castings of the G. 64 alloy are apt to contain very little microporosity and are not susceptible to hot tearing. The latter characteristic is believed to be associated with high hot-tensile elongation (Ref. 78): 4.9 per cent at 1832 F, 10.3 per cent at 2012 F, 16.5 per cent at 2102 F, and 21.8 per cent at 2192 F.

Neither the room-temperature nor the high-temperature properties of the G. 64 alloy were said to be sensitive to the as-cast grain size (Ref. 27). However, supporting data were not presented on the fine-grain castings poured at 2435 F (near the freezing point); medium-grain castings poured at 2590 F; and very coarse-grain castings poured at 2770 F. It should not be inferred that this characteristic applies to the other alloys in this series.

The specification for G. 64 castings calls for a minimum life of 50 hours at a stress of 33 10^3 psi at 1650 F (Ref. 27). From the stress-rupture data in Table VIII, it appears that this requirement is readily met. The impact strength of unnotched bars, 3/8 inch in diameter, cast at a normal temperature was 9 to 12 ft-lb when tested at 1650 F (Ref. 27).

The G. 84B alloy is apt to contain more microporosity and is more susceptible to hot tearing than the G. 64 alloy. The G. 84B alloy is similar to the IN-100 alloy in these two characteristics. Hot tearing is associated with the following low values for hot-tensile elongation (Ref. 78):

	Per Cent			
	1832 F	2012 F	2102 F	2192 F
G. 84B	4.4	4.1	4.5	4.7
IN-100	4.1	1.5	1.4	2.0

Impact strength on 3/8-inch-diameter, unnotched bars of these two alloys was the same at 1650 F, 28 to 30 ft-lb (Ref. 78).

The G. 94 alloy was developed to have the good castability characteristics (low microporosity and resistance to hot tearing) of the G. 64 alloy and the high strength of the G. 84B alloy and IN-100 alloy. The solidity of wedge castings confirmed that the susceptibility to microporosity was low. Resistance to hot tearing was judged to be good as evidenced by high hot-tensile-elongation values: 5.5 per cent at 1832 F, 7.0 per cent at 2012 F, 10.1 per cent at 2102 F, and 24.2 per cent at 2192 F. The stress-rupture data in Table VIII show that the G. 84B and G. 94 alloys are comparable in strength. Impact strength of 3/8-inch, unnotched bars tested at 1650 F was 25 to 27 ft-lb, which is only slightly lower than the strength of the G. 84B and IN-100 alloys. In a thermal-shock test in which a wedged-shaped specimen was rapidly heated in induction to 1650 F, the G. 94 alloy withstood 300 thermal cycles whereas the IN-100 alloy withstood only 25 cycles.

BRITISH AIR-MELTED ALLOYS

In general, the shrinkage in the air-melted alloys is large in both the liquid and solid states. The solid contraction is 1/4 inch per foot. Therefore, risers must be quite large and allowance must be made for unconstricted contraction during cooling to avoid hot tears in the casting.

All of the conventional sand molds are suitable (Ref. 22). Investment molds that are poured while at a high preheat temperature should have an alumina or zircon precoat because refractories containing appreciable amounts of free silica tend to react with those alloys that contain titanium and aluminum and cause surface defects. Good pouring and gating practice must be used to minimize turbulence because of the strong tendency for these alloys to form dross.

G. 39 and G. 69. These two alloys are melted and cast in air. They may be sand cast, but are investment cast for use in applications requiring strength and especially corrosion resistance at elevated temperatures (Ref. 112). The G.69 alloy is the stronger of the two as shown by the data in Table VIII. Aside from these data, the literature gives further details only on the G.39 alloy.

The G.39 alloy is austenitic, which contributes to its good resistance to thermal shock. In addition, the high chromium content makes it resistant to oxidation up to 2100 F. The alloy is also resistant to stress corrosion and grain growth at high temperatures. Although resistant to oxidation and carburization, the alloy has poor resistance in a sulfurous atmosphere. Its resistance to corrosion is very good in strongly oxidizing acid solutions or alkaline solutions. Its machinability rating is 30 per cent that of cast carbon steel. A low speed and heavy feed is recommended.

Nimocast Alloys. Table VII presents the composition and designation of the Nimocast alloys. Alternative designations are shown in parentheses. Nimocast 75, 80, and 90 are the cast counterparts of the wrought Nimonic alloys. Applications for Nimocast 75 and 80 are limited (Ref. 115). Nimocast 75 has relatively low strength (see Table VIII) and Nimocast 80 has useful stress-rupture properties up to about 1350 F. Both of these alloys have poor castability. Nimocast 90 has better castability (Ref. 115), useful stress-rupture properties up to 1600 F (Ref. 115), and good resistance to thermal shock up to about 2000 F (Ref. 113). Ductility suffers if the carbon content exceeds 0.10 per cent (Refs. 119, 120). The properties given in Table VIII for the Nimocast 90 alloys are for air-melted material that was remelted and cast under argon. The properties for all of the other Nimocast alloys are for material melted and cast in air.

Nimocast 242 is not as strong as Nimocast 90 at room temperature or at elevated temperatures. It was developed for applications requiring good resistance to thermal shock at temperatures up to about 2000 F (Refs. 113, 115). A pouring temperature of 2570 F, which is about 100 F superheat as compared with a pouring temperature of 2790 F, does not affect high-temperature properties, but does increase the room-temperature properties (Ref. 115).

Nimocast 257 was developed by reducing the titanium and aluminum content of Nimocast 90 to obtain higher ductility at 1200 F (Ref. 115). The goals were minimums of 10 per cent elongation and a yield strength of at least 33,000 psi in a centrifugally cast ring when used at 1200 F in a turbine engine. The data in Table VIII show that the elongation was shy of the goal, but this was attributed to the carbon content being 0.12 per cent, which was outside the specified range of 0.06 to 0.10 per cent. Without the aging treatment after the solution anneal, the yield strength and elongation at 1200 F were 38,300 psi and 20 per cent, respectively. However, the aging treatment is recommended to promote stability at the service temperature.

Nimocast 258 was developed to provide useful stress-rupture properties at above 1740 F (Ref. 115). Therefore it was patterned after the high-strength wrought alloy, Nimonic 100. Nimocast 258 has the highest titanium and aluminum content of the Nimocast series of alloys and has the highest hardness and highest strength at room temperature and at elevated temperatures. If it is necessary to stress relieve the castings, they may be heated for 2 to 4 hours at 1830 F, air cooled, aged for 16 hours at 1560 F, and air cooled. This treatment may lower the room-temperature strength and the stress-rupture strength a small amount.

Nimocast PE10 has a much higher stress-rupture strength than Nimocast 90 at 1200 F, but closely approaches the strength of Nimocast 90 at 1600 F. Strength is developed in the Nimocast PE10 by alloying with tungsten and columbium in lieu of titanium and aluminum. Embrittlement from formation of network carbides can occur if the carbon content exceeds the specified range (Ref. 118). When the carbon content is on the high side of the specified range, deoxidation with magnesium rather than with calcium gave less segregation of carbides at the grain boundaries.

The C.130 alloy is stated to have useful strength at elevated temperatures and good resistance to thermal shock (Ref. 113). No details are available in the literature other than the composition given in Table VII and the specified minimum room-temperature tensile properties given in Table VIII.

POWDER METALLURGY

From a fundamental standpoint, the powder-metallurgy production of nickel or nickel-base-alloy hardware of one kind or another involves the same basic principles used in making iron, copper, or other metal parts from their respective powders. In general, powder metallurgy involves the following steps:

(1) Powder production and conditioning

(2) Blending with additives and lubricants

(3) Pressing or compaction

(4) Sintering

(5) Further processing – this might be coining for densification and/or sizing, infiltration with a liquid metal or impregnation with a plastic

(6) Testing and inspection.

Ordinarily the selection of the factors to be considered in a production schedule will depend on numerous technical and economic considerations. For example, the furnace atmosphere used in a sintering operation may reflect an optimum balance between the mechanical properties required in a finished part and the allowable cost of production. In some instances, conventional procedures may be modified or displaced by new producers where warranted by the nature of the product.

The following sections dealing with nickel and nickel-base alloys are set against the general background of metal-powder technology as described, for example, in References 121 through 125. This broad background, therefore, will not be repeated here. The section "Standard Definitions of Terms Used in Powder Metallurgy" includes some terms commonly used in powder-metallurgy production. Although there are considerable bodies of information dealing with nickel and nickel-base alloys and on powder metallurgy, separately, there is very little assembled information under the heading "powder metallurgy of nickel". The summary by Price (Ref. 126) is an example of the latter. This probably is explained by the relatively high cost of nickel in comparison with iron or copper and the fact that it is not used extensively for making pressed and sintered parts.

POWDER PRODUCTION

Because surface reactions are often important in production of metal powders and in subsequent fabrication stages, the low reactivity of nickel is of practical interest to the overall processing. As a rule, nickel compounds are more easily reduced to metal-powder form than are compounds of many of the other metals. The ease of reduction of the oxide of nickel in comparison with that of iron is indicated by the equilibrium constants for the reaction: $MO + H_2 = M + H_2O$, where M represents divalent iron or nickel. The relatively large H_2O/H_2 values for nickel in Table IX are indicative of the ready reduction of oxide powder to metal powder. Further, during any subsequent sintering operation in a reducing atmosphere, it is not difficult to

TABLE IX. EQUILIBRIUM CONSTANTS FOR THE REDUCTION OF IRON AND NICKEL OXIDES[(a)]

Temperature			
K	F	$FeO + H_2 = Fe + H_2O$	$NiO + H_2 = Ni + H_2O$
1000	1340	0.466	166
1200	1700	0.631	123
1400	2060	0.760	98
1600	2420	0.876	81

(a) Based on data from Elliott, J. F., and Gleiser, M., Thermochemistry for Steelmaking, Volume I, Addison-Wesley Publishing Company, Inc., Reading, Massachusetts, 1960.

maintain clean surfaces on the powder particles. The increasing H_2O/H_2 values toward lower temperatures also suggest a low rate of contamination by oxygen during ordinary storage and handling of powder. It should be noted, however, that contamination rates are also dependent on particle size, and that very fine powders are more easily damaged by humid atmospheres. Table X lists several well-known procedures for making nickel powder. Most can be modified in such a way as to produce nickel-base alloys. For example nickel-iron alloys can be made by the carbonyl process. Nickel-base high-temperature alloys are produced by atomizing a melt in an inert-gas stream. Codeposition by electrolysis is possible in certain instances. The hydride-reduction process has been used in making nickel-base chromium alloys.

TABLE X. SOME POSSIBLE METHODS FOR THE PRODUCTION OF NICKEL POWDER

(1) Gaseous reduction of a solid: $NiO + 2H_2 = Ni + 2H_2O$

(2) Carbonyl decomposition: $Ni(CO)_4 = Ni + 4(CO)$

(3) Electrolytic deposition

(4) Atomization of a melt

(5) Arc vaporization and condensation

(6) Hydrogen reduction of an aqueous solution: $Ni(NH_3)_2SO_4 + H_2 = Ni + (NH_4)_2SO_4$

(7) Mechanical milling

(8) NiO reduction with calcium hydride

Hydrogen reduction to powder from an aqueous solution has received a considerable amount of attention in recent years and is the basis for commercial production of powder. Figure 10 is a flow chart of one process by which nickel and cobalt powders are produced.

References 127 through 137 contain descriptions of powder-production principles that are applicable to nickel and nickel-base alloys. In most of the processes noted, there are variations with regard to particle-size range, size distribution within the range, particle shape, and powder purity. Therefore, catalogues of powder procedures should be consulted for specifications and prices. Figure 11 indicates the variety of fine powders made by the carbonyl process. Table XI lists some of the nickel-powder grades that have been made by reduction from aqueous solution. Table XII lists some composite powders that have been made by this general process, where nickel is deposited onto particles of another material such as aluminum, chromium, carbon, etc.

TABLE XI. PROPERTIES OF REPRESENTATIVE SAMPLES OF SHERRITT GORDON PRODUCTION POWDERS (REF. 131)

Powder Grade:	C	MC	M	MF	F
Chemical Analysis, per cent					
Nickel plus cobalt	99.8	99.8	99.8	99.8	99.8
Cobalt	0.072	0.070	0.092	0.080	0.092
Iron	0.010	0.015	0.026	0.049	0.057
Sulfur	0.010	0.012	0.016	0.018	0.014
Screen Analysis, per cent					
+ 100 mesh	1.4	Trace	Trace	Trace	Trace
-100 + 150 mesh	56.5	15.2	2.1	1.0	0.5
-150 + 200 mesh	40.3	73.0	36.0	7.2	6.4
-200 + 250 mesh	1.2	7.5	11.0	25.6	2.5
-250 + 325 mesh	0.6	4.0	49.2	45.7	51.9
-325 mesh	Trace	Trace	1.5	20.5	38.5
Hall-Flowmeter Values					
Apparent density, g/cc	4.16	4.07	3.95	3.84	3.59
Flow rate, sec/50 g	16.6	16.5	16.7	16.9	17.2

The Metal Powder Industries Federation may be contacted for sources of nickel powders.

There are numerous test procedures for the evaluation of metal powder. These are summarized in Reference 122. The Metal Powder

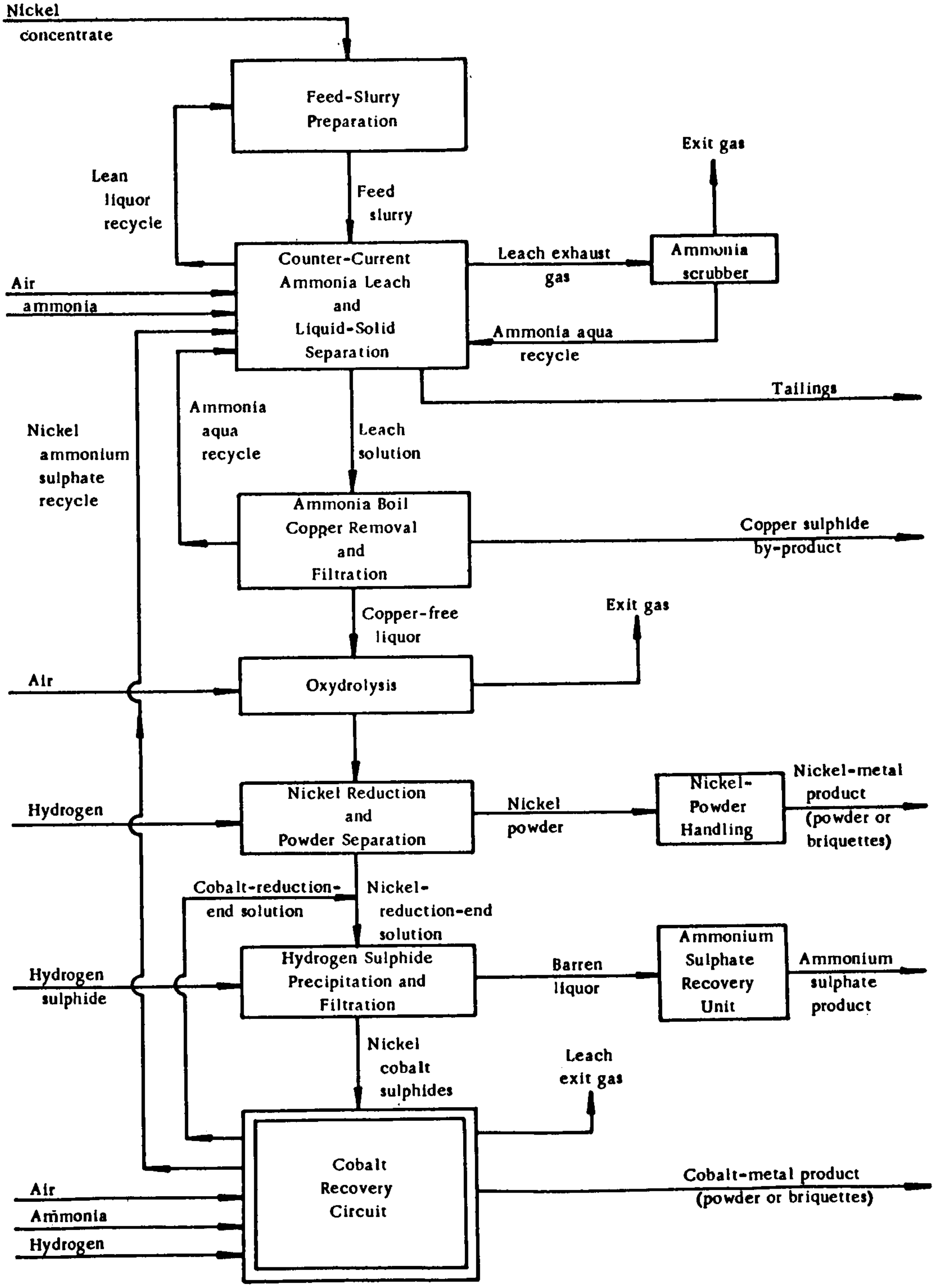
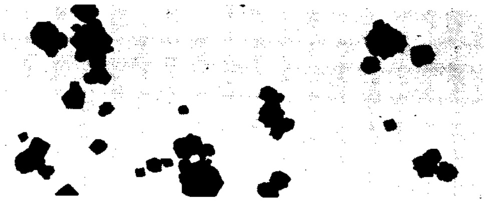


FIGURE 10. FLOW CHART OF SHERRITT GORDON MINES NICKEL REFINERY IN FORT SASKATCHEWAN (REF. 127)

a. Type 100 Carbonyl Nickel Powder

Average particle size – 3 to 5 microns
Apparent density – 1.6 to 2.0 g/cc
Typical chemical analysis – 0.020 per cent carbon, 0.010 per cent oxygen, 0.001 per cent sulfur or less, 0.01 per cent iron or less

b. Type 122 Carbonyl Nickel Powder
(formerly Type A)
Average particle size – 4 to 7 microns
Apparent density – 2.0 to 2.5 g/cc
Typical chemical analysis – 0.05 to 0.10 per cent carbon 0.10 per cent oxygen, 0.001 per cent sulfur or less, iron-less than 0.01 per cent



c. Type 128 Carbonyl Nickel Powder
(formerly Type D)
Average particle size – 7 to 9 microns
Apparent density – 2.5 to 3.0 g/cc
Typical chemical analysis – 0.05 to 0.10 per cent carbon, 0.10 per cent oxygen, sulfur-less than 0.001 per cent, iron-less than 0.01 per cent

d. Type 255 Carbonyl Nickel Powder

Average particle size – 2.6 to 3.4 microns
Apparent density – 0.5 to 0.6 g/cc
Typical chemical analysis – 0.05 to 0.15 per cent carbon, 0.05 to 0.15 per cent oxygen, sulfur-less than 0.001 per cent, iron-less than 0.01 per cent

e. Type 287 Carbonyl Nickel Powder
(formerly Type B)
Average particle size – 2.9 to 3.6 microns
Apparent density – 0.8 to 1.0 g/cc
Typical chemical analysis – 0.05 to 0.15 per cent carbc 0.05 to 0.15 per cent oxygen, sulfur-less than 0.001 per cent, iron-less than 0.01 per cent

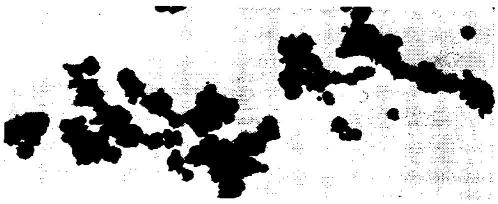


FIGURE 11. EXAMPLES OF CARBONYL NICKEL POWDERS

a, b, and c are general-purpose powders; d and e are special-purpose, large, specific-surface-area powders.

Courtesy of The International Nickel Corp.

TABLE XII. COMPOSITE POWDERS MADE BY HYDROGEN REDUCTION OF A NICKEL (OR COBALT) SOLUTION ONTO A SUSPENDED "CORE" MATERIAL

Sherritt Gordon Mines Ltd.

Typical Chemical Analyses

Alloy	Nickel	Cobalt	Copper	Iron	Sulfur	Carbon	"Core"
90 Ni/Al 10	89-91	0.7	0.01	0.01	0.05	0.05	9-11
67 Ni/Al 33	65-68	0.6	0.05	0.05	0.05	0.05	31-35
80 Ni/Cr 20	78-82	0.5	0.03	0.1	0.03	0.07	19-21
85 Ni/C 15	84-86	0.5	0.005	0.03	0.05	14-16	14-16
75 Ni/C 25	74-76	0.5	0.005	0.03	0.05	24-26	24-26
80 Ni/P 20	78-82	0.7	0.005	0.003	0.03	0.03	18-21
50 Ni/W 50	49-51	0.3	0.01	0.05	0.01	0.08	49-51
20 Ni/WC 80	18-21	0.3	0.01	0.05	0.02	--	79-81
20 Co/WC 80	0.5	18-21	0.01	0.05	0.02	--	79-81

Typical Screen Analyses

Alloy	+100	-100 +200	-200 +325	-325
90 Ni/Al 10	--	5 - 10	55 - 65	25 - 40
67 Ni/Al 33	--	1 - 10	40 - 60	40 - 60
80 Ni/Cr 20	1 - 5	60 - 80	10 - 30	--
85 Ni/C 15	--	0 - 10	50 - 80	20 - 40
75 Ni/C 25	--	0 - 10	40 - 70	30 - 60
80 Ni/P 20	--	5 - 10	40 - 70	25 - 50
50 Ni/W 50	--	0 - 5	20 - 35	60 - 80
20 Ni/WC 80	--	0 - 5	0 - 5	90 - 99
20 Co/WC 80	--	0 - 5	0 - 5	90 - 99

Typical Physical Data

Alloy	Apparent Density, g/cc	Hall Flow Rate(a), sec/50 g	Fisher Subsieve Size, microns(b)
90 Ni/Al 10	3.4	20	--
67 Ni/Al 33	1.4 - 1.6	47	--
80 Ni/Cr 20	2.5 - 2.8	32	--
85 Ni/C 15	1.4 - 1.5	55	--
75 Ni/C 25	1.1 - 1.2	75	8
80 Ni/P 20	1.5 - 1.7	38 - 46	--
50 Ni/W 50	2.6 - 3.6	25	9
20 Ni/WC 80	3.0 - 3.5	--	5
20 Co/WC 80	3.0 - 3.5	--	5

(a) ASTM B 213-48.

(b) ASTM B 330-58T.

Industries Federation has published a series of standards and specifications. The ASTM Standards on Metal Powders and Metal Powder Products also covers a number of evaluation tests that are applicable to nickel powders. These standards and specifications are ordinarily kept up to date and should be consulted for the most recent information on this topic.

METHODS OF COMPACTION

The first direct step in the consolidation process involves some method of metal-particle aggregation so that subsequent sintering can be accomplished in a reasonable time. The choice of a compaction procedure for any production operation depends on such factors as the shape and size and the physical and mechanical requirements of the finished part. Figure 12 illustrates six procedures that can be used in powder consolidation depending upon the quality required in the end product. In these procedures initial pressing probably would be done in a conventional cold die.

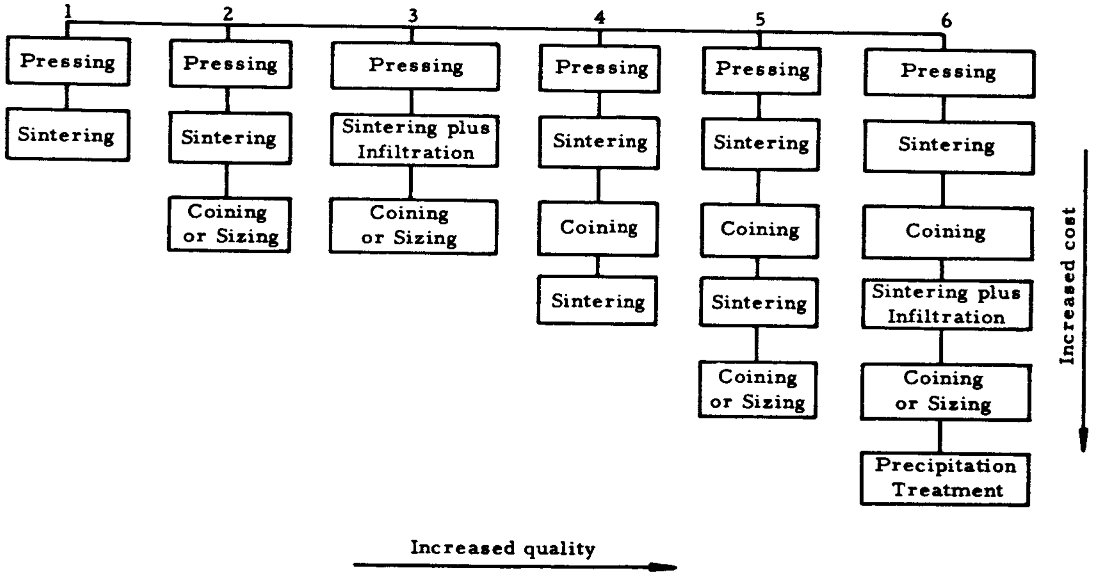


FIGURE 12. VARIOUS COMMON PROCESSES FOR THE MANUFACTURE OF SINTERED STRUCTURAL PARTS (REF. 138)

There are a number of other procedures for the initial consolidation step that may be useful, depending upon the kind of part or material being produced. These methods are listed in Table XIII.

TABLE XIII. SOME INITIAL CONSOLIDATION PROCESSES THAT MAY BE APPLICABLE TO NICKEL OR NICKEL-BASE ALLOYS

(1) Conventional cold pressing in a steel die
(2) Hot die pressing
(3) Isostatic or modified isostatic pressing by fluid-transmitted pressures (cold or hot)
(4) Slip casting
(5) Extrusion (cold or hot)
(6) Cyclic pressing
(7) Powder rolling
(8) Vibratory compaction
(9) Loose-powder die filling (gravity only)
(10) High-energy compaction (including explosive compaction)
(11) Infiltration

Where nickel-base alloys are to be consolidated, it may be necessary to consider such factors as the less favorable compactibility of the alloy powder under pressure, or to the increased possibility of contamination.

Cold Die Pressing. Figure 13 illustrates die fill and die action for three as-cold-pressed shapes. Figures 13 through 14g (Ref. 138) illustrate some of the die-design factors to be considered if satisfactory pressings are to be made. Reference 139 reviews similar design considerations in detail. A discussion by Silbereisen (Ref. 140) is especially clear in regard to powder behavior during die pressing of complex shapes.

The problem of uniform densification of powder during compaction and the achievement of uniform mechanical properties through a compacted section is illustrated by Figure 15. Even in the favorable H/D ratio illustrated and with die-wall lubrication, appreciable frictional hangup at the die-wall surfaces occurred.

In order to achieve the maximum possible uniform density in a powder compact, to reduce necessary compacting pressures, to assist

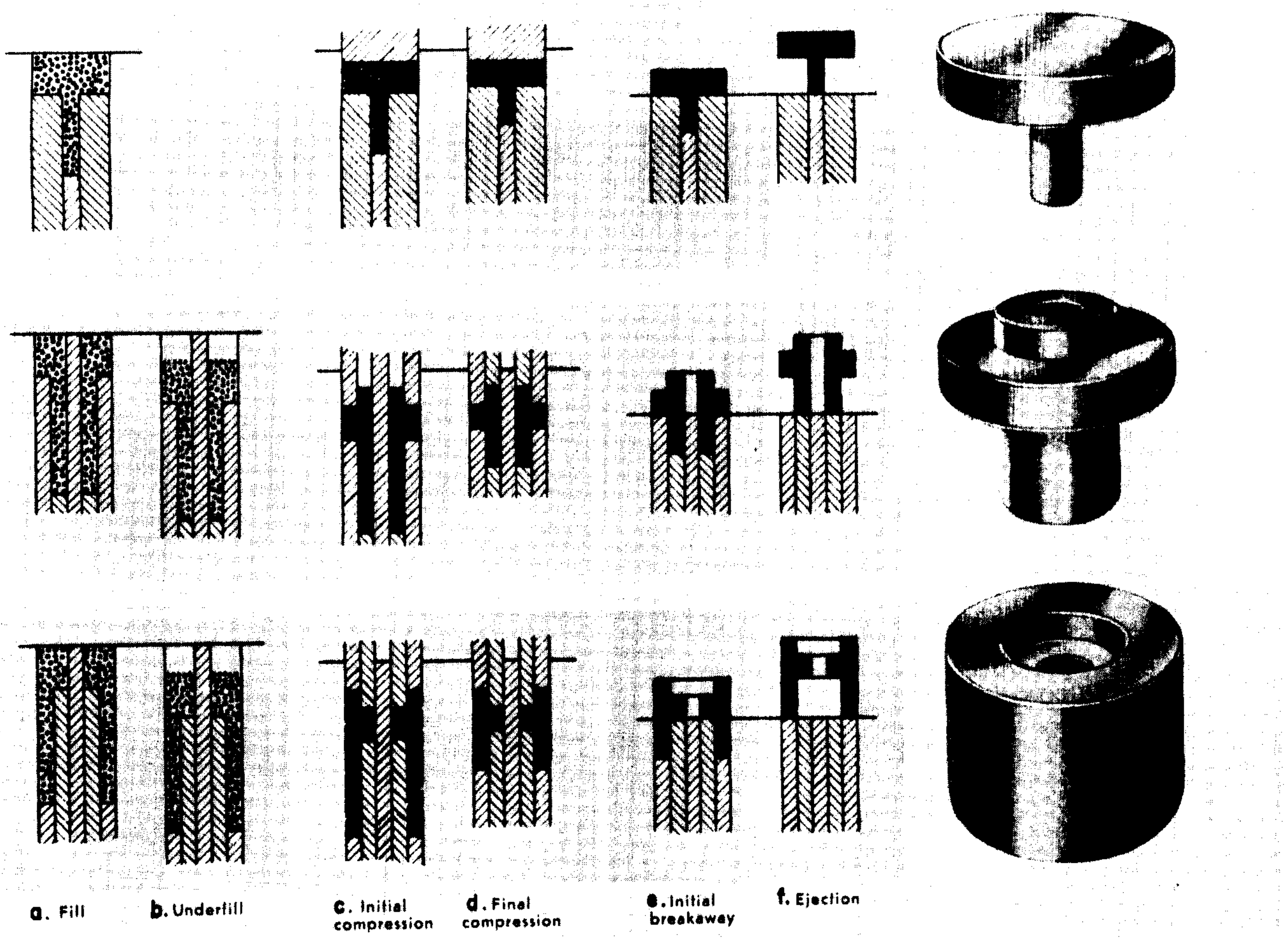
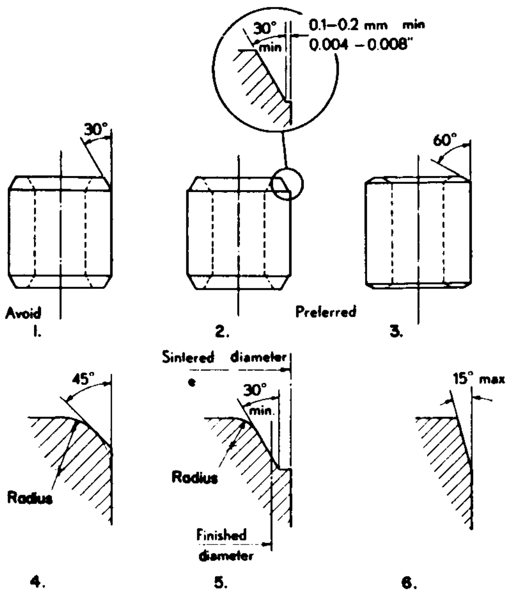


FIGURE 13. EXAMPLES OF DIE FILL AND DIE MOVEMENT IN THE CONVENTIONAL COMPACTION OF METAL POWDER IN STEEL DIES (REF. 138)

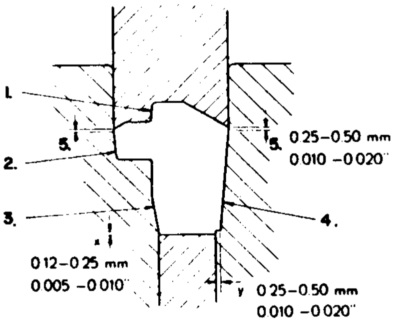
Chamfers with angles below 45 degrees should be avoided (1); chamfers with angles of 45 degrees or less require a flat land to avoid punch breakages (2); chamfers with angles greater than 45 degrees preferred (3); where a radius is considered essential, a useful compromise is sometimes found in a combination of radius and chamfer (4); in cases where the part is to be machined or ground on the outside diameter, e.g., for assembly into a housing, the form shown here is practical (5); and an acute angle for a lead-in can in some cases be formed in the compacting die, or produced by a coining operation if the chamfer is small (6) (Figure 14b).



a.

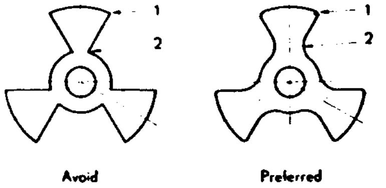
This is a section of a tool for a part having several tapers.

1. Shoulders in a solid top punch require 1 to 5 degrees taper to assist extraction.
2. 1 to 5 degrees taper on an unsupported flange assists extraction.
3. Angular forms such as this require a small parallel portion, x, to allow for variations in setting, etc. The maximum angle of taper, c, is 15 degrees if bottom compression is required.
4. To prevent trapping of powder in the acute angle between punch and die or core rod, a small horizontal step, y, is recommended.
5. A small parallel portion is required here to prevent jamming of the top punch.



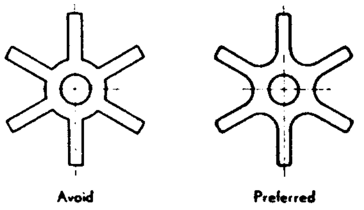
b.

FIGURE 14. DESIGN RECOMMENDATIONS FOR STRUCTURAL METAL-POWDER PARTS (REF. 138)



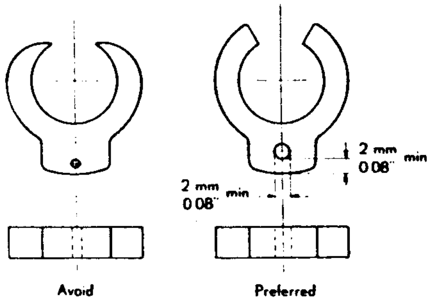
Avoid sharp edges which weaken die (1) and corners which weaken parts (2); radiused edges (1) and radiused corners (2) add strength to tools and parts.

c.



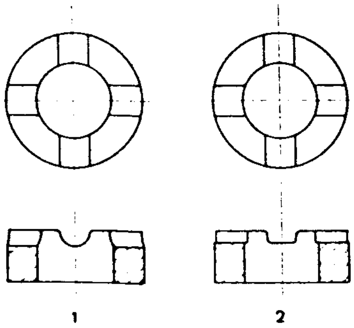
Design faults similar to the "Avoid" in Figure 14c, but even worse. Narrow splines cause density variation, particularly on long parts; radiused roots and rounded edges strengthen tools and parts.

d.



Profiles requiring feather edges on punches should be avoided. Holes should not be less in diameter than 20 to 25 per cent of their length. 2 mm (0.08 in.) is considered the practical minimum diameter for holes; "Preferred" redesigned for stronger punches and hole large enough for molding.

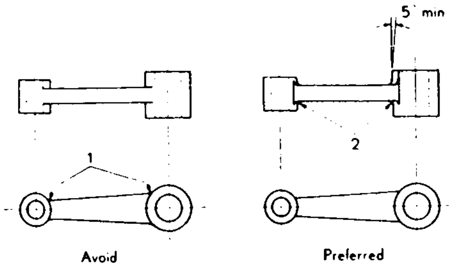
e.



Grooves in top face of parts can be molded within the following limits for low and medium density parts: Curved or semicircular grooves to a maximum depth of 30 per cent of total height of part (1); square grooves to a maximum depth of 20 per cent of total height; 5 degrees minimum taper is necessary on vertical sides. All corners and edges should be radiused. The form shown in (2) can be used in place of drilled holes in the face, e.g., for tightening screwed rings.

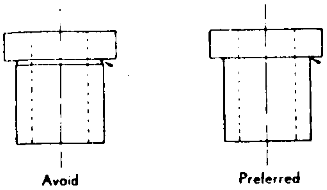
f.

FIGURE 14. (CONTINUED)



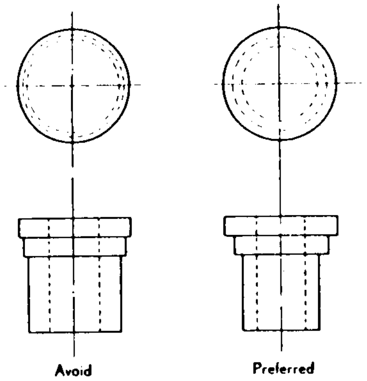
Radii (1) at change in profile require weak, feather-edged punches, liable to break down; radii (2) at change in height strengthen both punches and parts. Taper on portion of boss formed by top punch assists extraction. Remainder of boss, formed by die, must be parallel.

g.



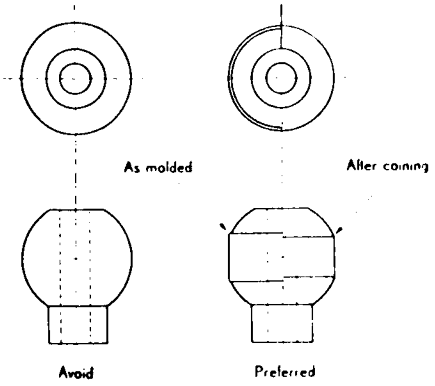
Undercuts cannot be molded. A radius should be provided between flange and body and the housing should have a corresponding radius or chamfer, otherwise the undercut must be subsequently machined.

h.



Parts with multiple steps should be designed with 0.9 mm (0.035 in.) minimum width for each step. Thin punches increase breakdown time.

i.



Complete spheres cannot be molded as sharp edges on punches would touch and break; a cylindrical section between two spherical sections can be molded. The cylindrical section will lie within the sphere after coining. If the cylindrical portion is less in height than 25 per cent of the spherical diameter, the punch edges become increasingly weak.

j.

FIGURE 14. (CONTINUED)

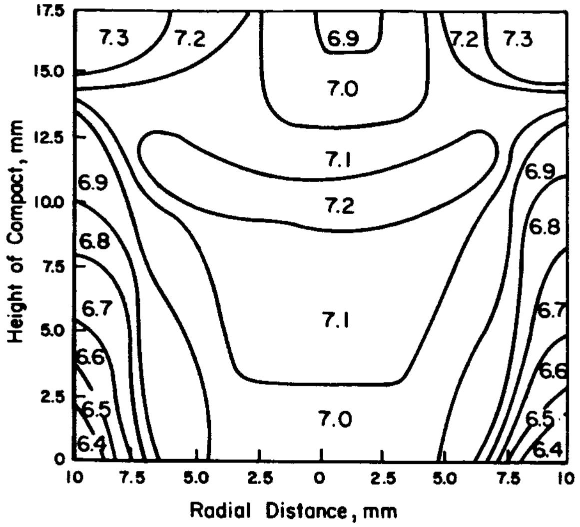


FIGURE 15. DISTRIBUTION OF DENSITIES IN A GREEN NICKEL COMPACT PRESSED AT 103,040 PSI

Die diameter, D, is 0.79 in.; H/D, 0.87 (Kuczynski and Zaplatynskyj) (Ref. 141).

in the successful extraction of the compact from the die, and to reduce die wear, adequate lubrication is necessary. Table XIV illustrates comparative die lives for a variety of die materials with and without additions of 1/2 per cent benzoic acid to the nickel powder. An excellent discussion of lubrication principles is found in Jones (Ref. 123). Die and press designs are also discussed by Jones and in a review paper by Oakley (Ref. 142). Manufacturers' brochures may be helpful in regard to presses and appertainment tooling.

TABLE XIV. LIFE OF VARIOUS DIES FOR COMPACTING ELECTROLYTIC NICKEL POWDERS -100 MESH (REF. 123)

Die Material	Number of Pressed Parts Without Lubricant	 With Lubricant[(a)]
SAE 1010 carbon steel	1,000	1,800
High-chromium carbon steel	5,000	8,000
Hard-chromium plated steel	50,000	65,000
Cemented carbide	700,000	950,000

(a) One-half per cent benzoic acid.

The hot pressing of nickel or nickel-base alloys in heated steel dies or in graphite dies represents a potentially useful consolidation procedure where 100 per cent densities must be achieved.

Contamination by carbon is less likely in the case of nickel than for stronger carbide-forming metal powders. Except in special instances, however, the added cost over that for cold-pressing operation has not been justified for nickel.

Isostatic Pressing (Refs. 143-145). Both cold hydrostatic pressing and hot gas-pressure pressing are applicable to nickel and nickel-alloy powders. These methods are in an early stage of development and thus far have not been used extensively. It may be expected that both procedures will find increased use in consolidating nickel and particularly nickel-alloy powders into preforms or billets for further processing by forging, extrusion, or rolling.

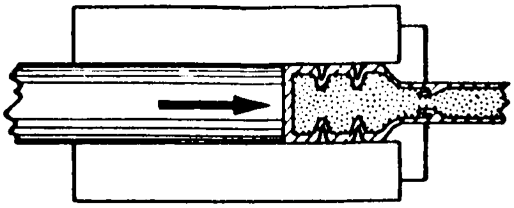
In cold hydrostatic pressing the powder is placed in a rubber or other elastomer mold or bag. The powder and bag are then placed within a pressure chamber and hydrostatic pressure in the approximate range of 20,000 to 10,000 psi is applied to the outside of the container. Uniform densification results because of the isostatic nature of the forces transmitted by the fluid.

In hot gas-pressure pressing the powder is placed within a thin-walled metal container, air is removed by evacuation, and the container is then sealed. The container and powder are placed in an autoclave and heated to an appropriate temperature for sintering. In the case of nickel alloys the temperature may be within the 2000 to 2400 F range. Gas pressure, usually an inert gas such as argon, is applied to the outside of the metal container. Autoclaves and auxiliary equipment are now available for pressures up to 100,000 psi. Following hot gas-pressure pressing it is usually necessary to remove the metal container either by machining or by acid pickling.

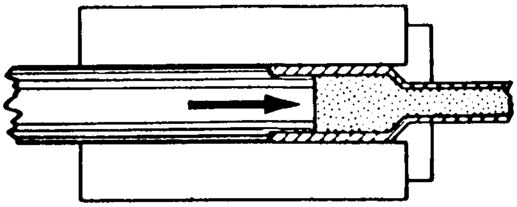
Slip Casting. Slip casting, vibratory casting, and loose-powder shaping conceivably are applicable to nickel powder but have not been used to any appreciable extent. In comparatively early work Hausner (Ref. 146) reported some results with nickel. The sintered material had low strength, possibly because of low density or contamination from the alginate deflocculant used. In more recent work, nickel-base alloys have been successfully slip cast, e.g., Udimet 700 and IN-100 (Ref. 147). The -325 mesh spherical-particle IN-100 responded favorably to slip casting in a turbine-blade shape. After vacuum sintering the slip casting for 8 hours in the temperature range 2315 to 2325 F, the tensile properties of the as-sintered material were comparable with those obtained by a casting process. The composition of the suspending fluid was as follows: 1.5 per cent Acrysol A3 polymer as a binder deflocculant, 1.5 per cent Marex (ammonium alginate), 2.0 per cent Aerosol OT solution as a wetting agent, and 95 per cent distilled water. Ammonium hydroxide was added to adjust the pH. A powder-to-fluid ratio of 10:1 was used. Continuous vibration was applied during feeding into the plaster mold in order to assure adequate filling along the thin trailing-edge section. The resulting slip casting was about 65 per cent dense. About 18 per cent shrinkage occurred during sintering. These results are tentative in nature due to the continuing status of the work. Slip casting, vibratory compacting, and pressureless shaping have been discussed in considerable detail by Hausner (Ref. 148).

Extrusion. Extrusion of nickel and nickel-base alloys can be carried out in a heated container so that practically full consolidation is effected. Figure 16 (Ref. 149) illustrates the use of an evacuated container for this purpose. TD nickel has been consolidated by this procedure (Ref. 150). Nickel, Monel, and Inconel powders have been extruded along with stainless steel powders in British work (Refs. 151, 152). Table XV lists the composition of the powders extruded. All the powders were used in the "as atomized" condition, apart from the removal of the +60 mesh fraction. Particle-size distribution varied somewhat but lie in the following general range: -60 + 100, 0 to 6 per cent; -100 + 200, 5 to 35 per cent; -200 + 300, 14 to 26 per cent, -300, 44 to 82 per cent. Powders were isostatically pressed at 50 tsi and canned prior to hot extrusion. The extrusion conditions are shown in Table XVI. Nearly full densities were achieved for the nickel-base alloys (Table XVII). Mechanical properties achieved are compared with those of conventionally wrought material (parentheses) in Table XVIII. As will be noted, most of the alloys had reasonably good ultimate tensile strengths. Some were a little deficient in ductility. A few creep tests on the Inconel-type alloys yielded disappointing results. However, in general, mechanical-property tests indicated extrusion of canned nickel-base alloys to be a promising method for conversion of metal powders into mill shapes.

In recent work conducted under U. S. Air Force sponsorship, the nickel-base alloys, Udimet 700 and Inconel 713 C, have been produced as powder having less than 0.05 per cent oxygen and 0.002 per cent nitrogen, by inert-gas atomization of the melt (Ref. 153). Considerable care was necessary in controlling sintering temperatures and atmospheres. The optimum sintering temperatures observed at 1/2 hour in vacuum was 2280 F for Udimet 700 and 2370 F for Inconel 713. Conventional pressing and sintering was not successful for large billets. Hot pressing steel-canned powders into billets proved satisfactory at a temperature of 2200 F and 40 tsi. Sound T-sections were



a. Folding of Metal Can With Loosely Packed Powder



b. Penetrator Technique to Avoid Folding of Can

FIGURE 16. EXTRUSION OF CANNED POWDER (REF. 149)

TABLE XV. CHEMICAL COMPOSITIONS OF ATOMIZED POWDERS EXTRUDED ACCORDING TO TABLE XVI (REF. 152)

Weight Per Cent.

No.	Powder	C	Si	Mn	Cr	Ti	Al	Mo	Nb	Fe	Cu	Ni
1	Nickel	0.02	0.12	--		--	--	--	--	0.19	--	Bal
2	Monel type	0.10	0.90	0.77	0.056	--	--	--	--	2.0	31.5	Bal
3	K-Monel type	0.15	0.69	1.44	0.095	--	2.93	--	--	3.18	29.4	Bal
4	Inconel X type	0.004	0.49	--	14.8	2.5	0.81	--	0.85	6.25	--	Bal
5	Incoloy type	0.042	0.61	0.56	19.3	--	--	--	--	Bal	--	30.8
6	347 L	0.021	0.56	--	17.9	--	--	--	0.2	Bal	--	13.0
7	316 L	0.011	0.68	--	18.2	--	--	2.06	--	Bal	--	13.1
8	316 L	0.033	0.61	--	16.3	--	--	1.89	--	Bal	--	13.8
9	410 L	0.029	0.95	--	13.3	--	--	--	--	Bal	--	--

TABLE XVI. EXTRUSION CONDITIONS FOR POWDER-METAL BILLETS (REF. 152)

No.	Powder	Extrusion No.	Extrusion Temperature, C	Extrusion Ratio
1	Nickel	1	1150	9
2	Monel type	2	1175	6.2
		3(a)	1150	9
		4(a, b)	1000	9
3	K Monel type	5	1175	6.2
4	Inconel X type	6	1275	6.2
5	Incoloy type	7	1275	6.2
6	347 L	8	1150	13.3
7	316 L	9	1150	13.3
8	316 L	10(a)	1200	9
		11(a, c)	1200	9
9	410 L	12(a)	1200	9
		13(a)	1200	9

(a) Can evacuated and sealed under low-pressure argon.

(b) Prepared from elemental atomized-nickel (No. 1) and electrolytic-copper powder.

(c) Contained a 0.1 wt % addition of CaH_2.

TABLE XVII. OBSERVED DENSITIES OF EXTRUDED BARS (REF. 152)

Powder	Extrusion No.	Density, g/cm^3	Percentage of Theoretical Density
Nickel	1	8.82	99.3
Monel type	2	8.80	99.6
	3	8.73	98.8
	4(a)	8.80	99.6
	4(b)	8.60	97.3
K Monel type	5	8.36	98.7
Inconel X type	6	8.22	99.6
Incoloy type	7	7.94	99.1
347 L	8	8.00	100
316 L	9	8.05	100
	10	7.95	98.8
	11	7.75	96.3
410 L	12	7.60	98.1
	13	7.50	96.8

(a) As extruded

(b) Homogenized at 1000 C for 16 hr.

TABLE XVIII. MECHANICAL PROPERTIES OF EXTRUDED BARS (REF. 152)

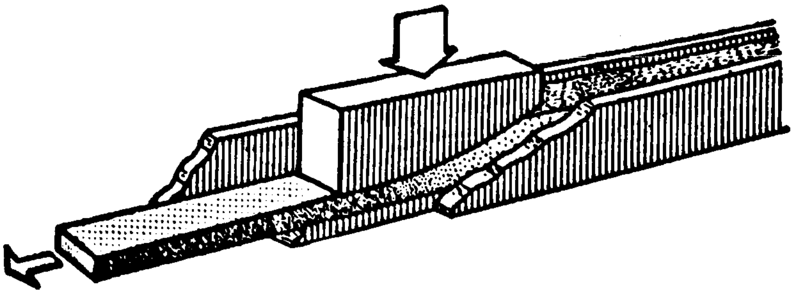
Powder	Extrusion No.	0.2 Per Cent Proof Stress(a), tsi	Maximum Strength, tsi	Elongation $L = 4\sqrt{A}$, per cent	Reduction in Area, per cent	Hardness, VPN
Nickel	1	10.0 (9.0)	24.6 (29.0)	39 (42)	52 (67)	122 (110)
Monel type	2	15.0 (15.9)	31.0 (33.6)	41 (40)	61 (65)	146 (146)
	3	15.4	32.0	47	60	155
	4(b)	13.2	27.2	39	55	125
K Monel type	5(c)	23.3 (20.0)	45.2 (44.4)	35 (40)	48 (55)	200 (169)
	5(d)	45.2 (40.0)	70.4 (66.0)	21 (25)	30 (35)	275 (275)
Inconel X-type	6(c)	34.4 (33.6)	62.8 (55.8)	31 (50)	40	245
	6(d)	54.5 (45.0)	78.0 (72.0)	20 (20)	28 (25)	358 (385)
Incoloy type	7	17.0 (17.8)	39.0 (40.0)	34 (40)	56 (70)	176
347 L	8	19.0 (15.0)	41.2 (39.0)	44 (50)	55 (65)	165 (155)
	8(e)	23.1	38.0	44	43	--
316 L	9	16.4 (14.0)	40.5 (35.0)	47 (55)	54 (65)	163 (145)
	9(e)	24.6	39.8	35	35	--
	10	19.2	38.8	45	55	162
	11	19.2	39.0	49	44	176
410 L	12(f)	41.6	50.0	17	28	236
	12(g)	40.8	52.4	20	35	260
	13(f)	41.6	48.0	17	26	265

(a) Values in parentheses are representative of conventional wrought material.
(b) Homogenized.
(c) Solution treated.
(d) Solution treated and aged.
(e) Transverse properties.
(f) As extruded.
(g) Hardened and tempered.

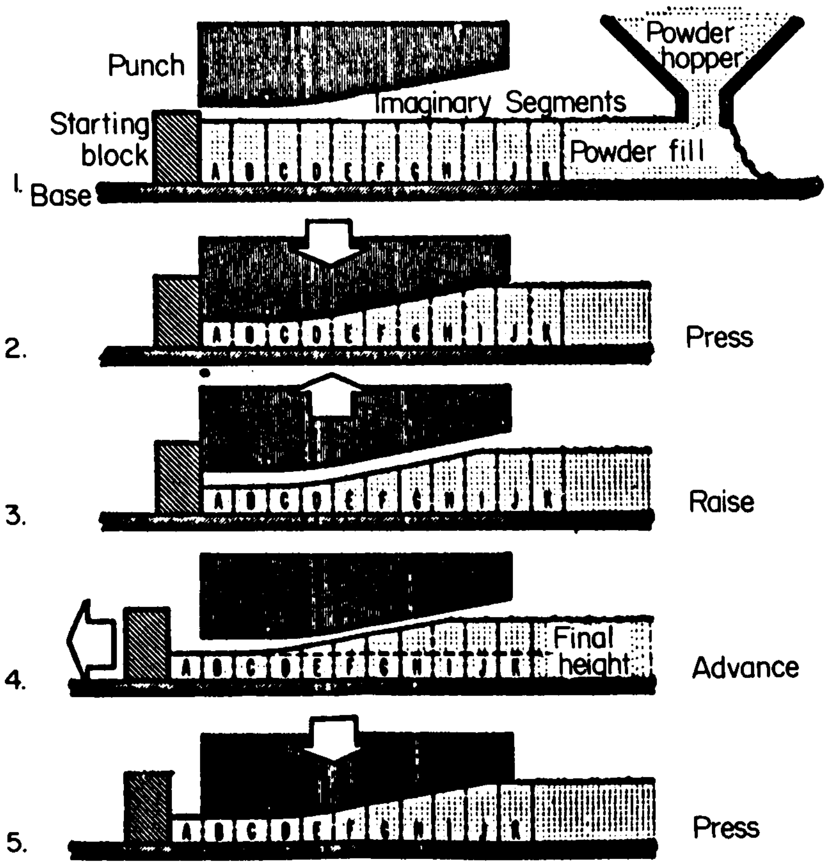
extruded from these hot-pressed billets. In the case of Udimet 700, the extrusion temperature was 2050 to 2100 F at extrusion ratios of 12 to 14. For Inconel 713 C alloy, corresponding ranges of temperature and extrusion ratios were 2100 to 2250 F and 12 to 14, respectively. For these two alloys, successful extrusion appeared to require low ram speeds (about 100 in. /min or less). Efforts to forge billets to shape indicated that adequate constraint on the billet by the die impression is necessary if cracking is to be avoided.

Cyclic Pressing. The compaction of nickel or nickel-alloy powders into bars by cyclic pressing has received a considerable amount of attention. Figure 17 illustrates the sequence of operations. The process has the advantage over direct rolling of powder because thicker strip or bars can be produced. In the continuous rolling process, even with large-diameter rolls, only relatively thin strip can be made. Figure 18 illustrates the steps used in fabricating an 80Ni-16Fe-4Mo magnetic alloy (Ref. 154). Figure 19 illustrates a processing schedule used for cyclic pressing carbonyl nickel powder (Ref. 155). This work demonstrated that pure nickel strip of high quality can be produced directly from a number of different types of nickel powder by cyclic pressing, sintering, and cold rolling (Tables XIX and XX). Of the five types of nickel powders examined, carbonyl nickel was the easiest to process, and one of the chemically precipitated powders was the most difficult. Four of the five types of nickel strip showed lower yield strengths, higher ultimate strengths, and higher elongation values than those listed for pure wrought nickel in the ASM handbook. This would imply a high degree of purity for the powder product. Nickel and nickel-base alloys have also been compacted by a cyclic operation in British work (Ref. 156) in which the compacting variables were analyzed. On the basis of the results of this work (Table XXI), the investigators concluded that almost any metal powder capable of being compacted under closed-die conditions can be cyclically compacted. The future of this process along with a number of other powder-metallurgy processes hinges on economic considerations.

Powder Rolling. Of all the compacting processes available for compacting nickel or nickel-base-alloy powders, there is a strong interest in the direct rolling of powder into strip or sheet form. In addition to the roll compaction of nickel powder direct roll compacting has been demonstrated for a number of other metals including stainless steel, copper, cobalt, and tungsten. Nickel powder is one of the more tractable materials for this type of compaction.



a. Cutaway Schematic View of Continuous-Compaction Apparatus



b. Sequence of Operations in Continuous Compaction

FIGURE 17. SCHEMATIC DIAGRAM OF CYCLIC-PRESSING PROCESS (REF. 154)

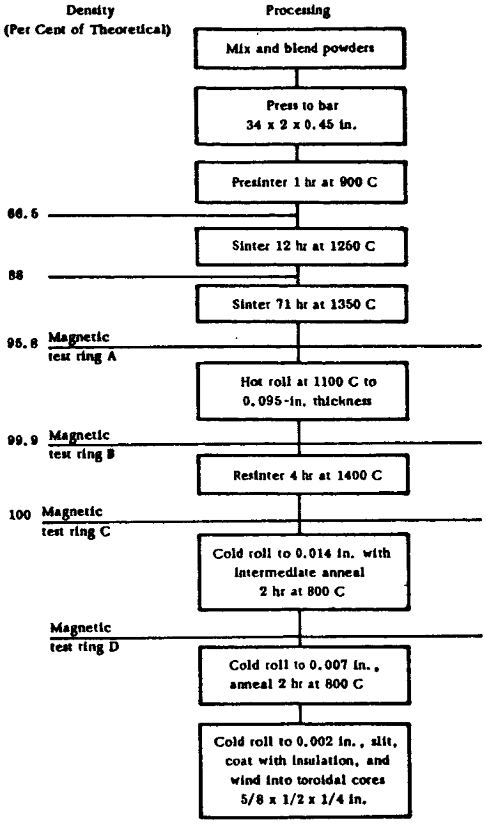


FIGURE 18. STAGES IN THE FABRICATION BY CYCLIC PRESSING OF AN 80Ni-16Fe-4Mo MAGNETIC ALLOY MADE FROM ELECTROLYTE NICKEL POWDER (REF. 154)

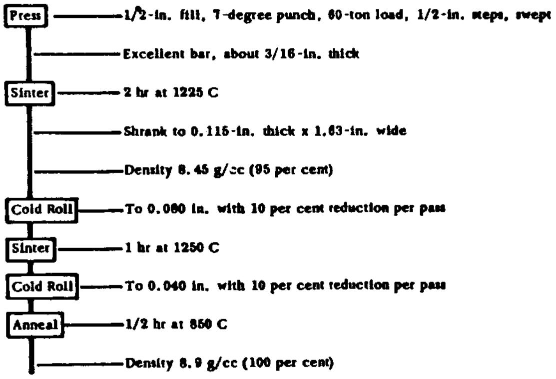


FIGURE 19. PROCESSING STEPS EMPLOYED IN PRODUCING NICKEL STRIP FROM CARBONYL NICKEL POWDER USING CYCLIC PRESSING (REF. 155)

TABLE XIX. MECHANICAL PROPERTIES OF ANNEALED PURE-NICKEL STRIP MADE FROM POWDERS (REF. 155)

Powder Type	Specimen	Strip Thickness, in.	Yield Strength, psi	Ultimate Strength, psi	Per Cent Elongation in 2 Inches[(a)]
Electrolytic	1	0.040	4,700	50,250	45.0
Electrolytic	2	0.040	4,700	50,250	44.0
Premium grade, high purity electrolytic	1	0.040	5,250	32,500	14.5
Premium grade, high purity electrolytic	2	0.040	5,250	36,500	20.0
Chemically precipitated "A"	1	0.035	6,000	47,350	35.9
Chemically precipitated "A"	2	0.035	7,550	45,400	28.5 Q
Carbonyl	1	0.040	8,150	51,750	39.6 Q
Carbonyl	2	0.040	7,750	51,400	37.4
Chemically precipitated "B"	1	0.040	5,650	50,750	50.5
Chemically precipitated "B"	2	0.040	4,400	49,750	49.0
Wrought material	99.95 per cent Ni + Co		8,500	46,000	30.0

(a) Q = quarter break.

TABLE XX. QUALITATIVE SPECTROCHEMICAL RESULTS OF THE VARIOUS NICKEL STRIPS IN THE ANNEALED FINISHED FORM (REF. 155)

Estimated Concentrations (Per Cent).

Elements Detected	Electrolytic	Pure Grade, High Purity Electrolytic	Chemically Precipitated "A"	Carbonyl	Chemically Precipitated "B"
Ni	Major	Major	Major	Major	Major
Fe	0.009-0.09	0.002-0.02	0.02-0.2	0.009-0.09	0.002-0.02
Al	0.003-0.03	0.002-0.02	0.003-0.03	0.003-0.03	0.003-0.03
Si	0.006-0.06	0.0009-0.009	0.009-0.09	0.002-0.02	0.002-0.02
Mn	0.0006-0.006	0.0003-0.003	0.003-0.03	0.002-0.02	0.0003-0.003
Cu	0.0009-0.009	0.00006-0.0006	0.0009-0.009	0.0003-0.003	0.0002-0.002
Mg	0.0006-0.0006	0.00006-0.0006	0.0002-0.002	0.0002-0.002	0.00003-0.0003
Co	0.02-0.2	--	0.02-0.2	--	0.02-0.2
Sn	--	--	0.002-0.02	--	--
Cr	--	--	0.003-0.03	--	--
Mo	--	--	--	0.002-0.02	--

Figure 20 illustrates an arrangement used for roll consolidating nickel powder. The cold compaction of the powder is ordinarily the most critical part of the operation. From geometrical considerations, the maximum thickness, T_2, of the issuing strip is (Figure 21):

$$T_2 = \frac{2r(1 - \cos \alpha)}{C - 1} ,$$

where $C = \frac{\text{density of the roll-compacted powder}}{\text{apparent density of uncompacted powder}}$

r = radius of the compacting rolls

α = maximum effective bite angle.

For nickel and copper powders, α was found to be about 7 degrees. As an example of the use of the above relationship assume that the rolls are 10 inches in diameter and that a 75 per cent dense strip is to be rolled from nickel powder having an apparent density of 3.5. With the resultant C value of 1.91, the maximum thickness, T_2, of green strip that can be made is 0.082 inch. The linear relationship of the expression for T_2 suggests that this figure might be doubled by doubling the diameter of the rolls.

Table XXII lists the mechanical properties of nickel strip made by the scheme in Figure 20. The unit pressure for roll compacting nickel

TABLE XXI. DETAILS OF PROCESSING RESULTS, AUTOMATED CYCLIC COMPACTING, FOLLOWED BY SINTERING AND COLD ROLLING (REF. 156)

		Green State			Sintered Condition				Cold-Rolled Condition					
							Hounsfield Three-Point Bend Test					Hounsfield Three-Point Bend Test		
Powder	Composition (As Analysed), wt %	Percentage (Solid Density)	Width, in.	Thickness, in.	Percentage (Solid Density)	Thickness, in.	Inclusive Bend Angle, degrees	Ultimate Stress, tsi	Percentage (Solid Density)	Thickness, in.	Percentage Reduction in Thickness	Inclusive Bend Angle, degrees	Ultimate Stress, tsi	Hardness, VPN (20-kg load)
Monel K type	C 0.16, Si 0.69 Mn 1.44, Cr 0.095, Ti 0.41, Al 2.93, Cu 29.4, Fe 3.18 + Ni	82.0	0.75	0.085	97.7	0.075	147	68.3	100	0.037	50.7	163	110	371
Moly-Perm-alloy type	C 0.016, Si 0.02, Mo 4.13, Fe 16.9 + Ni	78.6	0.75	--	80.1	0.086	163	17.49	96.4	0.065	24.5	145	63.3	164
		78.9	0.75	0.124	81.9	0.105	--	--	96.7	0.070	33.4	170	56.8	191
Monel type	C 0.1, Si 0.9, Mn 0.77 Cr 0.056, Cu 31.5, Fe 2.0 + Ni	88.5	0.75	--	--	0.072	124	34.25	100	0.048	33.3	158	75.8	261
		86.6	0.75	0.902	94.4	0.084	--	--	--	0.047	45	--	--	--
Nickel	Si 0.23, Co 0.044, Fe 0.85 + Ni	91.5	0.75	0.099	97.6	0.098	128	25.35	100	0.042	58	127	71.4	234
Stainless steel, type 316 L	C 0.029, Si 0.71, Cr 18.1, Ni 12.9, Mo 2.13 + Fe	90.7	0.75	0.110	--	0.110	--	--	99	0.055	50	135	101.3	329
		89.1	0.75	0.073	95.8	0.073	--	--	100	0.033	55.5	153	110.5	337
		83.8	0.75	0.045	96.5	0.044	--	--	100	0.015	59.5	--	--	333
		98.8	0.75	0.077	93.0	0.073	--	--	100	0.035	54.5	145	101.5	353

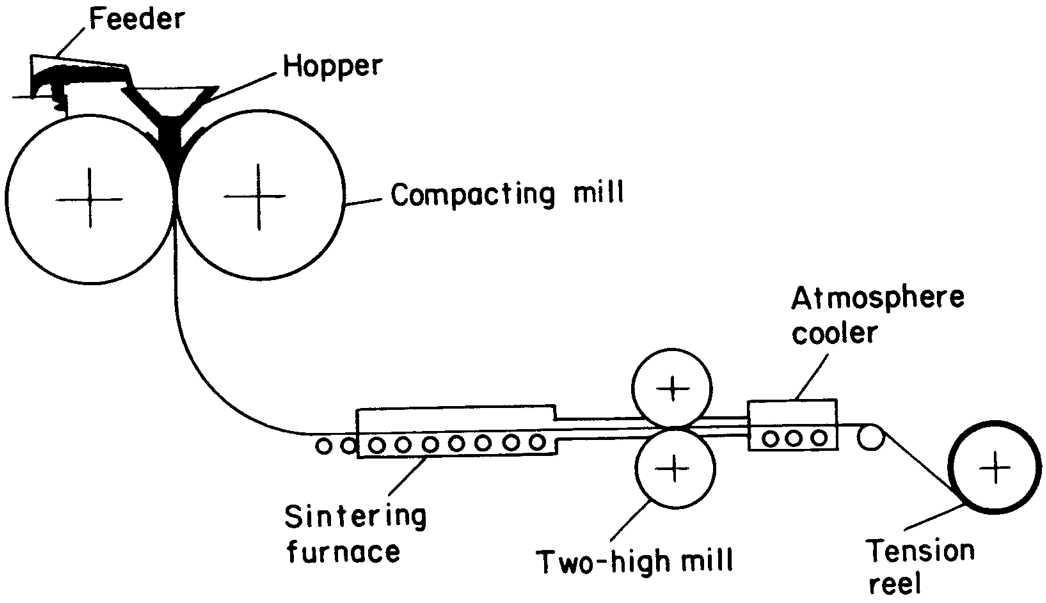


FIGURE 20. DIAGRAM OF CONTINUOUS POWDER-CONVERSION PROCESS (REF. 157)

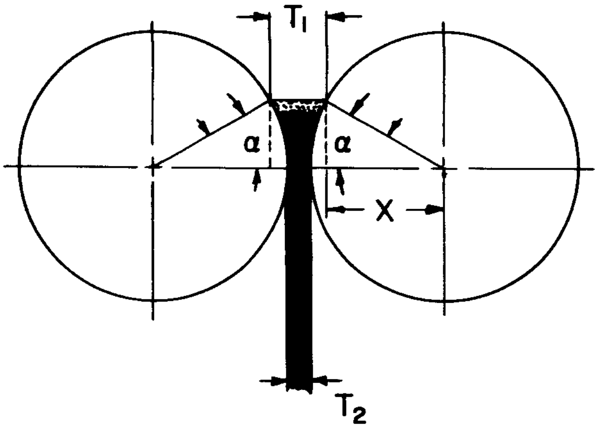


FIGURE 21. EQUATION THAT ESTABLISHES THE MATHEMATICAL RELATIONSHIP OF COMPACTOR ROLL RADIUS, r, TO MAXIMUM COMPACT THICKNESS, T_2, (REF. 157)

TABLE XXII. MECHANICAL PROPERTIES OF NICKEL STRIP MADE FROM POWDER BY A ROLLING PROCESS (REF. 157)

Thickness After Reduction, in.	Per Cent Reduction	Rockwell 15T	Tensile Strength, psi	Yield Strength, psi	Per Cent Elongation in 2 inches
0.0320	0	70.2	50,000	15,657	40.62
0.0110	65.7	90.6	97,900	93,722	0.87
0.0072	77.5	91.8	102,150	99,400	0.87
0.0067	79.0	92.1	104,300	100,500	1.00
0.0050	84.4	92.7	108,100	105,450	0.75

powders in this work was reported to be as high as 120,000 psi. A typical plant layout of the necessary mechanical equipment for converting 50 tons of powder to finished strip per a 24-hour day is presented in Reference 157. Reference 158 notes the successful pilot production of nickel strip from hydrometallurgically produced powder.

Table XXIII from a 1959 paper by Naeser and Zirm (Ref. 159) lists the patents dealing with powder-rolling processes in general. Evans (Ref. 160) has discussed the mechanics of powder behavior from a generalized standpoint, based on experimental work in a mill with roll axes in the same vertical plane. Figure 22 illustrates the hopper design used to introduce metal powders into the roll gap. This arrangement is less satisfactory than one in which the roll axes are disposed in a horizontal plane (Figure 23) (Ref. 161). Two basic procedures have evolved for feeding powder into the roll gap. In the

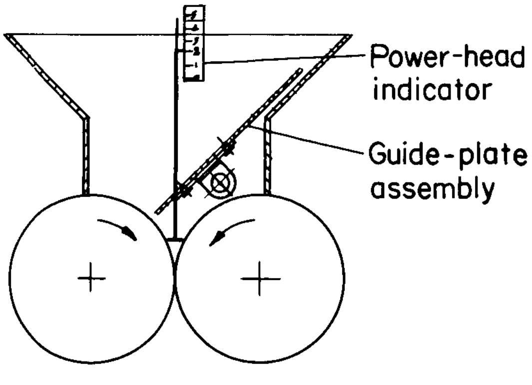


FIGURE 22. HOPPER FEED TO STANDARD TWO-HIGH MILL (REF. 160)

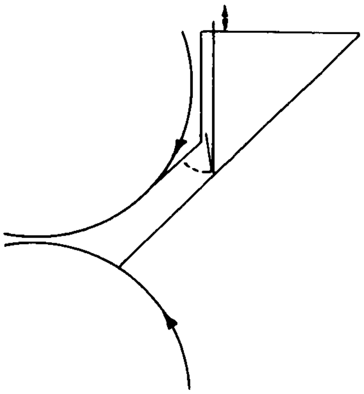


FIGURE 23. DIAGRAMMATIC ILLUSTRATION OF COMPACTING ROLLS AND POWDER-FEED HOPPER, SHOWING GUIDE-PLATE ASSEMBLY AND POWDER-HEAD INDICATOR (REF. 161)

TABLE XXIII. PATENT LITERATURE COVERING POWDER-ROLLING PROCESSES (REF. 159)

Country	Patent No.	Applied for by
		Rolling of Metal Powders
Germany	154,998	Siemens-Konzern
Great Britain	557,868	Minerals and Metals Corp.
	683,020	J. B. Brennan
	799,946	Stoesser, Sundwig
Switzerland	282,789	Mannesmann A.G.
U.S.A.	1,930,287	Moraine Production Co., Dayton O.
	2,134,366	Hardy Metallurgical Co.
	2,289,809	Chrysler Corp.
Germany	936,361	Mannesmann A.G.
	1,004,456	Ditto
	1,005,812	"
	1,023,657	Sundwiger Messingwerk
	1,027,041	Chemetals Corp. and Mannesmann A.G.
France	1,163,509	The Mond Nickel Co.
Austria	200,345	Ditto
		Appliances for the Rolling of Metal Powders
U.S.A.	2,341,732	General Motors Corp.
	2,350,179	Ditto
	2,758,336	H. Franssen
	2,851,354	J. P. Scanton, Yonkers
Germany	903,778	H. Franssen
	939,241	Ditto
	941,401	Mannesmann A.G.
	951,852	Ditto
	955,019	"
	957,618	Rochling, Völklingen
	959,789	H. Franssen
	961,413	Ditto
Great Britain	783,138	F. Heck, Hamburg
		Porous Facings and Sundries
U.S.A.	2,158,461	General Motors Corp.
	2,178,529	Chrysler Corp.
	2,198,253	General Motors Corp.
	2,198,254	Ditto
	2,222,251	Chrysler Corp.
	2,241,095	General Motors Corp.
	2,289,658	Ditto
	2,309,018	"
	2,332,746	"
	2,747,256	Aluminum & Brass Corp., Detroit, Mich.

saturated-feed system a workable head of powder is maintained at the roll bite. The quantity of powder passing through the roll gap to form strip is then dependent on the frictional force between the powder and the rolls, and the actual value of the resultant gripping angle [about 6 degrees maximum in the work of Evans (Refs. 160, 162) for the copper powder used]. In the saturated-feed system – the main alternative to the above method – there is no free powder in the roll bite. The feed rate is wholly dependent on a powder-dispensing system. Marshall (Ref. 163) lists several advantages for the unsaturated-feed system. For general discussions of the powder-compaction process, References 160 to 165 may be consulted. References 161 and 163 to 170 are oriented toward nickel or nickel-base-alloy powder rolling. Not all of them describe the latest equipment, but illustrate clearly the powder metallurgy involved.

In the production of nickel strip from powder as authored by Worn (Refs. 161, 165), the horizontally disposed rolls used were 7-5/8 in. in diameter x 12-in. face. They were driven by a variable speed d-c motor making possible continuous variations in speed of rolling over the range 2.5 to 10 ft/min. The roll surfaces were originally ground, without camber, to a 120-grit finish, but by the time about 5000 lb of nickel powder had been compacted, they had acquired a relatively stable semipolished surface. Strip from the compacting rolls was deflected into a horizontal plane by a polished guide plate and subsequently passed between spring-loaded scrapers arranged to continuously remove the soft low-density zone of the extreme edges (usually about 1/16 inch wide). The trimmed strip was then drawn through a sintering furnace by a coiling drum. Speed of the drum was synchronized with that of the rolling mill.

The 8-foot-long sintering furnace was fitted with a rectangular Inconel muffle with a water-cooled extension and was heated by four longitudinal silicon carbide resistor elements. Sintering was carried out in an atmosphere of cracked ammonia in the temperature range 1100 to 1150 C. The hot zone was nominally 18 inches long. To facilitate transport of the strip through the furnace, the muffle was fitted with a hearth plate of Nimonic 90, which provided a surface that was effectively "nonstick" at the temperature of operation. It was found that the rupture problems associated with relatively high-speed production could be overcome in part by the provision of a hydrogen atmosphere within the strip voids in place of the more viscous air.

Table XXIV lists the basic schedule finally adopted for processing the sintered strip. The increase in strip density with postsinter cold rolling is illustrated in Figure 24. Table XXV indicates that the

TABLE XXIV. BASIC SCHEDULE EMPLOYED IN THE PROCESSING OF SINTERED NICKEL STRIP (REF. 161)

Stage	Operation
1	Cold rolled dry from 0.030 to 0.024-in. thickness
2	Strand annealed in cracked ammonia at 1050 C; strip speed 1 ft/min through 4-ft hot zone
3	Cold rolled dry to 0.015-in. thickness
4	Strand annealed as in Stage (2) at 800 C; strip speed 4 ft/min
5	Cold rolled with lubricant to 0.008 in.
6	Strand annealed as in Stage (2) at 700 C; strip speed 4 ft/min
7	Cold rolled with lubricant to 0.005 in.
8	Strand annealed as in Stage (2) at 700 C; strip speed 6 ft/min

Note: Material finished "half hard", annealed at a thickness of 0.0055 in., and cold rolled to 0.005 in. without further treatment.

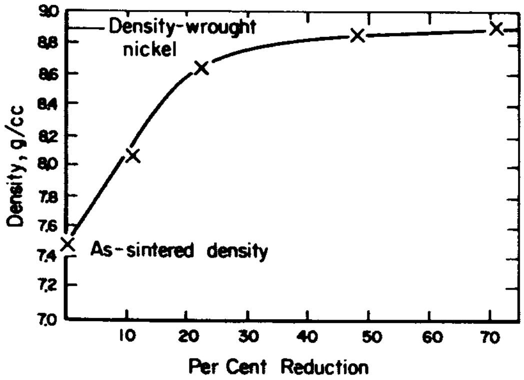


FIGURE 24. DENSITY INCREASE OF DIRECTLY ROLLED AND SINTERED NICKEL STRIP

Strip is initially 0.027 in. thick; cold rolled in successive stages; strip continuously annealed between every two stages (Ref. 161).

TABLE XXV. MECHANICAL PROPERTIES OF NICKEL STRIP PRODUCED BY THE DIRECT-ROLLING AND CONVENTIONAL PROCESSES (REF. 161)

Material	Tensile Strength, tsi	Elongation(a), per cent	Erichsen Value, mm
Directly rolled nickel			
Soft(b)	24.4	19.0	8.91
Half-hard(c)	29.8	14.5	6.22
Hard (64 per cent reduction)	36.5	~1	3.10
Conventionally made nickel			
Soft(b)	24.1	20.3	9.25
Half-hard(c)	34.0	17.7	7.29
Hard (38 per cent reduction)	44.3	~1	4.50

(a) Measured on standard tensile-test pieces, 2-in. gage length, cut from 0.005-in.-thick strip; test piece pulled in direction of rolling.

(b) Annealed to give a grain size of 0.02 mm; directly rolled nickel annealed for nominal period of 30 sec at 700 C; conventionally made nickel annealed for similar period at 925 C.

(c) Cold rolled from 0.0055 to 0.005 in. subsequent to final anneal.

properties of the powder-metallurgy strip were similar to those of melted, cast, and worked material. It may be noted, however, that the powder-metallurgy strip exhibited a relatively low work-hardening capacity, and that the ultimate tensile strength of the material cold rolled to 64 per cent reduction, for example, was significantly lower than that of conventionally made material rolled to only 38 per cent reduction. This difference was consistent with the higher purity of the powder-metallurgy material (Table XXVI – powder: 99.95 per cent Ni, cast and wrought: 99.6 per cent).

TABLE XXVI. TYPICAL ANALYSES OF NICKEL STRIP PRODUCED BY THE DIRECT-ROLLING AND CONVENTIONAL PROCESSES (REF. 161)

Element	Directly Rolled Strip, per cent	Conventionally Produced Strip, per cent
Carbon	0.01	0.04
Oxygen(a)	0.002	--
Nitrogen(a)	<0.001	--
Sulfur	0.001	0.002
Iron(b)	0.005	0.1
Silicon(b)	0.005	0.05
Manganese(b)	0.005	0.03
Aluminum(b)	<0.01	--
Magnesium(b)	0.005	0.1
Copper(b)	0.005	0.1

(a) Vacuum-fusion gas-analysis data.

(b) Spectrographic data.

The advantages of powder-metallurgy nickel strip over conventional cast and worked strip are as follows (Ref. 166):

(1) Better control of composition

(2) Improved electrical and mechanical properties made possible by controlled oxidation of impurities

(3) Unusual combinations of properties that can be "built into" material prepared by the powder-strip process. For example, electrical passivity and mechanical strength at high temperature can be achieved within the same material.

Table XXVII (Ref. 170) lists Sherrit Gordon's strip specifications. Figure 25 (Refs. 167, 170) illustrates Canadian coinage now being made from it. Although coinage from nickel strip directly rolled from powder does not represent parts fabrication, it is a full-production process.

Reference 168 describes one of a number of Soviet studies in nickel-powder rolling. The powders rolled in this study were as follows: Ni, Ni-Mo, Ni-Si, Ni-Ti, Ni-Cu-Al, and Ni-Ba-Al. The results of the study were considered to confirm the possibility and indicate the means of controlling thickness and density of strip rolled from metal and alloy powders.

FIGURE 25. CANADIAN FIVE-CENT COIN MINTED FROM A BLANK PRODUCED BY SHERRITT GORDON FROM DIRECT-ROLLED NICKEL STRIP (REF. 170)

The controlling factor in the continued application of direct-powder-rolling procedures to full-scale-production operations is more a matter of economy than of technology.

SINTERING

The rate of densification of nickel or nickel-base-alloy powders, or any other metal powders, is dependent on many factors during

TABLE XXVII. PROPERTIES OF SHERRITT GORDON GRADE SG-100 NICKEL STRIP (REF. 170)

Typical Chemical Analysis, per cent

Nickel	99.85 min	Iron	0.040
Cobalt	0.08	Sulfur	0.003
Copper	0.006	Carbon	0.008
		All others	0.001 or less

Typical Mechanical and Physical Properties

(Dead-Soft Condition)

Ultimate Tensile Strength, psi	45,000 to 55,000
Elongation, per cent	35 to 50

Nominal specific resistance, 46.0 ohms/cmf
Thermal expansion coefficient, 20 to 100 C per C 0.000014 in./in.

	Typical Hardness Figures			
	VHN	15T(a)	30T(a)	B Scale(a)
Strip thickness, in.	0.005 - 0.010	0.010 - 0.020	0.020 - 0.040	0.040 min
Dead soft	89 max	72 max	40 max	38 max
Soft, fine grain	104 max	78 max	54 max	60 max
Skin hard	104 - 116	78 - 80	54 - 57.5	60 - 65
Fourth hard	116 - 127	80 - 82.5	57.5 - 62	65 - 71
Half hard	127 - 150	82.5 - 85.5	62 - 68	71 - 80
Three-fourths hard	150 - 167	85.5 - 87.5	68 - 72	80 - 86
Hard	167 min	87.5 min	72 min	86 min
Full hard	198 min	89.5 min	76 min	92 min

(a) Rockwell scales.

Notes: No order accepted for thicknesses above 0.035 in.
No hard order accepted for thicknesses above 0.025 in.
No full-hard order accepted for thicknesses above 0.020 in.
No fine-grain order accepted for thicknesses above 0.010 in.

sintering. In general the classical diffusion equation, $D = D_o e^{-Q/RT}$, applies in at least a qualitative manner. Because of its exponential position in the expression, the effect of temperature is very great. However, there is a practical limit to increasing the sintering temperature because of factors such as furnace deterioration, excessive grain growth, or the appearance of an undesirable liquid phase might occur. Usually a combination of several mechanisms is operative during sintering, as indicated in Table XXVIII. The dominant mechanism may change with time or with temperature variations. The factors affecting the overall consolidation rate in solid-state sintering are summarized in Table XXIX. For practical purposes observation of the resultant effect of these factors is usually sufficient for specific instances.

The subject of sintering furnaces and appertenant atmosphere is too broad to be considered in detail. Figure 26 illustrates only one of many furnace types applicable to sintering of nickel-powder parts. Reference 123, manufacturers' catalogues, and other sources describe sintering furnaces and related equipment.

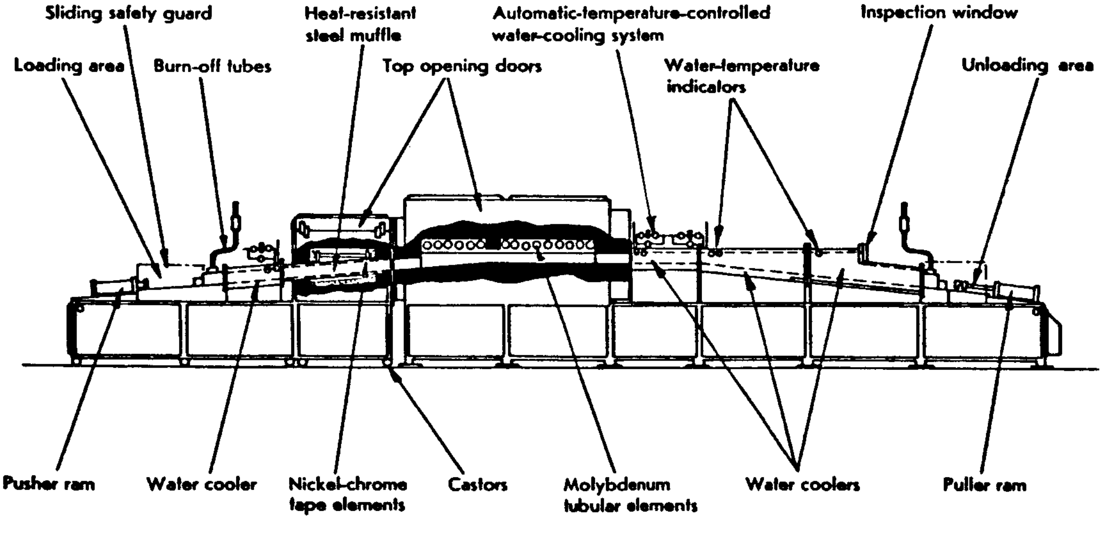


FIGURE 26. HUMPED-BACK, BOAT-TYPE, PUSH-PULL FURNACE (REF. 172)

Courtesy of Sintering & Brazing Furnaces Ltd., England.

TABLE XXVIII. POSSIBLE ELEMENTARY MECHANISMS INVOLVED IN SINTERING (REF. 171)

(1) Without transport of material	Adhesion	
(2) With transport of material (atom movements over long distances)	Surface diffusion Lattice diffusion (vacancies) Lattice diffusion (interstices) Grain-boundary diffusion Evaporation, condensation	Movement of individual crystal-lattice components
	Plastic flow Viscous (and quasiviscous) flow	Movement of whole regions in the lattice
(3) With transport of material (atom movements of the order of atomic distances)	Recovery or recrystallization	

TABLE XXIX. FACTORS AFFECTING SINTERING PROCESSES (REF. 171)

Liquid Phases Absent.

(A) Primary Factors - present in any sintering process

- (1) Total surface contact
- (2) Grain-boundary orientation
- (3) Diffusion coefficients
- (4) Surface tension
- (5) Viscosity coefficients
- (6) Critical shear stress (if plastic flow occurs)
- (7) Vapor pressure, vaporization rate (if vaporization, condensation occur)
- (8) Crystal structure, binding energy

(B) Secondary Factors - may or may not take part in the process.

- (1) Surface activity
- (2) Lattice activity (cold work, tensile and/or compressive stresses, lattice defects introduced during fabrication)
- (3) Activated (nascent) states; allotropic changes
- (4) Foreign constituents
  - (a) Soluble (homogeneous or heterogeneous)
  - (b) Insoluble
  - (c) Surface films (e.g., oxide skins soluble or insoluble during sintering, reducible or nonreducible, dissociable or nondissociable)
  - (d) Gases (adsorbed, occluded, dissolved, as well as sintering-atmosphere effect)

TABLE XXX. COMPOSITIONS OF A NUMBER OF ATMOSPHERES USED IN FURNACE TREATMENTS (REF. 172)

Type of Atmosphere	Atmosphere No.	Air/Gas Ratio	Composition, per cent						Dew Point	
			H_2	CO	CO_2	O_2	CH_4	N_2	F	C
Exothermic Hydrocarbon Gas										
Lean - unpurified	1	Approx 4:1	0.2-0.5	0.5-1.0	11-14	Nil	1	83	Saturated	
Lean - purified	2	4:1	1	2	Trace	Nil	1	95+	-40	-40
Rich - unpurified	3	2.5:1	9	12	6	Nil	2	71	Saturated	
Rich - purified	4	2.5:1	12	15	0.1	Nil	2	71	-40	-40
Endothermic Hydrocarbon Gas										
Lean endothermic (wet)	5	1.5:1	40	20	2	Nil	Trace	38	Saturated	
Rich endothermic (dry)	6	0.6:1	46.5	25	Trace	Nil	0.4	28	10	-12
Dissociated Ammonia										
Unburnt	7	--	75	--	--	--	--	25	-40	-40
Partially burnt	8	1:1 (NH_3)	24	--	--	--	--	76	Saturated	
Completely burnt	9	1.75:1 (NH_3)	1	--	--	--	--	99	Saturated	
Nitrogen (bottled - purified)	10	--	1	--	--	--	--	99	-67	-55
Hydrogen (bottled - purified)	11	--	100	--	--	--	--	--	-60	-51
Carbon Dioxide (bottled - unpurified)	12	--	--	--	99.8	--	--	0.2	-30 to +27	-34 to -3

Furnace atmospheres for sintering nickel or its alloys are also a somewhat flexible matter, depending on process economics, quality of product required, and equipment available. In general a somewhat reducing, sulfur-free atmosphere is adequate. For some alloys, e.g., those containing appreciable amounts of chromium, low-dew-point atmospheres or, in some cases, even vacuum may be called for. Table XXX lists some commercial atmospheres that may be used in sintering of metal powders. Vacuum (not included in the list) may be required for powders unusually sensitive to contamination. Atmospheres 1, 2, 6, 7, or 11 are available for nickel powder. However, the first is commonly used for economical reasons. For nickel-chromium alloys Atmospheres 7 or 11 yield desired results. Ordinarily, Atmosphere 7 is used because of its lower cost.

The general effects of precompaction levels and sintering temperatures on densification rates of nickel are illustrated in Figure 27. The adverse effect of lower as-cold-compacted density is diminished as higher sintering temperatures are used. Ordinarily, there is a practical limit to the compaction pressures used, because excessive pressures require higher capacity presses and because the compact may fracture on the release of elastic forces upon ejection from a die.

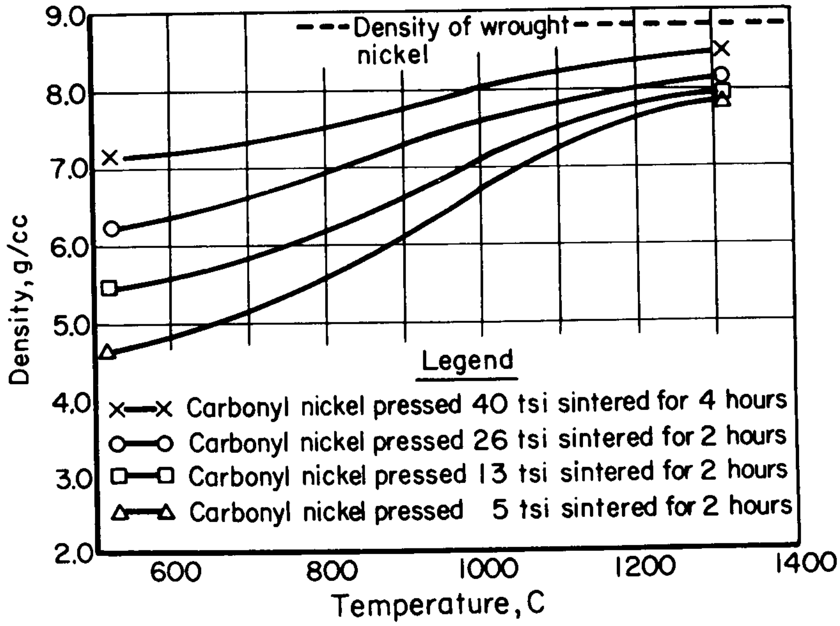


FIGURE 27. EFFECT OF PRECOMPACTION LOADS AND TEMPERATURE ON THE DENSIFICATION OF NICKEL POWDERS (REF. 126)

Furthermore, density-increment advantage tends to fall off with increasing pressure (Table XXXI).

TABLE XXXI. DENSITY OF NICKEL SAMPLES
(REF. 173)

Sample	Pressure, psi	As-Sintered Density[(a)]
1	140,000	8.722
2	120,000	8.787
3	100,000	8.783
4	80,000	8.763
5	60,000	8.725
6	40,000	8.624
7	20,000	8.480

(a) 32 hr, 1375 to 1400 C.

The shrinkage of nickel-powder compacts has been investigated extensively by Tikkanen and Mäkipirtti (Refs. 174, 175). Figure 28 shows the particle-size distributions of two types of powders used in this work. Figures 29 and 30 compare the different amounts of shrinkage of the two powders. The shrinkage (i. e., the densification) of the carbonyl nickel powder amounted to 15 per cent in 2 hours at 1200 C; whereas, in the case of the atomized powder, a 15-hour sinter at 1300 C resulted in a shrinkage of less than 6 per cent. These differences in consolidation rate are related to the differences in particle size and density of the two powders. The shrinkage of carbonyl-nickel-powder compacts has also been studied by Margerand and Eudier (Ref. 176), who concluded that the extent of elimination of porosity was dependent on the size of the compact. Tracey and Perks (Ref. 177), Prill and Upthegrove (Ref. 178), and Evans and Ashall (Ref. 179) also studied the sintering of nickel powder. Figures 31 and 32 and Tables XXXII to XXXIV illustrate properties obtainable with INCO 100 (carbonyl) powder. It was concluded that

(1) Sintered carbonyl nickel parts, produced using conventional sintering times and temperatures with moderate compaction pressures, exhibit good strengths and elongations, exceptional toughness, and high densities.

(2) The addition of zinc stearate to carbonyl nickel powder increases sintered strength and impact toughness.

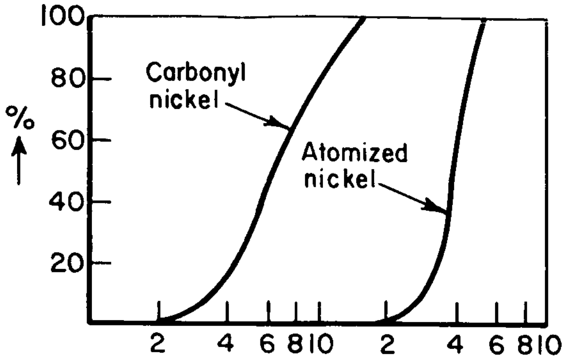


FIGURE 28. PARTICLE-SIZE DISTRIBUTION OF TWO NICKEL POWDERS (REF. 174)

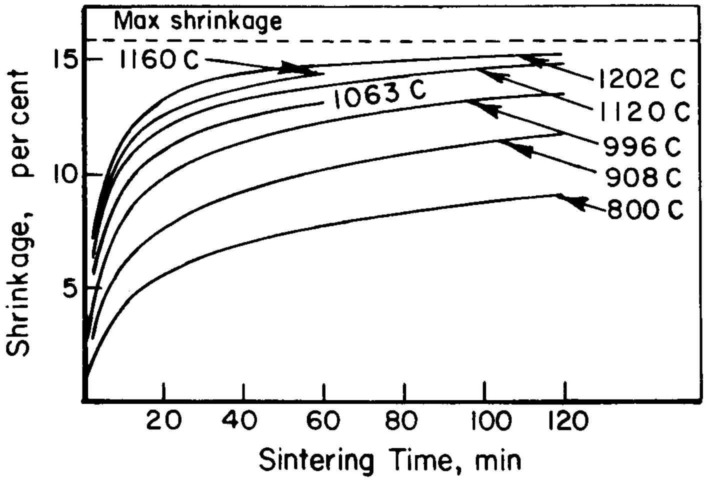


FIGURE 29. SINTERING OF CARBONYL NICKEL POWDER (REF. 174)

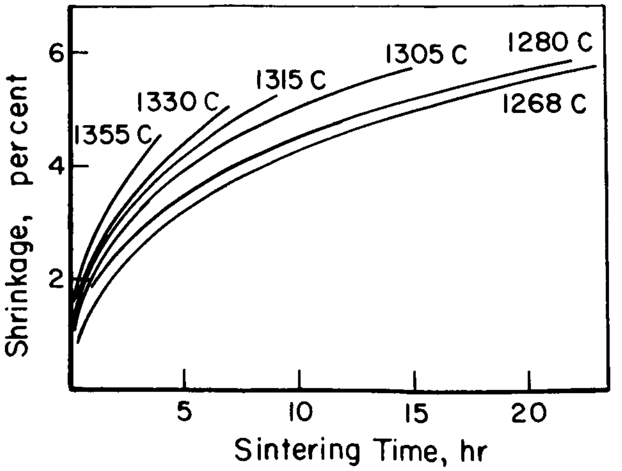


FIGURE 30. SINTERING OF ATOMIZED NICKEL POWDER (REF. 174)

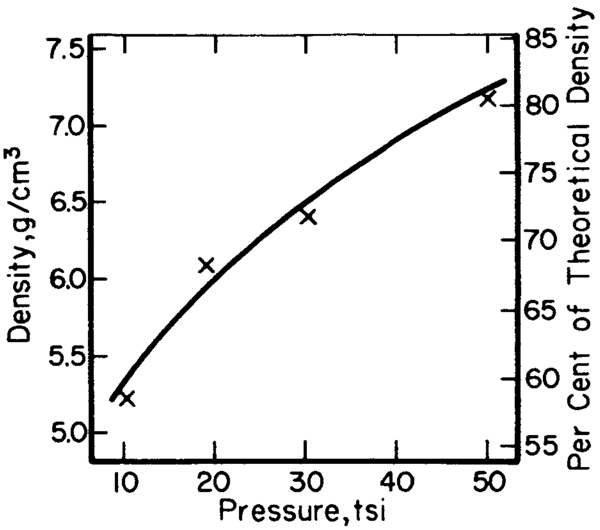


FIGURE 31. GREEN DENSITY AS A FUNCTION OF COMPACTION PRESSURE FOR CARBONYL NICKEL-POWDER TYPE 100 CONTAINING 1 PER CENT ZINC STEARATE (REF. 178)

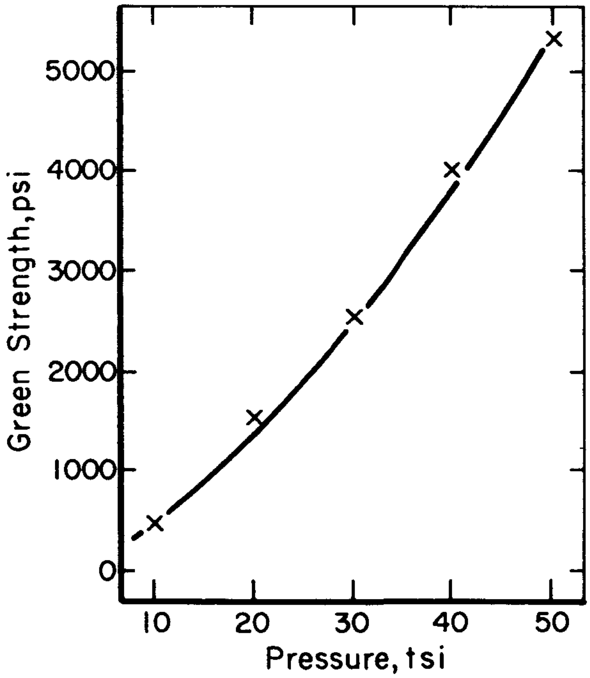


FIGURE 32. GREEN STRENGTH AS A FUNCTION OF COMPACTION PRESSURE FOR CARBONYL NICKEL-POWDER TYPE 100 CONTAINING 1 PER CENT ZINC STEARATE (REF. 178)

TABLE XXXII. TYPICAL PROPERTIES OF SINTERED CARBONYL NICKEL POWDER 1[(a)] (REF. 178)

Compaction Pressure, tsi	Sintering[(b)] Temperature, F	Sintering[(b)] Time, Min	Green Density[(c)], per cent	Change in Diameter, per cent	Change in Thickness, per cent	Final Density, per cent	Yield Strength (0.2 % Offset), psi	Ultimate Tensile Strength, psi	Elongation in 1 Inch, per cent	Hardness, R_B
--	--	5	71.4	5.95	3.85	84.1	17,550	33,100	13.0	15
--	--	15	71.5	5.20	--	84.2	16,750	44,700	21.0	25
30	2000	30	71.7	8.79	5.96	91.2	14,600	45,200	19.5	28
--	--	45	71.6	9.14	6.03	92.3	13,750	44,100	19.5	28
--	--	60	71.5	9.45	6.20	93.0	11,800	44,040	19.0	25
--	--	1440	71.5	10.80	7.60	97.5	8,200	46,300	34.0[(d)]	--
30	2100	5	71.5	6.26	3.75	85.3	19,100	39,200	16.0	--
50	2000	5	79.9	4.10	2.70	89.6	21,000	45,800	14.0	31
--	--	5	80.2	4.84	3.08	91.5	21,400	51,000	17.0	28
50	2100	15	80.2	5.74	3.70	94.1	14,500	55,000	31.5	--
--	--	30	80.3	6.24	4.12	95.1	10,600	55,300	34.5	24
70	2100	5	85.9	3.29	2.47	94.4	19,700	58,900	26.5	45

(a) Each datum represents an average of two or more identical specimens.

(b) Sintered in 5-inch-diameter semiproduction furnace. Hydrogen atmosphere had a dew point of -20 to +20 F.

(c) Per cent of theoretical density.

(d) Specimen necked extensively.

TABLE XXXIII. MECHANICAL PROPERTIES OF SINTERED NICKEL AND HIGH-PURITY WROUGHT NICKEL

		Sintering Conditions					
Material	Yield Strength (0.2% Offset), psi	Pressure, tsi	Temperature, F	Time, min	Density, per cent	Tensile Strength, psi	Elongation, per cent
Nickel 270[b]							
Rod and bar, hot finished	13,000	--	--	--	--	50,000	50
Strip, cold rolled, annealed	13,000	--	--	--	--	50,000	45
Sintered carbonyl nickel	--	5.7	2370	120	87.5	36,000	<10
powder (data of Schlecht	--	28.4	2370	120	91.0	42,000	<12
and coworkers)	--	66.4	2370	120	93.5	52,000	<15

(a) Values given for Nickel 270 represent typical values. Values for sintered compacts are single datum points.

(b) A product of Huntington Alloy Products Division, The International Nickel Company, Inc., Huntington, West Virginia.

TABLE XXXIV. MECHANICAL PROPERTIES OF COINED CARBONYL NICKEL 1 SINTERED COMPACTS (REF. 178)

	Sintering							
Compaction Pressure, tsi	Temp, F	Time, min	Coin Pressure, tsi	Final Density, per cent	Yield Strength (0.2% Offset), psi	Ultimate Tensile Strength, psi	Hardness in 1 Inch, per cent	Elongation, R_B
30	2050	10	0	89.2	25,100	45,200	10.0	20
			30	89.3	49,200	64,500	12.5	57
			50	91.2	62,100	72,100	7.5	66
50	2050	10	0	93.5	19,800	61,300	18.5	38
			30	93.5	47,800	72,000	25.5	64
			50	93.6	59,300	74,700	21.5	70

(3) A 1200 F presintering treatment markedly reduces the mechanical strength of sintered carbonyl powder with little effect on ductility.

(4) Changes in the purity of the sintering atmosphere markedly affect the mechanical properties of sintered carbonyl nickel powder.

(5) Coining can be of significant value in greatly increasing the strength of sintered carbonyl nickel powder with little or no reduction in ductility.

Figure 33 from Evans and Ashall shows the effect of sintering time at five different temperatures on carbonyl nickel. The activation energy, Q, for grain growth was found to be 29.3 kcal/g atm.

The sintering of low-density porous plates from nickel powder is the subject of References 177 and 180. In the former, carbonyl powder was used; in the latter hydrometallurgically produced powder.

The existence of activated states as a factor affecting sintering processes was listed in Table XXIX. An example pertinent to nickel is that of oxide-film reduction during the sintering operation. Figure 34 illustrates the strengthening effect resulting from the initial presence a thin oxide film on carbonyl nickel (Ref. 179). To obtain a maximum in tensile properties the critical oxide-film thickness was about 625 A. In a hydrogen atmosphere the accelerated sintering is accounted for by the additional chemical reactivity due to reduction of the oxide film. In vacuum and in nitrogen, accelerated sintering is ascribed to increased diffusion and to evaporation and condensation. It was noted earlier that the oxide of nickel is less stable than many other metal-powder oxides. The favorable sintering behavior observed in the case of thin oxide on nickel is not to be expected for any nickel-base alloy in which the oxides are substantially more stable.

NICKEL-BASE ALLOYS

Table XXXV lists references dealing with a variety of nickel-base alloys that have been made from powders. (Those strengthened with Al_2O_3 or ThO_2 are not included.) Alloys that are easily maintained in a reduced condition during sintering, e.g., Ni-Cu or Ni-W, have been made by sintering the elemental powders. Alloys having components difficult to maintain in a reduced condition have been made from prealloyed or partially prealloyed powders.

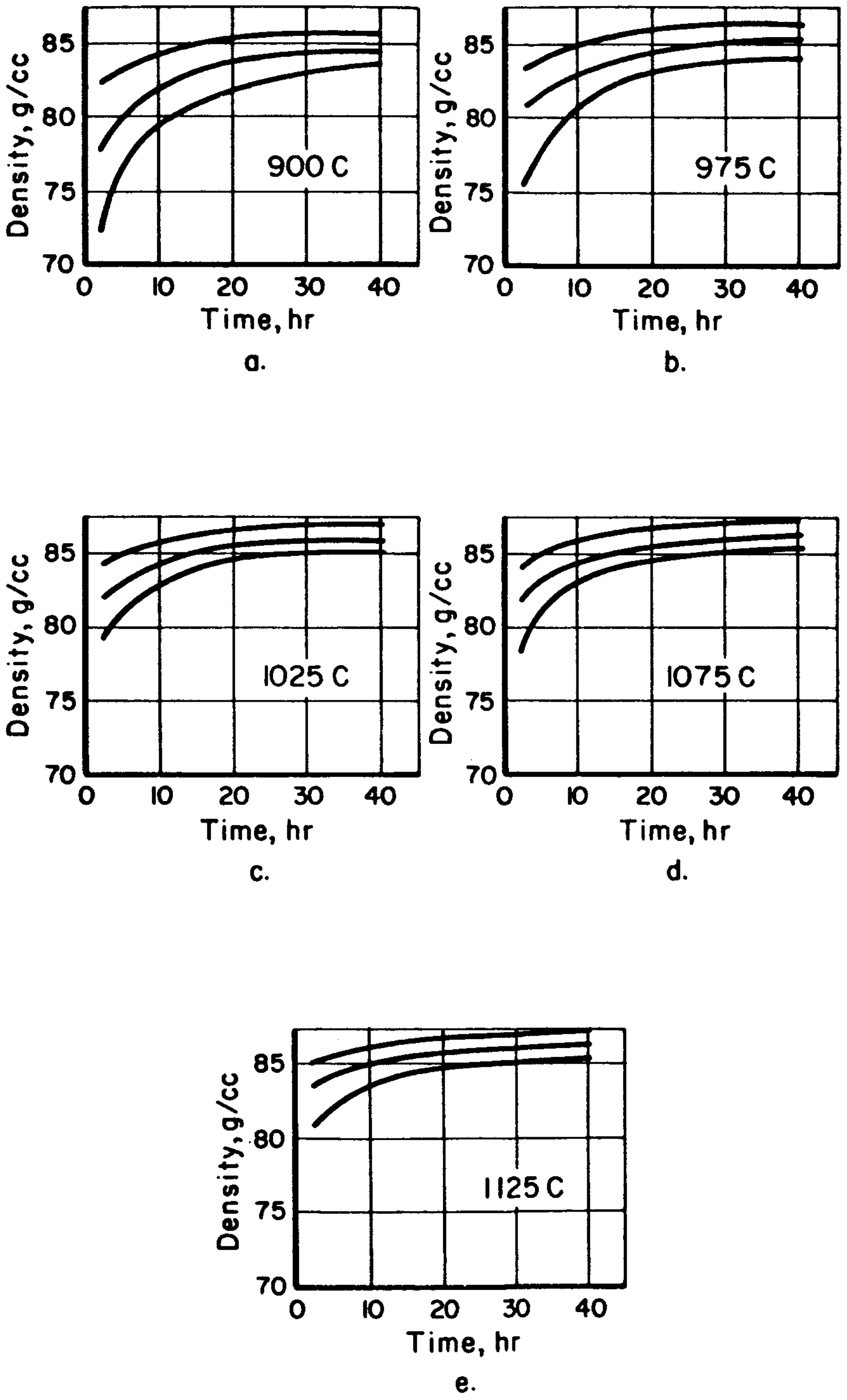


FIGURE 33. EFFECT OF SINTERING TIME ON THE DENSITY OF CARBONYL NICKEL COMPACTS SINTERED BETWEEN 900 AND 1125 C (REF. 179)

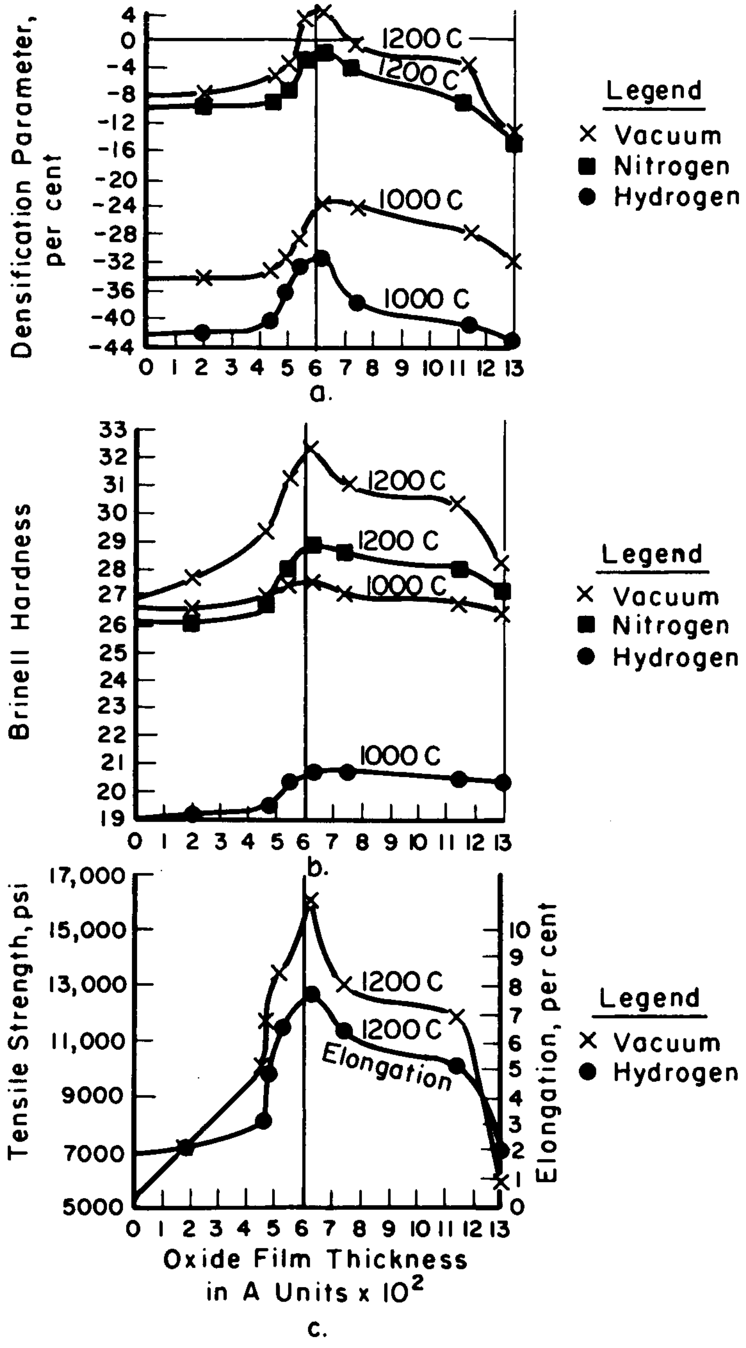


FIGURE 34. EFFECT OF INITIAL OXIDE-FILM THICKNESS ON DENSIFICATION, BRINELL HARDNESS, AND TENSILE STRENGTH FOR CARBONYL NICKEL POWDER SINTERED IN DIFFERENT ATMOSPHERES (REF. 181)

TABLE XXXV. REFERENCES DEALING WITH POWDER-METALLURGY NICKEL-BASE METALLIC ALLOYS

System	Reference
Ni-Cu	182-189(a), 190(b)
Ni-Mo-Fe	191(a)
Ni-Ta	192(a)
Ni-TaC	
Ni-Ta-TaC	
Ni-Fe	190(a), 193(b), 194(a)
Ni-W	195(a)
Superalloys	
Ni-Co-Cr (Nimonic)	196-200(b)
Ni-Cr-Co (Udimet 500)	190(b), 201(b) (Udimet 700)
Ni-Co-Cr (Cosint 1000)	202(b)
Ni-Co-Fe	203(b)
Ni-Al	204-207(b)
Ni-Al-Cr	208(b)
Ni-Al-Ti	
Ni-Al-Zr	
Ni-Al-Nb	
Ni-Fe-Mo-Cu	194(a)

(a) Elemental powders.

(b) Prealloyed or partially prealloyed powders.

In the sintering of elemental powders to yield an alloy by solid-state diffusion, there is usually a problem of estimating the time required at temperature to arrive at a substantially homogeneous alloy. A rough approximation may be made based on Fick's law (Ref. 209). Closer estimates require experimental determinations in view of the unevaluated factors affecting the rate of sintering.

The production of high-temperature nickel-base alloys by powder metallurgy is of considerable current interest. Figure 35 indicates an advantage in stress to rupture of powder-metallurgy Nimonic 100 over the wrought product. The advantage in this instance was partially offset by a lower hot-fatigue-endurance life (Table XXXVI). References 197, 200, and 202 are especially good sources of information on this type of alloy.

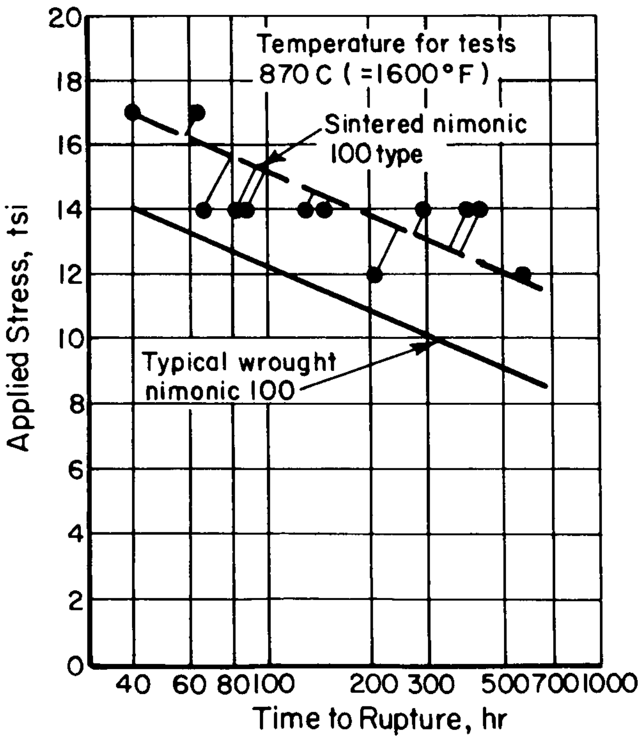


FIGURE 35. STRESS/TIME-TO-RUPTURE DATA COMPARING WROUGHT NIMONIC 100 AND SIMILAR SINTERED MATERIAL (B. S. A. DATA) (REF. 196)

The sintering of alloys in this general category requires more care than that normally required for nickel powder or simple nickel-base alloys, e.g., Ni-Fe. The atmosphere must contain lesser

TABLE XXXVI. PRESSED AND SINTERED VERSUS TYPICAL WROUGHT NIMONIC 100 (REF. 196)

Specimen Test Temperature, 870 C	Stress to Rupture	Hot-Fatigue Endurance
Typical wrought Nimonic 100	1.0	1.0
Pressed and sintered Nimonic 100-type powder	1.3	0.75 - 0.90 (According to process)

amounts of oxidizing gases; some of the alloys are best sintered in a vacuum of better than 1 micron. In some instances the workable sintering range is narrow. For example, for prealloyed Udimet 700 powder the optimum sintering range is 2350 to 2370 F, and for Inconel 713 C it is 2450 to 2480 F (Ref. 190). The maximum temperatures in these cases are limited by liquid-phase formation. The lower temperatures are limited by the lack of sufficient densification. Figure 36 illustrates the densification rates of these alloys as a function of temperature.

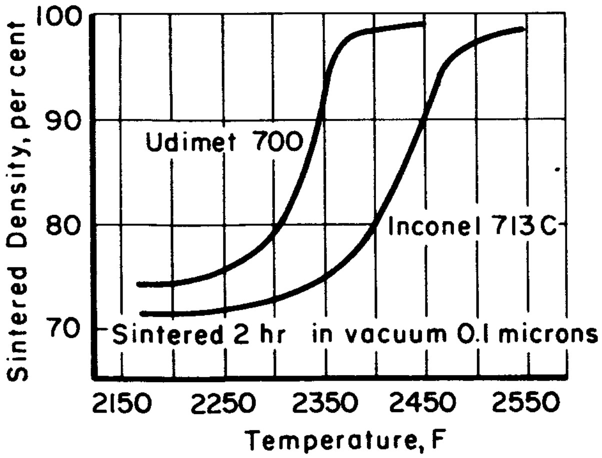


FIGURE 36. DENSIFICATION RATES OF UDIMET 700 AND INCONEL 713C AS A FUNCTION OF TEMPERATURE (REF. 190)

The pressing and sintering characteristics of prealloyed powders are influenced strongly by the sizes and shapes of individual particles. As can be seen from Figure 37, the green strength in particular is influenced by particle size.

References 210 through 213 refer to the production of nickel alloys for magnetic applications.

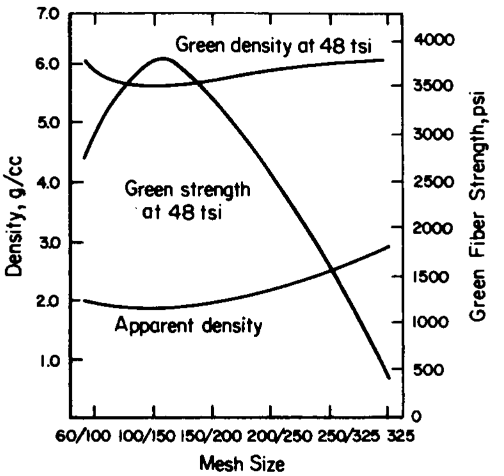


FIGURE 37. EFFECT OF PARTICLE SIZE ON PRESSING CHARACTERISTICS (REF. 190)

INERT-DISPERSOID-STRENGTHENED NICKEL

At very high service temperatures, strengthening, which is dependent on heat treatment (for example, an aging treatment) tends to be rendered ineffective. Some strengthening mechanism that is comparatively stable thermodynamically at elevated temperatures is called for, e.g., either solid-solution strengthening or inert dispersoid strengthening. A considerable amount of work has been done recently with the latter class of alloys. The classical example of this type of strengthening is thoriated tungsten, and more recently Du Pont's TD nickel.

References 214 and 215 include general information on dispersion strengthening. Table XXXVII lists some sources of information on dispersion-strengthened alloys.

Although the detailed mechanics of dispersion strengthening are still arbitrary matters, there is general agreement that the inert dispersoids block or at least delay the movement of dislocations and thus inhibit plastic deformation. For this reason interparticle spacing of dispersoids should be small – less than 1 micron. For a fixed volume per cent of dispersoid, say 2 per cent, the particle must be small in order to reduce the interparticle distance to small values. Figure 38 shows calculated interparticle spacings for oxide dispersoids of various diameters and present in amounts up to 50 volume per cent.

TABLE XXXVII. REFERENCES DEALING WITH INERT DISPERSOIDS IN NICKEL

System	References
Ni-ThO_2	216-222
Ni-Al_2O_3	221, 223-225, 226-230
Ni-Cr-TiC Ni-Cr-Al_2O_3 Ni-Cr-MgO Ni-Cr-ThO_2	231, 227
Ni-MgO	221,229, 232
Ni-Cr_2O_3	228, 229, 230
Ni-SiO_2	229
Ni-TiO_2	229
Ni-CaO	232

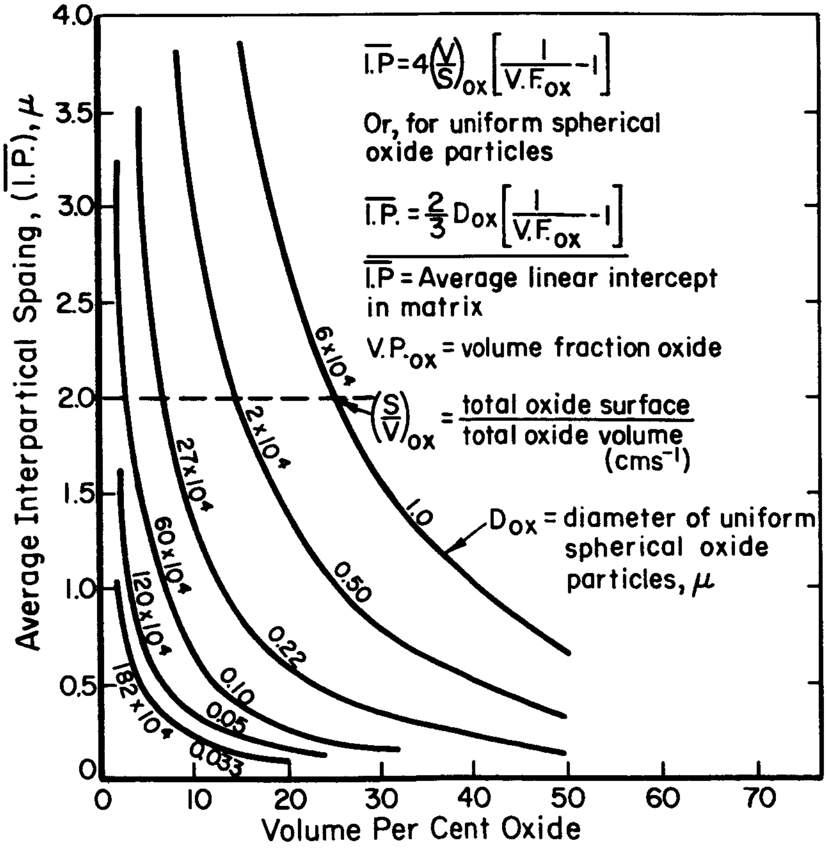


FIGURE 38. CALCULATED AVERAGE INTERPARTICLE SPACING VERSUS OXIDE CONTENT (REF.223)

There are several ways of producing dispersed phases in metals. These include blending of fine nonmetals with fine metal powders, internal oxidation in solids, and reduction of oxide solid solutions or of mixed oxides. A comparison of some of these methods has been made by Smith (Ref. 229) who has summarized fabrication procedures as follows:

> "Having obtained a fine, oxide/metal powder mixture this must then be pressed and sintered. The changes occurring here are not the same for all preparation techniques. For example, in compacting and sintering internally oxidized powder particles no sintering is required of metal to oxide, since the oxide particles have already been precipitated in situ in the metal. On the other hand, with oxide/metal mixtures, sintering of oxide to metal and possibly interaction between the two must take place, if residual porosity, existing on a microscale in the particle/matrix interface, is to be avoided. With powders prepared by the oxide solid-solution technique it is not clear what conditions exist initially at the particle/matrix interface. Interfacial energies will be of importance in the sintering behaviour, and the results of Tikkanen, Rosell, and Wiberg are of much interest in this context.
>
> "A survey of the sequence of the operations employed in producing dispersion-hardened alloys indicates that in most cases a single pressing and sintering operation does not give sufficient density and strength in the final structure. This was soon found to be the case with SAP, and it can be readily understood that the tenacious oxide film on the surface of the particles acts as an efficient barrier to sintering, even allowing for the partial disruption of these films by the pressing operation.
>
> "Even with metal/oxide mixtures, the general conclusion appears to be that less densification of a pressed compact takes place as a result of pressing and sintering than would occur with the pure metals. Thus Zwilsky and Grant working with copper/silica and copper/alumina mixtures found only 2-4% shrinkage of compacts even with a sintering temperature of 1000°C, and the final density was only 90% of the theoretical. Sintering must therefore usually be followed by, or combined with, a working operation.
>
> "There may, however, be advantages in giving the powder mixture an initial high-temperature sinter. For example, Bonis and Grant investigating nickel-alumina alloys pointed out that prior sintering could be advantageous since it could help to

'fix' the position of the dispersed phase, so that 'stringering' of the oxide would be less likely during subsequent working. Also, the sintering ensures that the oxide in the alloy is in the stable high-temperature form, if the oxide is one in which allotropic modifications are possible. This may be important since allotropic changes occurring in the dispersed phase during service may have a deleterious effect upon the properties.

"The working operation used for further densification is most commonly hot extrusion, and in this way near theoretical densities can be achieved, although small amounts of residual porosity are probably present. The amount of this porosity usually increases with increasing percentage of the dispersed phase and there are indications that above 10% dispersed phase it is increasingly difficult to achieve high final densities."

Reference 233 summarizes a patent covering Du Pont's TD nickel production.

Tables XXXVIII, XXXIX, and XL summarize physical and mechanical properties for TD nickel and compare them with those of other alloys.

For further details on production, references in Table XXXVII should be referred to.

Figure 39 shows a first-stage turbine blade fabricated from TD-nickel sheet (Ref. 234). The airfoil contour was formed at room temperature by conventional sheet-metal techniques. The trailing edge was joined by resistance-diffusion bonding and back filled with 60Pd-40Ni braze alloy. The TD-nickel platforms were attached by brazing with the 60Pd-40Ni alloy. This type of vane is now under test.

TABLE XXXVIII. PHYSICAL PROPERTIES OF TD NICKEL AND OTHER NICKEL-BASE ALLOYS (REF. 219)

		Properties of Indicated Alloys			
	Temp, F	TD Nickel	Nickel 200	Hastelloy X	MAR-M320
Density, lb/in.^{3}	70	0.322	0.321	0.297 (72 F)	0.333
Melting temp, F		2650	2615	2350	2425
Thermal conductivity Btu/ft^2/in/hr/F) (extrapolated)	70	600	390	62.8	130
	500	380	372	94.4	144
	1100	300	--	143.5	154
	1500	320	--	173.5	158
	1700	340	--	188.5	164
Electrical resistivity (microhm-cm)	70	7.6	9.6	118.3 (72 F)	--
Mean coefficient thermal expansion (10^{-6} in/in/F)	70-1000	8.5	8.5	8.39 (79-1000 F)	7.6
Total normal emittance (heated in air)	1600	0.55	--	--	--
	2000	0.65	0.80	--	--
	2400	0.72	0.90	--	--
Specific heat (calculated) (Btu/lb/F)	70-212	0.107	0.109	0.105	0.12

TABLE XXXIX. MECHANICAL PROPERTIES OF TD NICKEL (REF. 219)

Temp, F	Tensile Strength, psi		Yield Strength, (0.2% Offset), psi		Elongation, per cent		Reduction in Area, per cent
	Bar (1-1/4-In. Diam)	Sheet	Bar (1-1/4-In. Diam)	Sheet	Bar	Sheet	Bar
70	90,000	65,000	80,000	45,000	25	15	80
1800	21,500	18,000	18,000	17,000	8	5-9	15
2000	16,000	14,000	15,000	12,000	6	5-9	12
2400	10,000	9,000	9,000	7,000	4	4-7	10
	Bar (1/2-In Diam)		Bar (1/2-In. Diam)				
2000	26,000		25,000		5		10

TABLE XL. STRESS-RUPTURE STRENGTH OF TD NICKEL AND OTHER NICKEL-BASE ALLOYS (REF. 219)

Temp, F	Material	Stress to Rupture 100 Hr, psi
1800	TD nickel bar (1-1/4-in. diam)	13,000
	TD nickel bar (1/2-in. diam)	18,000
	TD nickel sheet	9,500-14,500
	Haynes R-41	9,000
	Haynes 25	7,000
	Hastelloy X	3,500
	MAR-M302	15,000
2000	TD nickel bar (1-1/4 in. diam)	10,000
	TD nickel (1/2-in. diam)	14,000
	TD nickel sheet	6,500-9,500
	Haynes 25	2,800
	MAR-M302	2,500

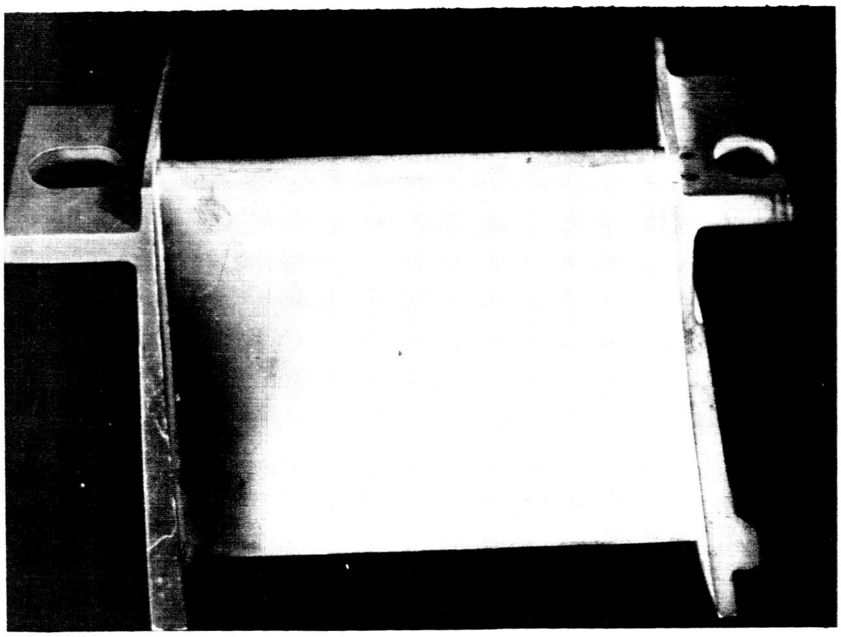


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FIGURE 39. TD-NICKEL VANE FOR JET-ENGINE APPLICATION BEING EVALUATED BY PRATT AND WHITNEY (REF. 234)

STANDARD DEFINITIONS OF TERMS USED IN POWDER METALLURGY*,**,***

Acicular Powder – Needle-shaped particles.

Air Classification – The separation of powder into particle-size fractions by means of an air stream of controlled velocity.

Alloy Powder – A powder all particles of which are composed of the same alloy of two or more metals.

Apparent Density – The weight of a unit volume of powder, usually expressed as grams per cubic centimeter, determined by a specified method.****

Arborescent Powder – See Dendritic Powder.

Atomization – The dispersion of a molten metal into particles by a rapidly moving gas or liquid stream.

Binder – A cementing medium; either a material added to the powder to increase the green strength of the compact, and which is expelled during sintering; or a material (usually of relatively low melting point) added to a powder mixture for the specific purpose of cementing together powder particles that alone would not sinter into a strong body.

Blank – A pressed, presintered or fully sintered compact, usually in the unfinished condition, requiring cutting, machining, or some other operation to give it its final shape.

Blending – The thorough intermingling of powders of the same nominal composition (not to be confused with mixing).

Bridging – The formation of arched cavities in a powder mass.

Briquet – See Compact.

Bulk Density – Synonymous with Apparent Density (U. S.),**** and Loading Weight (British).

Bumping Down – The consolidation of a mass of metal powder by vibration before the pressing operation (British).

Cake – A coalesced mass of unpressed metal powder.

Carbonyl Powder – A metal powder prepared by the thermal decomposition of a metal carbonyl.

Chemical Deposition – The precipitation of one metal from a solution of its salts by the addition of another metal or reagent to the solution.

Classification – Separation of a powder into fractions according to particle size.

---

*Under the standardization procedure of the Society, these definitions are under the jurisdiction of the ASTM Committee B-9 on Metal Powders and Metal Powder Products.

**ASTM Designation: B 243-61. This Standard of the American Society for Testing Materials is issued under the fixed designation B 243; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

***Adopted, 1955; revised, 1958, 1961. Prior to adoption as standard, these definitions were published as tentative from 1949 to 1955.

****See Method of Test for Apparent Density of Metal Powders (ASTM Designation: B 212), p 7.

Coining – The final pressing of a sintered compact to obtain a definite surface configuration (not to be confused with repressing or sizing).

Cold Pressing – The forming of a compact at room temperature.

Cold Welding – Cohesion between two surfaces of metal, generally under the influence of externally applied pressure at room temperature.

Compact – An object produced by the compression of metal powder, generally while confined in a die, with or without the inclusion of nonmetallic constituents. Synonymous with Briquet.

Composite Compact – A metal-powder compact consisting of two or more adhering layers of different metals or alloys with each layer retaining its original identity.

Compound Compact – A metal-powder compact consisting of mixed metals, the particles of which are joined by pressing or sintering or both, with each metal particle retaining substantially its original composition.

Compressibility – A density ratio determined under definite testing conditions.

Compression Ratio – The ratio of the volume of the loose powder to the volume of the compact made from it.*

Continuous Sintering – Presintering, or sintering, in such a manner that the objects are advanced through the furnace at a fixed rate by manual or mechanical means. Synonymous with Stoking.

Core Rod – A member of a die used to produce a hole in a compact.

Cored Bar – A compact of bar shape heated by its own electrical resistance to a temperature high enough to melt its interior.

Cut – See Fraction.

Dendritic Powder – Particles, usually of electrolytic origin, having the typical pine-tree structure. Synonymous with Arborescent Powder.

Density Ratio – The ratio of the determined density of a compact to the absolute density of metal of the same composition, usually expressed as a percentage.

Die – The part or parts making up the confining form in which a powder is pressed. The parts of the die may be some or all of the following: die body, punches, and core rods. Synonymous with Mold.

Die Body – The stationary or fixed part of a die.

Die Insert – A removable liner or part of a die body or punch.

Die Lubricant – A lubricant applied to the walls of the die and punches to facilitate the pressing and ejection of the compact.

Die Set – The parts of a press that hold and locate the die in proper relation to the punches.

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*See Method of Test for Compressibility of Metal Powders (ASTM Designation: B 331), p 67.

Disintegration – The reduction of massive material to powder.

Electrolytic Powder – Powder produced by electrolytic deposition, or by the pulverization of an electrodeposit.

Exudation – The action by which all or a portion of the low-melting constituent of a compact is forced to the surface during sintering. Synonymous with Sweating.

Fines – The portion of a powder composed of particles that are smaller than a specified size, currently less than 44 microns. See also Superfines.

Flake Powder – Flat or scalelike particles whose thickness is small compared with the other dimensions.

Flow Rate – The time required for a powder sample of standard weight to flow through an orifice in a standard instrument according to a specified procedure.*

Fraction – That portion of a powder sample that lies between two stated particle sizes. Synonymous with Cut.

Fritting – Sintering in the presence of a liquid phase. (A term seldom used in the United States.)

Gas Classification – The separation of powder into particle-size fractions by means of a gas stream of controlled velocity.

Graining (British) – See Granulation.

Granular Powder – Particles having approximately equidimensional nonspherical shapes.

Granulation – The production of coarse metal particles by pouring the molten metal through a screen into water, or by violent agitation of the molten metal while solidifying.

Green – Unsintered (not sintered); for example: green compact, green density, green strength.

Growth – An increase in dimensions of a compact which may occur during sintering. (Converse of shrinkage.)

Hot Pressing – The simultaneous heating and molding of a compact.

Hydrogen Loss – The loss in weight of metal powder or of a compact caused by heating a representative sample for a specified time and temperature in a purified hydrogen atmosphere – broadly a measure of the oxygen content of the sample, when applied to materials containing only such oxides as are reducible with hydrogen and no hydride forming element.

Impregnation – A process of filling the pores of a sintered compact, usually with a lubricant; also a process of embedding particles of a nonmetallic substance in a powder-metal matrix, as in diamond-impregnated tools; also a process of filling the pores of a sintered, or unsintered, compact with a metal or alloy of lower melting point.

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*See Method of Test for Flow Rate of Metal Powders (ASTM Designation: B 213), p 9.

Permeability – A property measured as the rate of passage of a liquid or gas through a compact; measured under specified condition.

Plates – Flat particles of metal powder having considerable thickness.

Plus Sieve – The portion of a powder sample retained on a standard sieve of specified number (See Minus Sieve.)

Pore-Forming Material – A substance included in a powder mixture that volatilizes during sintering and thereby produces a desired kind and degree of porosity in the finished compact.

Powder – An aggregate of discrete particles that are usually within the size range 1 to 1000 microns.

Powder-Flow Meter – An instrument for measuring the rate of flow of a powder according to a specified procedure.[*]

Powder Lubricant – An agent mixed with or incorporated in a powder to facilitate the pressing and ejecting of the compact.

Powder Metallurgy – The arts of producing metal powders and of the utilization of metal powders for the production of massive materials and shaped objects.

Preforming – The initial pressing of a metal powder to form a compact that is subjected to a subsequent pressing operation other than coining or sizing. Also, the preliminary shaping of a refractory-metal compact after presintering and before the final sintering.

Presintering – The heating of a compact at a temperature below the normal final sintering temperature, usually to increase the ease of handling or shaping the compact, or to remove a lubricant or binder prior to sintering.

Pressed Bar – A compact in the form of a bar; a green compact.

Pressed Density – The weight per unit volume of an unsintered compact. Synonymous with Green Density.

Pressing Crack – See Slip Crack.

Puffed Compact – A compact expanded by internal gas pressure.

Pulverization – The reduction of metal powder by mechanical means; a specific type of disintegration.

Punch – Part of a die assembly that is used to transmit pressure to transmit pressure to the powder in the die cavity.

Radial Crushing Strength – The relative capacity of a plain sleeve specimen of sintered metal to resist fracture induced by a load applied between flat parallel plates in a direction perpendicular to the axis of the specimen.[**]

Rate-of-Oil-Flow – The rate at which a specified oil will pass through a sintered porous compact under specified test conditions.

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[*] See Method of Test for Flow Rate of Metal Powders (ASTM Designation: B 213), p 9.

[**] See also Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (ASTM Designation: B 202), p 1.

Infiltration – A process of filling the pores of a sintered, or unsintered, compact with a metal or alloy of lower melting point.

Intercommunicating Porosity – That type of porosity in a sintered compact in which the pores are connected so that a fluid may pass from one to the other completely through the compact.

Irregular Powder – Particles lacking symmetry.

K-Factor – The strength constant in the formula for Radial Crushing Strength of a plain sleeve specimen of sintered metal. See also Radial Crushing Strength.*

Loading – The filling of the die cavity with powder.

Lower Punch – The lower member of a die that forms the bottom of the die cavity. It may or may not move in relation to the die.

Lubricating – Mixing with, or incorporating in, a powder, some agent to facilitate pressing and ejecting the compact from the die body; applying a lubricant to the die walls and punch surfaces.

Matrix Metal – The continuous phase of a polyphase alloy or mechanical mixture; the physically continuous metallic constituent in which separate particles of another constituent are embedded.

Milling – The mechanical treatment of metal powder, or metal-powder mixtures, as in a ball mill, to alter the size or shape of the individual particles, or to coat one component of the mixture with another.

Minus Sieve – The portion of a powder sample which passes through a standard sieve of specified number. (See Plus Sieve.)

Mixing – The thorough intermingling of powders of two or more materials.

Mold – See Die.

Molding – The pressing of powder to form a compact.

Needles – Elongated rodlike particles.

Nodular Powder – Irregular particles having knotted, rounded, or similar shapes.

Oversize Powder – Particles coarser than the maximum permitted by a given particle size specification.

Packing Material – Any material in which compacts are embedded during the presintering or sintering operation.

Particle Size – The controlling lineal dimension of an individual particle as determined by analysis with sieves or other suitable means.

Particle-Size Distribution – The percentage by weight, or by number, of each fraction into which a powder sample has been classified with respect to sieve number or microns. (Preferred usage: "particle size distribution by weight" or "particle size distribution by frequency.")

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*See also Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (ASTM Designation: B 202), p 1.

Repressing – The application of pressure to a previously pressed and sintered compact, usually for the purpose of improving some physical property.

Roll Compacting – The progressive compacting of metal powders by the use of a rolling mill.

Rolled Compact – A compact made by passing metal powder continuously through a rolling mill so as to form relatively long sheets of pressed material.

Rotary Press – A machine fitted with a rotating table carrying multiple dies in which a material is pressed.

Screen Analysis – See Sieve Analysis.

Segment Die – A die made of parts that can be separated for the ready removal of the compact. Synonymous with Split Die.

Shrinkage – A decrease in dimensions of a compact that may occur during sintering. (Converse of growth.)

Sieve Analysis – Particle-size distribution; usually expressed as the weight percentage retained upon each of a series of standard sieves of decreasing size and the percentage passed by the sieve of finest size.

Sieve Classification – The separation of powder into particle size ranges by the use of a series of graded sieves.

Sieve Fraction – That portion of a powder sample that passes through a standard sieve of specified number and is retained by some finer sieve of specified number.

Sintering – The bonding of adjacent surfaces of particles in a mass of powder, or a compact, by heating.

Sinterings – A product made by sintering metal powders. Synonymous with Powder Metallurgy Parts, and Sintered Metal Powder Parts.

Sizing – A final pressing of a sintered compact to secure desired size.

Slip Crack – A rupture in the pressed compact caused by the mass slippage of a part of the compact. Synonymous with Pressing Crack.

Specific Surface – The surface area of one gram of powder, usually expressed in square centimeters.

Spherical Powder – Globular shaped particles.

Split Die – See Segment Die.

Spongy – A porous condition in metal-powder particles usually observed in reduced oxides.

Stoking – See Continuous Sintering.

Stripper Punch – A punch that, in addition to forming the top or bottom of the die cavity, later moves further into the die to eject the compact.

Subsieve Fraction – Particles all of which will pass through a 44-micron (No. 325) standard sieve.

Superfines – The portion of a powder composed of particles that are smaller than a specified size, currently less than 10 microns.

Sweating – See Exudation.

Tap Density – The apparent density of a powder obtained when the receptacle is tapped or vibrated during loading under specified conditions.

Upper Punch – The member of a die assembly that moves downward into the die cavity to transmit pressure to the powder contained in the die cavity.

Warpage – Distortion that may occur in a compact during sintering.

ELECTROFORMING

Electroforming can provide structural shapes of complex design with excellent dimensional accuracy and smooth surfaces. It has been especially advantageous for fabricating up to a few hundred identical or nearly identical parts, particularly for the aircraft industry, because tooling costs, inevitably high for stamping, drawing, and other fabricating operations, are usually much lower for electroforming. However, several examples can be cited in which several thousand identical parts have been produced by electroforming in preference to other methods of fabrication not only because costs were lower, but also because structural characteristics were improved. Also, dimensional and physical changes in items subject to frequent design or dimensional changes sometimes can be made quickly and inexpensively by modifying the contour or dimensions of the mandrels employed for electroforming. These advantages have recently focused the attention of design engineers in the aircraft industry on electroforming in preference to other fabricating procedures.

Because of its high strength and relative low cost, nickel is the most popular metal for electroforming, but copper and iron also are used for electroforming many products. Bronze, silver, or gold are sometimes laminated with nickel or nickel alloys, or used individually for fabricating special, unusual structures.

ELECTROFORMING PROCEDURES

Electroforming is defined as the production or reproduction of articles by electrodeposition upon a mandrel or mold that is subsequently separated from the deposit (Ref. 235). Typical fabrication cycles include: (a) the design and construction of mandrels with contours compatible with the electrodeposition process, (b) cleaning conductive mandrels or metallizing nonconducting surfaces,

(c) electrodeposition, (d) rinsing, (e) increasing wall thickness by metal spraying or casting, (f) trimming to cut off edge growths, (g) machining or grinding outer surfaces, and (h) removing the mandrel by pulling, prying, melting, or dissolving. Steps (e), (f), and (g) are not required for some shapes. The above steps replace rolling, shearing, stamping, drawing, spinning, swaging, welding, and/or brazing for fabricating articles with a wall thickness of 0.003 to 0.1 inch. For fabricating thicker walled structures up to 3/4 inch, electroforming has competed successfully with forging, and with rolling and machining.

Hard-surfaced mandrels recycled repeatedly are known as permanent mandrels, useful for cylindrical, hemispherical, and other shapes with no undercuts or reentrant angles. Permanent mandrels with fusible coatings of tin or graphite-wax mixtures can be used for parts requiring no draft or having undercuts up to 0.0005 inch deep. The part is removed by pulling it away from the mandrel while the coating is melted; the mandrel must be resurfaced after each cycle.

Destructible mandrels must be used for complex shapes having large undercuts or concave sections. Materials for destructible mandrels include both lacquered plaster (broken out mechanically), plastic and low-melting lead alloys (melted out), and die-casting alloys of aluminum or zinc (dissolved chemically). Hollow vanes like those in Figure 40 can be electroformed on fusible, low-melting alloys. Parts made on destructible mandrels are usually the most expensive.

Nonmetal mandrels must be metallized to make their surfaces conductive. Silver deposited by chemical reduction has replaced graphite as the surface-conducting film employed in the electrotyping industry, but graphite and graphite-wax mixtures are still used for small lots of parts with complex shapes. Electroless copper or nickel have been useful for metallizing nonconductors. Silver has been deposited by the vacuum process for metallizing glass mandrels employed for electroforming small, experimental solar concentrators.

Many materials have been employed for permanent or destructible mandrels and for metallizing the nonconductors. Some mandrel materials appropriate for specific shapes are given in Table XLI. Other materials are described in several literature references (Refs. 236-242).

TABLE XLI. MANDREL MATERIALS APPROPRIATE FOR SPECIFIC SHAPES

Shape of Electroformed Article	Volume of Production, units/year	Wall Thickness, in.	Finish, Maximum Rms, in.	Dimensional Tolerances, in.	Suggested Mandrel System
Foil	Continuous foil	0.005-0.002	1	0.002	Chromium-plated steel drum
Flat shape, reproducing detail with high fidelity on one side	Several thousand	0.005-0.1	5	0.002	Silvered plastic, separated mechanically
Enclosed hollow shells with a symmetrical cross-section	Several thousand	0.005-0.1	5	0.001	Tin-plated steel, separated mechanically after melting the tin
Enclosed hollow shells as above	Several thousand	0.01-0.1	2	0.0005	Chromium-plated steel removed mechanically by impacting shell against shoulder of a die
Nonsymmetrical, branched shells	Less than 100	0.02-0.1	4	0.003	Silvered plastic
Nonsymmetrical, branched shells	More than 100	0.02-0.1	3	0.002	Tin-plated, closely fitted steel segments drawable individually
Corrugated shells	1 to 1000	0.02-0.1	10	0.005	Low-melting lead alloy
Complex cavities (split molds, dies)	1 to 100	0.02-0.1	5	0.005	Silvered "high melting" wax or plastic, or lacquered plaster
Complex cavities	Several thousand	0.02-0.1	5	0.005	Zinc-alloy die castings
Interconnected multitubular structure	1 to 1000	0.02-0.05	10	0.010	Low-melting lead alloy
Contoured screen	More than 100	0.001-0.010	3	<0.001	Silvered, silicone rubber

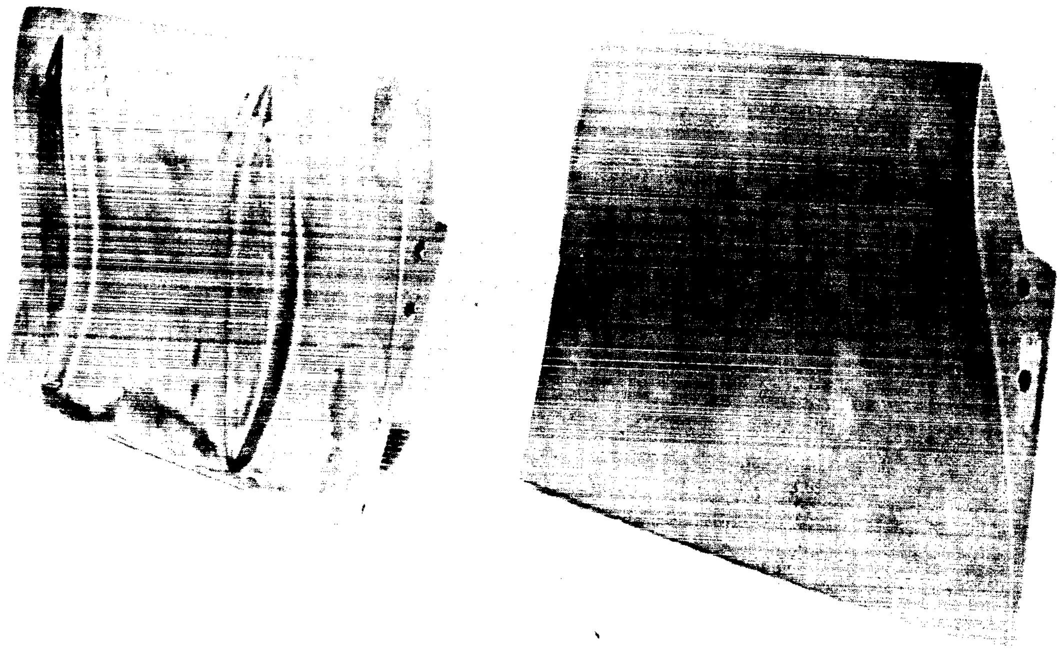

FIGURE 40. HOLLOW VANES WITH A 0.05-INCH WALL THICKNESS ELECTROFORMED WITH FUSIBLE, LOW-MELTING-ALLOY MANDRELS

Courtesy of General Electric Company.

CAPABILITIES AND LIMITATIONS

The largest articles produced by electroforming have included 21-foot-long shields, 16-foot-long tunnel nozzles, 11-foot-diameter parabolic panels, fuel bulkheads, cones, jet nozzles, solar concentrators, and tubing. Figure 41 shows one of the tunnel nozzles. Descriptions of large and small structures are given in a 1964 RSIC report (Ref. 243), and in a recent publication reviewing the state of the art of electroforming (Ref. 244). Perhaps the smallest nickel articles produced by electroforming were 0.00025-inch-thick by 0.003-inch-wide filaments (Ref. 245). A small (8 inch long) wave guide is shown in Figure 42. Electrodes for electric-discharge machining may be made by electroforming (Ref. 246).

A dimensional accuracy of 0.002 to 0.005 inch, overall, is relatively easy to obtain for small or large parts, but a greater tolerance (up to perhaps 0.010 inch) is sometimes necessary for some dimensions of very complex shapes that require the use of expendable mandrels made of wax, plaster, low-melting alloys, or flexible

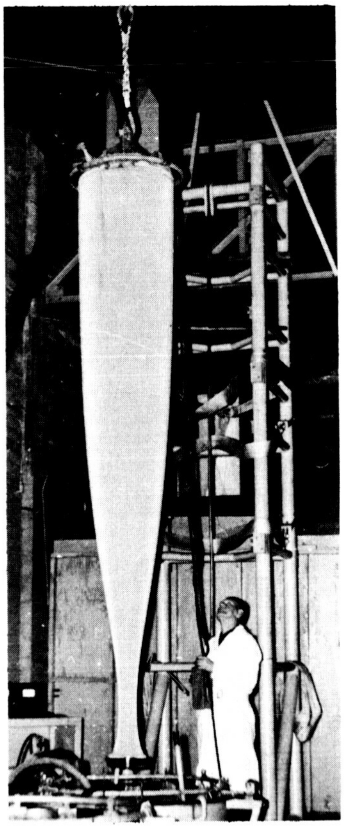

FIGURE 41. ELECTROFORMED NICKEL LINER FOR HYPERSONIC WIND-TUNNEL NOZZLE

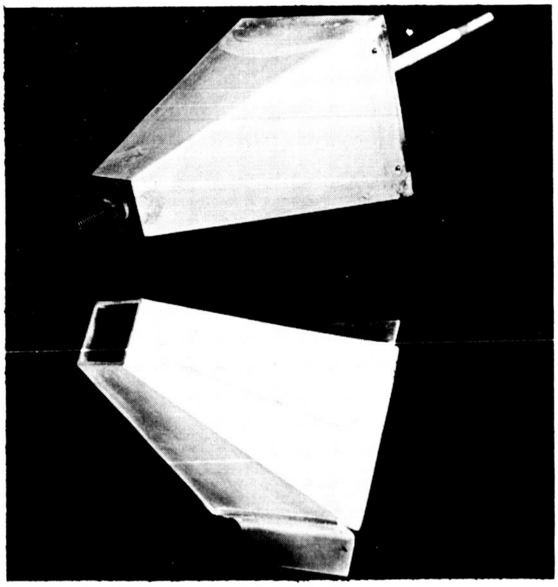

FIGURE 42. NICKEL WAVE-GUIDE WITH PREFORMED GROWN-IN FLANGE

Courtesy of General Dynamics Corporation.

rubber. As a rule, a 0.001 or 0.002-inch tolerance is maintained with expendable plastic mandrels or permanent, reusable metal mandrels. Distortion beyond these tolerances has been encountered in the past when the internal stress in the electroformed metal was not controlled at a low level (below about 5000 psi). Organizations engaged in electroforming are now aware of the importance of stress control to avoid such distortion.

A surface finish of 8 to 16 rms microinches has been fairly common for the mandrel side of electroformed shapes. A better finish (2 rms microinches, for example) has been obtained for some objects when the identical finish was applied (at a premium cost) on the mandrel surfaces.

For thin-walled objects, the exterior surface finish is usually the same, or nearly the same, as the mandrel side. A 16 or 32-rms-microinch finish is practical for any shape with a wall thickness of 0.1 inch, or less, by maintaining good filtration, anode diaphragmming, and agitating facilities. Particle roughness that is difficult to avoid with a wall thickness of 1/4 inch or more must be removed by polishing, grinding, or machining. Procedures for machining and grinding electroformed nickel have been examined (Ref. 247).

Limitations in contour and shape sometimes require modifications in the designs that engineers are accustomed to create for structures produced by other fabricating procedures (Ref. 243). These modifications will not always be major, nor will they have a harmful influence on performance. Design changes suggested or requested by personnel with electroforming experience can usually improve performance and/or reduce costs.

Limitations in electrodeposition procedures ordinarily result in nonuniform wall thickness at different areas on complex shapes. Deep and narrow recesses in mandrel surfaces receive much less current and less thickness of metal than the surfaces projecting closer to the anodes. With anodes that conform, or partly conform, to the shape of the mandrel, wall-thickness variations are restricted to between 5 and 20 per cent, where variations of 100 per cent or more would occur with ordinary anodes. Conforming anodes are particularly appropriate for articles with large, recessed areas, and can sometimes be used without excessive cost.

Electrical-current shields placed over corners and edges reduce the wall thickness at those places and increase the thickness in holes and recessed areas, thereby improving thickness uniformity. Good uniformity of wall thickness is possible with plastic or plastic-coated steel shields integrated with the electroforming fixture. Figure 43 shows how a wave guide is produced with a uniform wall thickness. It is understood that nickel-thickness variations on the bellows' convolutions (1/8 inch deep by 3/32 inch wide) of expandable joints is restricted to only 15 per cent by employing shields.

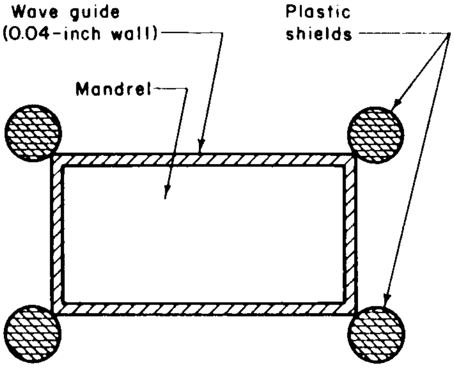

FIGURE 43. SECTION OF WAVE GUIDE SHOWING POSITION OF PLASTIC SHIELDS TO REDUCE THICKNESS AT EDGES AND IMPROVE THE WALL-THICKNESS UNIFORMITY

Although specially shaped anodes can be inserted in the recesses to compensate, at least in part, for the unequal current distribution, recessed areas narrower than about 3/4 inch would require complex and costly anode fixturing that would greatly limit the practicability of the process. The depth of a recess with a width less than 3/4 inch should be not more than 30 or 40 per cent of the width if economical factors do not justify complex and costly fixturing.

The limiting depth-to-width ratio for a narrow recess depends on the radii specified for angles between planes. A generous radius is recommended wherever it is acceptable on a performance basis. Inside right angles with a radius of less than about 0.03 inch are not practical, because the metal that is formed in such angles is frequently structurally unsound (Refs. 248-255). Acute angles with sharp corners result in voids in electroformed metals with a columnar grain structure (Ref. 256). Specially shaped anodes do not overcome these difficulties, but grow-ins, or preformed inserts, can usually be employed, at a premium in cost, to accommodate sharp right angles or acute angles, if they are necessary.

Many of the limitations that were at first imposed on designers of electroformed articles were later overcome by the use of preformed inserts that are integrated with the electroformed object. In some instances, the inserts have been fabricated separately by an electroforming procedure before being attached to the mandrel prepared for electroforming the structural unit. In other cases, inserts have been machined from bar stock. The bond that can be obtained between nickel inserts and electroformed nickel structures is equally as strong or stronger than the nickel. Any bond failures encountered in practice can be attributed to an improper selection of cleaning and activating procedures. In addition to nickel, steel, brass, carbon, and even quartz have been used as materials for making preformed inserts.

Figure 44 shows a wave guide transitional element with grown-on, preformed flanges. Preformed connectors grown-on to an electroformed nickel panel are shown in Figure 45. The sketches in Figure 46 show other examples of preformed inserts.

Inserts must be tightly fitted against the mandrel and aligned carefully. If a gap of more than 0.001 inch exists between insert and mandrel, or if the insert and mandrel are aligned on different planes, the strength of the electroformed article will be reduced there, and cleavage might occur during the final separation step. Gaps of the order of 0.0005 inch or less are strongly bridged with a wall thickness of 0.015 inch or more. Preformed flanges and segmented mandrels must be as closely fitted as inserts.

ELECTROFORMING SOLUTIONS

Nickel ordinarily is electroformed in a Watts-type solution or a nickel sulfamate bath. Watts-type solutions contain, as a rule, 300 to 450 g/l of nickel sulfate ($NiSO_4 \cdot 7H_2O$), 30 to 50 g/l of nickel chloride ($NiCl_2 \cdot 6H_2O$), 40 to 55 g/l of boric acid and a wetting agent such as sodium lauryl sulfate to reduce the surface tension to less than 40 dyne/cm and prevent pitting. The pH of all nickel solutions should be maintained within a relatively narrow range of about 0.5 unit for good control of physical and mechanical properties, but a relatively low pH of about 3 is preferred for some applications, whereas a pH of 4 to 4.5 is preferred for others.

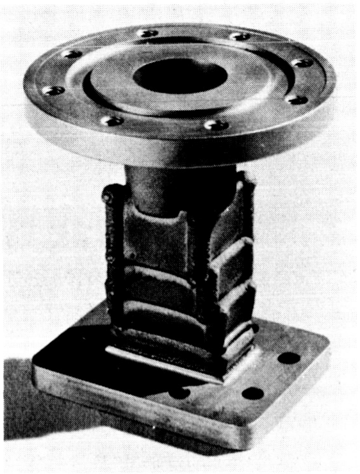

FIGURE 44. NICKEL-WAVE-GUIDE TRANSITION WITH GROWN-ON, PREFORMED FLANGES

Courtesy of GAR Precision Parts, Inc.

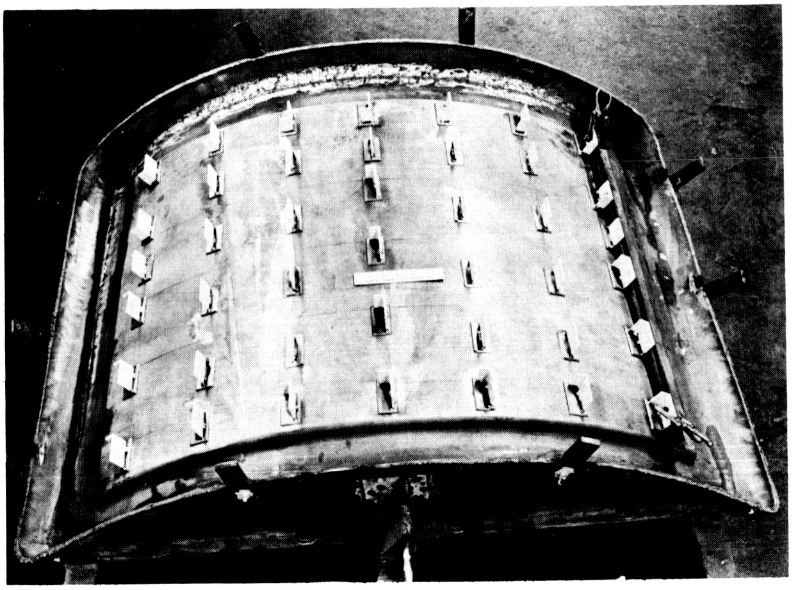

FIGURE 45. PREFORMED CONNECTORS GROWN ON ELECTROFORMED NICKEL PANEL

Courtesy of General Dynamics Corporation.

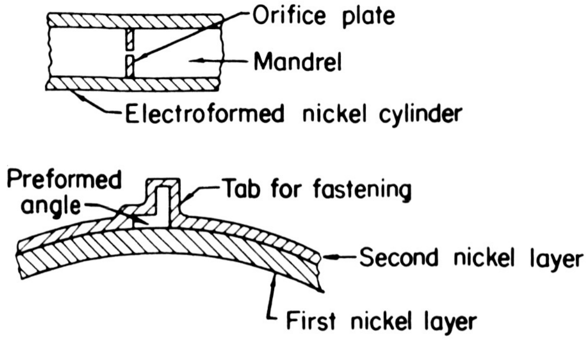

FIGURE 46. SECTIONS OF ELECTROFORMED STRUCTURES WITH PREFORMED, GROW-IN INSERTS (REF. 243)

The principal constituent of the sulfamate bath is nickel sulfamate, 400 to 600 g/l. This solution also contains 30 to 50 g/l of boric acid and a wetting agent, and sometimes contains a stress-reducing agent. A small concentration (5 to 10 g/l) of nickel chloride is sometimes added to insure proper anode dissolution when electroforming must be carried out with a nickel anode current density greater than about 10 amp/ft^2. The chloride induces stress, however, in the electroformed nickel.

Temperature ranges for electroforming solutions are between 110 and 140 F. Cathode-current densities from 20 to 60 amp/ft^2 are employed for complex shapes that are costly or impractical to agitate, but a higher current density is sometimes adopted for simple shapes that can be rotated rapidly.

Rates for electroforming usually range from 0.002 to 0.005 in./hr, except for relatively simple shapes that can be formed at 0.01 to 0.02 in./hr with higher than normal current densities. For thin-walled structures, less than about 0.04 inch, a rate of 0.003 to 0.005 in./hr is fast enough to complete the electrodeposition process in a normal working day. A new electrodeposition solution including sulfamate and fluoborate ions, which was used for electroforming at 0.01 to 0.02 in./hr, was announced recently (Ref. 257). A new, high-concentration nickel sulfamate bath also was described recently for electroforming at 0.01 to 0.02 in./hr (Ref. 258).

EQUIPMENT AND FACILITIES

Electroforming facilities include the following equipment:

Cleaning and electrodeposition tanks
Direct-current-power sources
Anodes and bus bars
Filters and pumps
Heat exchangers
Mechanical stirrers and other agitation devices
Conveyors and/or hoists.

Figure 47 shows the electrodeposition tanks and some of the auxiliary equipment especially installed for electroforming heat sinks. Auxiliary equipment is also required, such as tools for machining, casting, or other means for making mandrels, and for trimming or machining outer surfaces of electroformed articles.

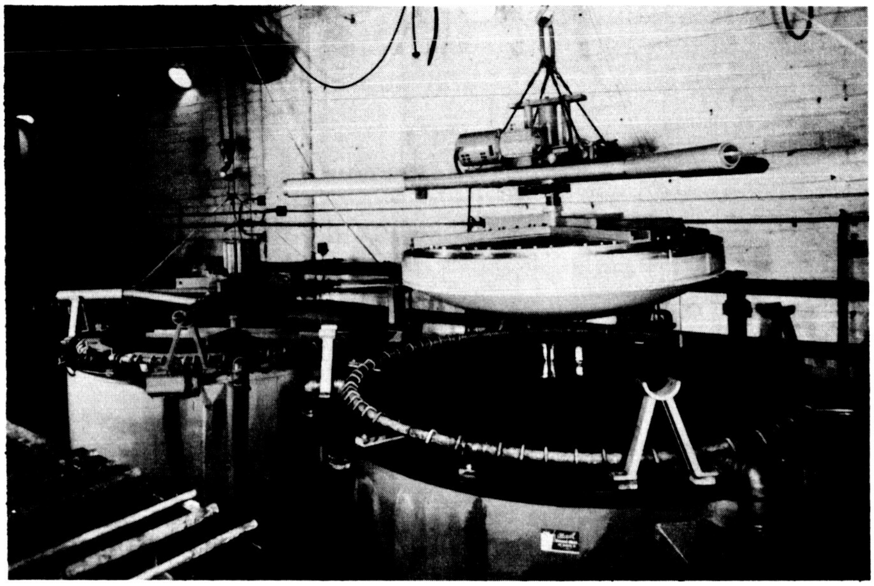

FIGURE 47. ELECTRODEPOSITION TANKS INSTALLED FOR ELECTROFORMING HEAT SINKS

Several companies producing aircraft or jet engines maintain production and/or pilot facilities for electroforming nickel or nickel alloys. About 20 independent organizations commercially produce shaped articles by electroforming, in addition to many others specializing in sound record stampers and electrolytes. Electrodeposition tanks maintained by these companies range in sizes from 2 x 4 x 4 feet to 4 x 12 x 12 feet. The dollar value of equipment in the hands of thirteen of these organizations with electroforming equipment is about $3,500,000 (Ref. 243).

PROPERTIES OF ELECTROFORMED NICKEL

The structure and properties of electroformed nickel (or alloy) depend on electrodeposition conditions such as: kind and concentration of the nickel salts, concentration of chloride or other anion employed for assisting the dissolution of anodes, pH of the solution, impurity concentrations, solution temperature, cathode-current density, kind and amount of solution agitation and, interrelation of these factors. The kind and concentration of organic chemical agents added to the electrodeposition solution for avoiding pitting, reducing stress, or refining the grain structure frequently have profound effects on properties.

Physical and mechanical property ranges for electroformed nickel are as follows (Refs. 257-277):

Density, lb/in.^{3}	0.321 to 0.327
Modulus of elasticity, lb/in.^{2}	23,000,000 to 28,000,000
Electrical resistivity, microohm-cm	7.4 to 10.9
Coefficient of linear expansion (near room temperature), micro-inch/degree F	3.1 to 6.3
Ultimate tensile strength, lb/in.^{2}	55,000 to 215,000
Yield strength, lb/in.^{2}	32,000 to 128,000
Elongation in 2 inches, per cent	2 to 37

Density, strength, and ductility data for nickel electroformed in five solutions are given in Table XLII. These data correspond to nickel with a wall thickness of 0.02 inch or more. The maximum values match or exceed handbook data for cast and rolled or forged, high-purity nickel. Properties differ significantly for thin-walled electroformed nickel (<0.003 inch), however.

As a rule, an increase is observed in the tensile and yield strengths for electroformed nickel, and a decrease in ductility is evident when the temperature of the electrodeposition bath is lowered. Representative data for nickel electroformed in a Watts-type, nickel sulfate-nickel chloride solution confirmed this trend (Ref. 262). Microhardness for the Watts-type nickel increased in direct proportion to the increase in ultimate tensile strength.

Stronger nickel with a higher yield strength is obtained with the nickel chloride and nickel sulfamate solutions than with the other solutions. The sulfamate bath is the most popular, not only because its deposits are strong, but because they exhibit less stress (without organic stress-reducing agents) than those produced in the other

TABLE XLII. PROPERTIES OF ELECTROFORMED NICKEL AT ROOM TEMPERATURE

Electroforming Bath	Density, lb/in.^{3}	Tensile Strength, 10^3 psi	Yield Strength, 10^3 psi	Elongation in 2 Inches, per cent	Reference
Nickel chloride	0.331-0.338	88-124	72.8-9.15	5-21	260-263
Nickel fluoborate	0.314-0.331	55.5-74.5	--	16.6-32	261, 262, 264
Nickel sulfamate	0.323-0.332	85-152	60.8-93.3	3.5-20	259-261, 264-273
Nickel sulfamate with stress reducing agent	0.320-0.327	80.6-215	67.8-128	0.7-11.5	266, 267 269, 274 275
Nickel sulfamate, annealed at 600 F	--	74.2-74.7	51.3-51.6	15-18	274
Nickel sulfamate, annealed at 700 F	--	66.1-68.6	45.8-48.5	19-23.5	274
Nickel sulfamate, annealed at 1000 F	--	63.9-64.5	38.6-38.7	33-34	274
Nickel sulfamate, annealed at 1250 F	--	54.3	16.8	44.2	268
Nickel sulfamate, annealed at 1500 F	--	50	--	--	265
Nickel sulfamate, annealed at 1710 F	--	50	--	40	269
Nickel sulfamate-fluoborate	--	42-101	--	12-18	257, 276
Watts type (nickel sulfate and nickel chloride)	0.327-0.332	51-156	32.4-118	4-37	259-262, 264, 277
Watts type with sulfur-containing stress reducer (disulfonated benzene)	--	170-233	--	2-5	262
Watts type, annealed at 750 F	--	53-62	--	35-39	262
Watts type, annealed at 1850	--	35-39	--	23-37	262

Sulfur contents have been correlated with the tensile strength and notch sensitivity of nickel electroformed in nickel sulfamate solutions with a temperature of 115 to 125 F (Ref. 243). These correlations are summarized in Table XLIII. Data for nickel electroformed in Watts-type (sulfate chloride) solutions containing no stress-reducing agent (Ref. 260), like the data for nickel containing 0.017 per cent sulfur, or less, and electroformed in sulfamate baths, showed high ratios (1.0 or >1.0) of tensile strengths for notched and unnotched specimens.

Data from one source (Ref. 268) for electroformed nickel welds showed strengths of 34,700 and 38,900 psi for TIG welds. Yield strengths were 19,110 and 25,100 psi, and elongations were 5.3 to 9.1 per cent. Higher strengths, up to 68,800 psi but usually 38,400 to 44,700, were developed by another source for nickel-to-nickel butt welds (Refs. 267, 273). These values corresponded to a joint efficiency of about 40 per cent. A higher joint efficiency of about 50 per cent was obtained with stainless steel filler welds. Neither the joint-strength data nor the reported efficiency variations correlated directly with the sulfur or oxygen contents of the electroformed nickel; the available data are too limited to establish the effects of welding-procedure variations.

TABLE XLIII. RELATIONSHIP BETWEEN THE STRENGTH AND NOTCH SENSITIVITY OF ELECTROFORMED NICKEL AND ITS SULFUR CONTENT[(a)]

Sulfur Content, per cent	Ultimate Tensile Strength, psi	Notch-Sensitivity Ratio at about 75 F[(b)]	Notch-Sensitivity Ratio at -423 F[(b)]
<0.005 to 0.005	80,000-150,000	1.0-1.3	0.9-1.3
0.01 to 0.017	160,000-214,000	1.0-1.2	0.9-1.1
0.02 to 0.025	170,000-210,000	0.5-0.7	0.7-1.0

(a) Data for representative, but not all, specimens electroformed with a current density of 30 or 40 amp/ft^2 in solutions maintained at 115 to 125 F. Sulfur contents of 0.01 to 0.025 per cent were induced with organic stress-reducing agents containing sulfur (Ref. 243).

(b) The ratio of tensile strengths for notched (Kt = 6.3) and unnotched specimens.

baths. Nickel electroformed in the chloride bath is highly stressed, which sometimes causes distortion or warpage.

Nickel, with an ultimate tensile strength of about 90,000 to 120,000 psi and a yield strength of 60,000 to 78,000 psi, has been electroformed by several sources in a nickel sulfamate solution containing no organic stress-reducing agent. Elongation in 2 inches for these specimens ranged from about 6 to 14 per cent. Although no stress reducer was added, the stress level probably was relatively low, below 9000 psi in tension, because solution temperatures were in the range of 120 to 135 F.

The thermal stability of nickel electroformed in such a sulfamate solution was better than that for nickel electroformed in chloride or Watts-type baths (Ref. 259). Density changes attributed to the vaporization of water molecules complexed with a small amount of occluded salts amounted to 0.6, 1.2, and 3.5 per cent for nickel electroformed in sulfamate, chloride, and Watts-type solutions, respectively, as a result of heating 2 hours at 1925 F.

Nickel, with an ultimate tensile strength of about 200,000 psi and a yield strength of about 100,000 psi, was formed in 120 or 130 F nickel sulfamate solutions containing a stress-reducing agent (saccharin or trisulfonated naphthylene), that introduce 0.005 to 0.025 per cent sulfur in the deposit. The ultimate tensile strength of nickel electroformed at 140 F was reduced to between about 100,000 and 150,000 psi. These data were reported for specimens electroformed with a cathode-current density of 30 to 40 amp/ft^2.

The lower strength and ductility values reported for nickel may have been a result, at least in part, of codeposited metals (such as copper or zinc) stemming from soluble impurities or occluded particles originating as insoluble materials in the electrodeposition bath. Metallographic examination of electroformed nickel with a tensile strength of 133,000 psi resolved particles causing discontinuities in the crystal structure and weak cleavage planes responsible for a lower than expected strength and ductility (Ref. 275). Thus, soluble and insoluble impurities must be avoided in the electrodeposition bath to electroform high-strength, high-ductility nickel. Soluble impurities can be reduced to tolerable concentrations, as a rule, with electrolytic and/or chemical purification. Good anode bagging and good filtration practice will generally keep insoluble impurities below a harmful amount.

Higher weld-strength values also were reported for nickel to 301 stainless steel joints: 50,700 to 59,800 psi for butt-fusion welds and 59,800 to 72,400 for roll-seam welds, corresponding to joint efficiencies of about 50 per cent and 60 to 90 per cent, respectively. The nickel was electroformed in sulfamate solutions in all cases, and contained 0.01 to 0.025 per cent sulfur in some cases.

Internal stresses in electrodeposited metals can cause warpage and dimensional changes (or cracking) when the electroformed part is separated from the mandrel. A high internal stress reduces the stress that can be applied externally during service. Thus, much effort has been given to development of procedures for eliminating internal stress or keeping it at a low level.

Nickel electroformed in nickel chloride solution is highly stressed. Stress levels usually are between 40,000 and 50,000 psi (Ref. 261). Stress in nickel electroformed in Watts-type solutions usually ranges from about 16,000 to 24,000 psi, but levels up to 60,000 psi have been measured for nickel deposited in high pH solutions (Ref. 261). A lower stress level - about 8,000 psi – can be achieved with good electrolytic purification for removal of iron and other impurities from the solution. Organic additive agents that keep stress at a low level (below 10,000 psi) include saccharin (Ref. 278), cysteine hydrochloride (Refs. 278, 279), sodium benzene disulfonate (Refs. 262, 279), and p-toluene sulfonamide (Ref. 280).

The low level of stress that is customary for nickel electroformed in sulfamate solutions is one of the reasons for the popularity of these electroforming baths; 500 psi is the lowest stress reported for nickel deposited in sulfamate solutions containing no chloride ions or organic stress-reducing agents (Refs. 271, 281-283). This low value was obtained in a 60-ounce bath operated at 140 F with an average current density of 60 amp/ft^2; it has been confirmed by others. A slightly higher stress was observed for nickel electrodeposited at a low-current density (20 amp/ft^2) and a high-current density (100 amp/ft^2). A decrease in the temperature of the electroforming bath, or the addition of chloride ions to improve nickel anode dissolution, causes an increase in stress (Ref. 269). A stress of 5,000 to 10,000 psi is customary for high-strength nickel (>100,000 psi) electroformed in sulfamate chloride solutions containing no stress reducer.

An increase in the temperature of nickel sulfamate solutions reduces stress considerably, and an increase in the concentration of nickel sulfamate from the usual one of about 450 g/l to 600 g/l also reduces stress (Ref. 258). Cationic impurities that greatly increase stress include trivalent and hexavalent chromium, divalent and quadravalent tin, and nitrate ions (Ref. 284). Lead, zinc, iron, phosphate, iodide, ammonium, fluoride, chloride, bromise, sulfate, and sodium ions have a lesser harmful effect (in decreasing order).

Stress in the nickel was described as a major difficulty in electroforming parabolic mirrors with good optical accuracy (Ref. 285). A stress of only 600 psi was excessive. Current density is one of the most critical factors affecting stress, and a new device was developed with improved sensitivity for measuring stress as a function of current density.

A compressive or near-zero stress can be obtained with organic stress reducers in nickel sulfamate solutions. Trisulfonated napthalene is effective for this purpose (Ref. 269). Saccharin, p-toluene sulfonamide, and nickel dibenzene sulfonate also reduce stress (Ref. 286). An automated procedure has been described for regulating stress at a low tensile level with continuous addition of saccharin to a Watts nickel-plating bath (Ref. 287). The stress in nickel-cobalt alloy, which is relatively high without a stress-reducing agent, can be reduced to a maximum of 7, 000 psi in tension by adding saccharin (Ref. 288). Additions of sulfur-containing stress reducers, which are reduced at cathode surfaces to form nickel sulfide, induces notch sensitivity if the sulfur content of the nickel exceeds about 0. 017 per cent, and will reduce ductility if the structure is heated above about 500 F. The material selected for the mandrel also has an influence on stress for very thin-walled optical mirrors (Ref. 289). A large grain size is desirable for the mandrel material.

While measuring stress with a helical-foil device, the nickel thickness usually is in excess of 0. 001 inch. Stress in nickel with a thickness of <0. 001 inch is very much higher than it is in 0. 002-inch nickel, but changes very slightly as thickness is increased to 0. 007 inch (Ref. 242).

Nickel electroformed in Watts-type or nickel sulfamate solutions exhibits a columnar structure, as a rule. The columnar grains grow in a direction perpendicular to the mandrel surfaces. Additions of stress-reducing agents frequently refine the grain structure considerably and eliminate directional structure and properties. Advantage can be taken of effective grain-refining agents that eliminate directional properties, which do not introduce sulfur in the electroformed nickel. Coumarin is an example of such a grain-refining agent.

Electroformed nickel with an ultimate tensile strength of 230,000 psi at 70 F, which was electroformed in a nickel bath containing an organic stress reducer, exhibited a tensile strength of 360,000 psi at -400 F. Property data at -400, 800, and 1200 F for nickel electroformed in other solutions are summarized in Table XLIV.

POROUS NICKEL

Nickel tubing with 0.002 to 0.004-inch-diameter pores perpendicular to the wall of the tubing was produced experimentally by electroforming (Ref. 290). The tubing was permeable to gases and liquids. For a tube with 58 per cent porosity, the permeability was 0.375 lb/min/in.^{2} with a pressure of 50 psi. A nickel tube with 45 per cent porosity passed 0.19 lb/min/in.^{2} at 50 psi pressure.

A higher porosity (up to 80 per cent) was obtained in nickel electrodeposited in similar solutions containing a small concentration of methanol and graphite particles (Ref. 291). High porosity and good coherence were not obtained with other forms of carbon.

NICKEL ALLOYS

Applications for electroformed nickel-cobalt, nickel-iron, nickel-manganese, and nickel-tungsten alloys have been developed, but relatively little property data have been reported. These data are summarized in Table XLV.

Nickel-Cobalt. Tensile-strength values for an alloy containing 1 per cent cobalt were slightly higher than the values for cobalt-free nickel electrodeposited in similar solutions (Ref. 236). A much higher tensile strength (170,000 to 206,000 psi) was observed for alloy containing 40 per cent cobalt, however. Alloy containing about 40 per cent cobalt, balance nickel, was about two times as hard as unalloyed, high-purity nickel when the alloy and the nickel were electroformed in solutions containing no organic-addition agents. Heating at 1100 F softened both materials, but the alloy remained about twice as hard as the nickel.

The highest tensile strength reported for nickel-cobalt alloy was obtained with a cobalt content of about 40 per cent (Refs. 292, 293).

TABLE XLIV. PROPERTIES OF ELECTROFORMED NICKEL AT CRYOGENIC AND HIGH TEMPERATURES (REF. 243)

Electroforming Bath	Ultimate Tensile Strength, 10^3 psi at -400 or -320 F	70 F	800 F	1200 F	Yield Strength, 10^3 psi -400 or -320 F	70 F	800 F	1200 F	Elongation, per cent -400 or -320 F	70 F	800 F	1200 F
Nickel sulfamate with stress reducer, 110-120 F	360	230	--	--	260	130	--	--	<1	2	--	--
Nickel sulfamate with stress reducer, 130-140 F	230	120	--	--	90	75-80	--	--	18	10	24	9
Nickel sulfamate with no stress reducer	101-150	80-120	50-70	25	59-89	45	30	20	33	12	--	--
Watts type, 140 F	80	60	30	25	40	30	25	20	48	30	28	14

TABLE XLV. MECHANICAL PROPERTIES OF ELECTROFORMED NICKEL ALLOYS

Alloy	Ultimate Tensile Strength, 10^3 psi	Yield Strength, 10^3 psi	Elongation, per cent
Nickel, 40 per cent cobalt (Ref. 292)	200 to 220	130 to 160	2
Nickel, 40 per cent cobalt(a)	170 to 206	--	1 to 4
Nickel, 1 per cent cobalt chloride bath (Ref. 264)	102	--	14
Nickel, 1 per cent cobalt sulfate bath (Ref. 264)	82	--	20
Nickel, 2 per cent cobalt, annealed (Ref. 270)	50	--	20
Nickel, 20 to 50 per cent iron (Ref. 293)	Up to 257	--	2 to 5
Nickel, 0.5 per cent manganese (Ref. 293, 294)	167 to 190(b)	140 to 163(b)	5.0 to 6.5(b)
Nickel, 20 per cent tungsten (Ref. 295)(c)	115 to 162	--	Very low, unless heated at 1125 F, or higher

(a) Battelle Memorial Institute unpublished data for alloy deposited in nickel sulfamate solutions containing no stress-reducing agent.

(b) After heat treatment for 16 hours at 800 F. The modulus of elasticity was 29,000,000 psi.

(c) The modulus of elasticity for nickel alloy containing 17 per cent tungsten was 25,000,000 psi. The density of a 22 per cent tungsten alloy was 0.354 lb/in.^{3} and the density of a 17 per cent tungsten alloy was 0.36 lb/in.^{3}

This 200,000 to 220,000 psi strength level was reached with alloy electroformed in sulfamate solutions containing no stress reducing agent. Ductility was improved by heating at 1150 F, so the sulfur content probably was <0.01 per cent. After the heat treatment, the alloy retained a tensile strength of 75,000 to 85,000 psi. Higher tensile strengths from 100,000 to 120,000 psi were obtained after heat treatment at 850 F. Thus, the alloy is a promising material for electroforming components that must be heated during assembly.

Nickel-Manganese. For fabricating airfoils, deflectors, and vanes reaching 500 F during service, nickel alloy containing about 0.5 per cent manganese exhibited attractive properties comparable with those of some stainless steel and rolled nickel alloys (Ref. 294). The tensile strength of the alloy, electroformed in a sulfamate solution containing no stress-reducing agent, ranged from 167,000 to 190,000 psi after the alloy was heated for 16 hours at 800 F. Tensile strengths at elevated temperatures were 130,000 psi at 400 F, 99,000 psi at 600 F, and 56,000 psi at 750 F. Elongation was 5 or 6.5 per cent at temperatures up to 400 F, 18 per cent at 600 F, and 26 per cent at 750 F. The alloy was equally as hard at 600 F as it was at 200 F or room temperature. The 55,000 psi endurance limit reported for the nickel-manganese alloys at 10^7 cycles is about 50 per cent higher than the endurance limit reported previously (Ref. 260) for Watts-type nickel (which ranged from 30,000 to 44,000 psi).

Nickel-Chromium. Effort to codeposit nickel and chromium with a dense structure has been unsuccessful. However, Ni-20Cr alloy has been fabricated experimentally in the forms of sheet and tubing by electrodepositing alternate layers of nickel and chromium, removing the mandrel and diffusing the layers (Ref. 259). The homogenized alloy displayed good resistance to oxidation in air at 1800 F and scaled at a much lower rate than cobalt-tungsten alloy, nickel, or chromium. Its density was 0.29 lb/in.^{3}, which is 99.6 per cent of the theoretical density for the 20 per cent chromium alloy.

Other Alloys. Nickel-iron alloy has been used for electroforming magnetic tape, but no other use of the alloy has been reported. Nickel-tungsten, like cobalt-tungsten, has been examined experimentally, but the brittleness of these alloys has discouraged commercial applications (Ref. 295). They might prove useful as one layer of a laminate or sandwich that includes another layer of a more ductile nickel alloy for applications requiring a hard, wear-resistant surface layer.

Nickel strengthened with submicron particles of alumina has also been fabricated by electroforming. About 2 volume per cent of alumina greatly increased the yield strength of the nickel (Ref. 296).

Nickel alloys of phosphorus (3 to 7 per cent) or tin (35 per cent) can be electrodeposited adherently on metallic substrates, but are considered too brittle for electroforming applications. Electroforming might be feasible with refinements in the electrodeposition processes. Composites (sandwiches) of ductile nickel and nickel-tin alloy have been experimentally electroformed in the shape of tubing.

ECONOMICS

In several articles, electroforming has been judged as a costly process. Yet, other articles include descriptions of parts that were electroformed with cost savings in comparison with former fabrication methods. For example, an $8.10 unit cost for stamping and soldering small nickel chutes was reduced to $0.85, by electroforming (Ref. 297). Air scoops costing $344/unit when fabricated by bending and welding were electroformed in nickel, at first for $239, later for $185, and finally were purchased from an electroforming vendor for $15 (Ref. 266). A $1700 cost for electroforming 20 hollow guide vanes represented a 66 per cent cost reduction when compared with a $5000 estimate for fabrication by bending and welding (Ref. 266). Costs for an air-intake lip were reduced 50 per cent by adopting electroforming (Ref. 298).

In a survey conducted in 1962 on molecular-forming procedures*, typical total costs for electroforming simple shapes were estimated at about $25 per pound of nickel (Ref. 299). The estimate was misleading, because several structures have been electroformed at a much lower figure. Some single prototypes cost a great deal more when tooling costs had to be charged to the production of just one part. Cost data supplied by vendors replying to questionnaires ranged from about $2 to $667 per pound of nickel (Ref. 243). Three vendors reported examples of parts they had produced at <$3 per pound of nickel, and another reported examples in the range of $5 to $9 per pound of nickel, including the tooling cost that was prorated over at least 100 parts in each case.

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*These molecular-forming procedures included fabrication by chemical vapor deposition, which is not so far advanced as electroforming.

CHEMICAL VAPOR DEPOSITION

Chemical vapor deposition (CVD) has been used extensively in the preparation of high-purity metals, in the application of coatings to particles or extended surfaces, and in the production of integrated electronic circuits. The application of CVD to the problem of shaping has been far less extensive.

Chemical vapor deposition is accomplished by causing gaseous molecules to react chemically at, or near, a heated surface under conditions such that one, and only one, of the reaction products is a solid and is deposited on the heated surface. As the reaction continues, atoms (or molecules) are added to the growing solid lattice one at a time. Such a procedure is called a "molecular forming" process. In general, to produce a given shape, the deposition reaction is carried out with a suitable mandrel as the deposition surface. After building up a layer of the desired thickness, the process is stopped and the mandrel is removed by melting, leaching, dissolution, or by other means, thus leaving a free-standing body.

A general description of chemical vapor deposition as well as a detailed discussion of some specific areas of application is given in a report prepared by J. M. Blocher, Jr., and others (Ref. 300). One chapter of a forthcoming publication (Ref. 301) deals with vapor forming and joining. The application of CVD to molecular forming is also discussed in a report prepared for Wright-Patterson Air Force Base (Ref. 302).

Nickel can be deposited by the thermal decomposition of nickel carbonyl, thermal decomposition of nickel acetyl acetonate, or by the hydrogen reduction of nickel chloride. The bulk of experimental study has been applied to the carbonyl process and only that system will be considered here.

The vapor pressure of liquid nickel carbonyl is sufficiently high that the feed supply can be maintained a few degrees below room temperature and the vapor pumped through the reaction chamber. In this way the supply lines and other parts of the system exposed to the vapor can be at room temperature without danger of condensation. The carbonyl is introduced into the reaction chamber at pressures ranging from 1 to 200 torr. The nickel carbonyl vapor contacts the substrate that is heated to 100 to 225 C. Thermal decomposition results in the deposition of nickel on the substrate and release of carbon monoxide as a by-product. Microstructure varies from that of very fine grains, from dendritic to columnar grains. Considerable detail concerning the experimental arrangements and the effect on the deposit of varying the pressure, substrate temperature, flow rate of nickel carbonyl, etc., were given in a series of papers by L. W. Owen (Ref. 303). The kinetics of the deposition reaction and the vapor-phase diffusion limitations have been reported by Carlton, Oxley, and Blocher (Ref. 304).

Chemical vapor deposition forming of nickel shapes has reached the commercial development stage in apparently only one instance, and this has since been discontinued. The Budd Company manufactured molds, forming dies, patterns, and tools by the carbonyl process (Refs. 305-307). Most of the production was for the automotive industry. The steps of the process were as follows:

(1) A master was fabricated of wood or plastic and then sprayed with a eutectic alloy of tin to form a negative mold that was separated from the master.

(2) The negative mold was placed in a deposition chamber that was then sealed and purged with carbon dioxide. The mold was heated to the range 160 to 175 C by strip heaters.

(3) $Ni(CO)_4$ vapor was then passed through the chamber. On contact with the hot mold the molecules decomposed depositing nickel on the surface of the mold. The process was continued until the desired thickness of nickel was obtained.

(4) The deposition chamber was again purged and the coated mold was removed. Separation of the nickel shell destroyed the tin negative mold. The thin nickel shell, an exact positive of the original master, could be used without further treatment, or filled with epoxy for additional support.

The company indicated that the nickel molds so produced were suitable for permanent mold casting of aluminum, gray iron, zinc, and brass.

The process apparently has not been employed for the deposition of nickel alloys. In principle, alloys could be deposited by mixing the nickel carbonyl vapor with a second metal-bearing vapor capable of depositing the second metal at the same temperature as the nickel. This temperature restriction would seem to limit the second compound to a carbonyl. Iron, chromium, molybdenum, and tungsten carbonyls might be considered. The temperature required to obtain optimum deposition from these compounds is generally higher than from nickel carbonyl. The composition of the vapor would have to be adjusted so that the resulting rates of deposition of the two metals at a given temperature would produce the desired composition in the solid phase. Any alloy produced in this way would undoubtedly contain appreciable amounts of carbon.

The commercial application of CVD forming of nickel parts included patterns, dies, tools, and molds. Other possible applications include wave guides, nozzles, ducts, and, in general, complex shapes that are difficult to machine. It is doubtful if CVD forming of pure nickel offers any real advantage over electroforming. However, there is the possibility of codepositing alloys when desired. Elements such as molybdenum and tungsten cannot be electrodeposited from aqueous plating baths, but could be codeposited by CVD. A disadvantage of chemical vapor deposition from the carbonyls is their extreme toxicity. Stringent safety precautions must be employed in their use.

METAL SPRAYING

Although the production of free-standing bodies by spraying of difficult-to-fabricate materials, such as tungsten, has been extensively studied, very little work has been done in the fabrication of free-standing nickel bodies by spraying techniques. The primary reason is that nickel and nickel-alloy parts can be made easier by forging, machining, or casting. There has been considerable work done, however, in spraying of nickel-alloy coatings for abrasion, corrosion, and oxidation resistance. Since the application of sprayed nickel coatings is not covered elsewhere in this series of reports, a discussion of the recent advances in the spraying of nickel-alloy coatings is presented.

Five excellent reviews of metal and ceramic spraying techniques were recently published (Refs. 308-312). These reviews discuss the fundamentals of metallizing*, processing and handling techniques,

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*Metallizing is the process of depositing finely divided particles of metals, intermetallics, or metal oxides in the heated, semimolten condition to form an adherent coating.

properties and quality of sprayed deposits, safety hazards, and common applications of sprayed coatings. The reviews cover other alloys as well as nickel and are highly recommended for the reader interested in obtaining a general background in metallizing. The numerous examples of metallizing problems and their solutions cited should be of interest to all in the metallizing industry.

Because processing techniques are treated thoroughly in the above references, processing details will be largely omitted in the following discussion. However, fundamentals of the various processes used in spraying nickel-alloy coatings, the properties of the coatings, and possible applications of sprayed nickel coatings will be discussed. Also, fused-metallized coatings, nickel aluminide coatings, plasma spraying, and flame plating will be discussed.

FUSED-METALLIZED COATINGS

Fused-metallized coatings are usually applied by either a wire or powder-spray gun and subsequently fused, providing a dense, uniform coating with a metallurgical bond to the base material. The application of the fused-metallized coating generally involves four distinct operations:

(1) Preparation of the surface

(2) Spraying the surface material

(3) Fusing to form a metallurgical bond with the base material

(4) Finishing to the required surface and dimensional tolerances.

The last operation is not required in all instances, since the fused deposit may be sufficiently smooth for applications not classified as machine element parts.

Coating Materials. With the exception of pure-nickel wire, nickel alloys available for application as fused-metallized coatings are mainly hard-surfacing alloys and are normally supplied in powder form, primarily because they are too hard or too brittle to be drawn into wire. The mesh size of the powder is usually 100 to 150. Typical nickel alloys available for fused-metallized coatings are listed in Table XLVI. The Ni-Cr-Si-B and Ni-Si-B alloys have coefficients of expansion similar to those of austenitic steel and have fusing points in the range of 1800 to 2050 F.

TABLE XLVI. TYPICAL NICKEL-BASE MATERIALS AVAILABLE FOR METALLIZED COATINGS (REFS. 308-310)

	Per Cent												Hardness,	Fusion
Type	Ni	Cr	B	Si	Fe	C	Co	Cu	Mo	Al	W	WC	R_C	Temp, F
AMS 4775	65.0	13.0	2.75	3.0	3.0	--	--	--	--	--	--	--	56-61	--
	75.0	20.0	4.75	5.0	5.0	--	--	--	--	--	--	--	56-61	--
Ni-Cr-Si-B	65.0	13.0	2.75	(10 per cent maximum)			--	--	--	--	--	--	56-61	--
	75.0	20.0	4.75											
	71.0	10.0	2.0	(9 per cent maximum)			--	--	--	--	--	--	45-60	--
	81.0	17.0	4.0											
	75.0	8.0	2.0	(8 per cent maximum)			--	--	--	--	--	--	35-40	--
	Bal	5.0	1.25	3.25	2.0	0.3	--	--	--	--	--	--	21	2000
	Bal	26.0	3.5	4.0	1.0	0.95	--	--	--	--	--	--	54	1975
	Bal	11.0	2.5	3.25	3.75	0.55	--	--	--	--	16.0	--	50-55	2050
	73.5	14.0	3.0	4.0	4.5	0.65	--	--	--	--	--	--	56-61	--
	80.0	10.5	2.3	3.5	3.5	0.30	--	--	--	--	--	--	46-51	--
	82.0	9.5	1.8	3.2	3.3	0.25	--	--	--	--	--	--	37-42	--
	70.0	15.0	3.0	4.5	4.5	--	--	--	--	--	--	--	59-60	1870
	77.0	10.0	1.5	4.0	4.0	--	--	--	--	--	--	--	44-52	1920
	85.0	7.5	1.5	4.0	1.5	--	--	--	--	--	--	--	35-42	2010
	82.0	7.0	2.9	4.5	--	--	--	--	--	--	--	--	60	--
	81.0	11.0	2.0	2.0	2.0	0.3	1.5	--	--	--	--	--	38	--
	78.0	11.5	3.0	3.5	3.5	0.4	--	--	--	--	--	--	48	--
	Bal	14.0	3.25	4.0	0.75	--	--	--	--	--	--	--	60	--
	Bal	9.0	2.0	3.0	3.75	0.45	--	--	--	--	--	--	45	--
	Bal	17.0	3.30	3.9	2.0	0.95	--	--	--	--	--	--	61	--
	83.0	10.0	2.5	2.5	2.0	0.10	--	--	--	--	--	--	35	1875
	70.5	17.0	3.5	4.0	4.0	1.0	--	--	--	--	--	--	60-63	1875
	67.0	16.0	4.0	4.0	2.5	0.5	--	3.0	3.0	--	--	--	58-61	1875
	Bal	10.0	2.5	2.5	2.5	0.15	--	--	--	--	--	--	--	--
	Bal	14.0	2.75	3.5	4.0	0.6	--	--	--	--	--	--	50-54	1875
	46.0	11.0	2.5	2.5	2.5	0.5	--	--	--	--	--	35	--	--
Ni-Si-B	Bal	--	1.5	3.5	1.5	0.25	--	--	--	--	--	--	30	1975
	93.0	--	1.9	3.5	--	--	--	--	--	--	--	--	38	2000
	96.0	--	1.5	2.5	--	--	--	--	--	--	--	--	8-12	2050
	93.0	--	1.8	3.0	--	--	--	--	--	--	--	--	18-22	2025
	91.0	--	2.9	4.5	--	--	--	--	--	--	--	--	60	--
	72.0	--	3.4	4.4	0.6	0.14	20.0	--	--	--	--	--	60	--
Ni-Cr	80.0	2.0	--	--	--	--	--	--	--	--	--	--	--	--
Ni-Al	80.0	--	--	--	--	--	--	--	--	20	--	--	--	--

Two important factors to be considered when choosing a coating material are:

(1) The fusion temperature and its effect on the base metal

(2) The coefficient of expansion in relation to that of the base metal where considerable fluctuation will exist in the operating temperature. If coefficients of expansion are not well matched, cracking and failure of the coating may result.

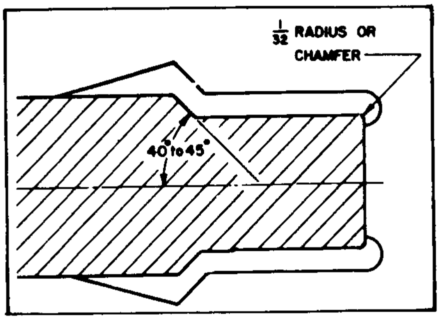
Base Metals. Fused-metallized nickel-alloy coatings have been applied readily to a wide range of steels, cast irons, pearlitic malleable irons, nickel and nickel alloys, copper and copper alloys, and refractory alloys. For specific coating procedures applicable to the base materials mentioned, the manufacturer or equipment supplier should be consulted.

Nickel-alloy coatings may be sprayed on almost any size workpiece. Size may vary from that of bridge structures, which are sprayed in the field, to the head of a pin. Parts as large as 24 inches in diameter, 23 feet in length, and weighing in the range of 2 to 3 tons have been successfully sprayed and fused.

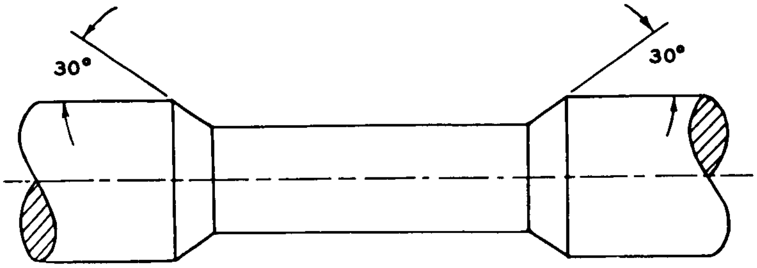
Configuration of the workpiece, however, greatly influences the choice of spraying method or defines the applicability of the process. Flat plates can be sprayed with a back and forth motion to attain uniform and full coverage. Cylindrical parts, that can be rotated in a lathe, are ideal for spraying. The spray gun or guns can be mounted on the carriage of the lathe and transversed at a uniform feed for complete spraying of the surface area. A cam can be sprayed manually. Internal diameters and small holes are, on the other hand, more difficult or impossible to spray. If the internal diameter is large enough, extensions are available for the spray gun, which allows uniform deposition of the coating material. Deep, small-diameter holes are difficult to coat because of overlapping of the sprayed metal.

Surface Preparation. Before the spray coatings are applied, the substrate surface is cleaned and prepared so that the sprayed metal particles will adhere to it. Oil and other contaminants are removed by a suitable solvent. Sand or grit blasting is often used to remove contaminants and to roughen the surface prior to metal spraying. Areas that are not to be coated are masked prior to blasting.

Undercutting of the surface of cylindrical shafts and similar parts is usually necessary to permit a sufficient thickness of sprayed deposits on the finished parts. Typical shaft preparation is illustrated in Figure 48. Sharp corners are to be avoided.



a. Undercut Shoulder and End Chamfer



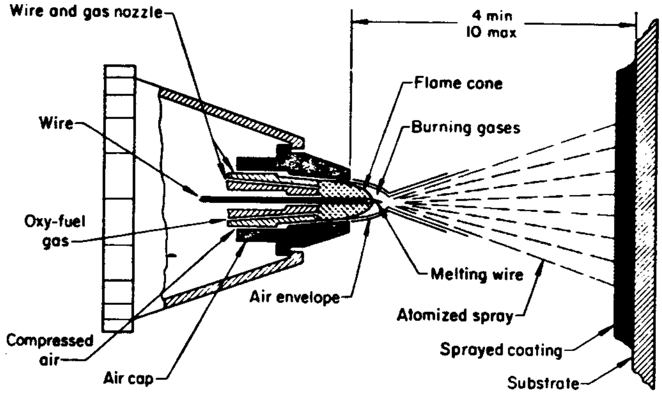
b. Commercial Machine Finish

FIGURE 48. TYPICAL SHAFT PREPARATIONS FOR APPLICATION OF FUSED METALLIZED COATINGS (REFS. 308,310)

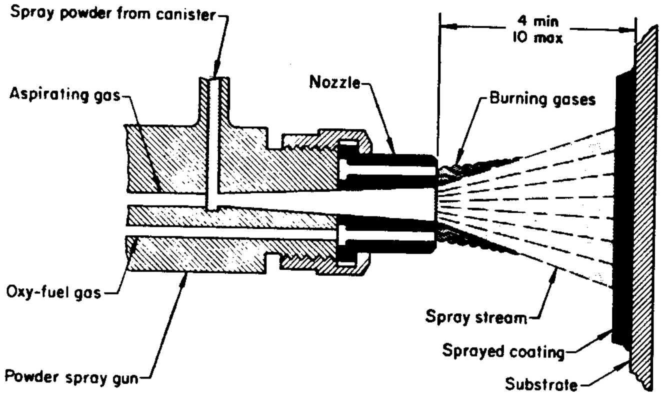
Surfaces of cylindrical parts are prepared by threading or grooving when thick coatings are required, or if the part is subjected to heavy duty service. Sometimes the grooving operation is followed by a thin bonding coat of pure molybdenum applied with a spray gun.

Spraying. Pure nickel can be sprayed with either a wire or powder gun. Cross-sectional drawings of a wire gun and a powder gun are shown in Figure 49. In the wire gun, the wire is drawn through the nozzle by a pair of powered feed rolls. The wire is continually melted in the oxy-fuel-gas flame and is atomized by a blast of compressed air that carries the metal particles to the surface being sprayed. The individual particles mesh at the surface to produce a coating.

Nickel alloys are usually sprayed in a powder gun because they are more readily available in powder form. The powder is fed into



a. Wire Gun



b. Powder Gun

FIGURE 49. CROSS-SECTIONAL DRAWING SHOWING DETAILS OF A WIRE GUN AND A POWDER GUN (REF. 312)

the spray gun by a trigger-actuated vibrator from a reservoir attached directly to the gun. In the gun, the powder is impelled with an aspirating gas, usually air, through an intense heat source and caused to impinge on the workpiece. The molten powder particles flatten out to form thin platelets or lamellae that conform to any irregularities on the surface. The heat source can be any fuel gas (usually acetylene or hydrogen) and oxygen. Figure 50 is a drawing of a typical installation for fused coatings.

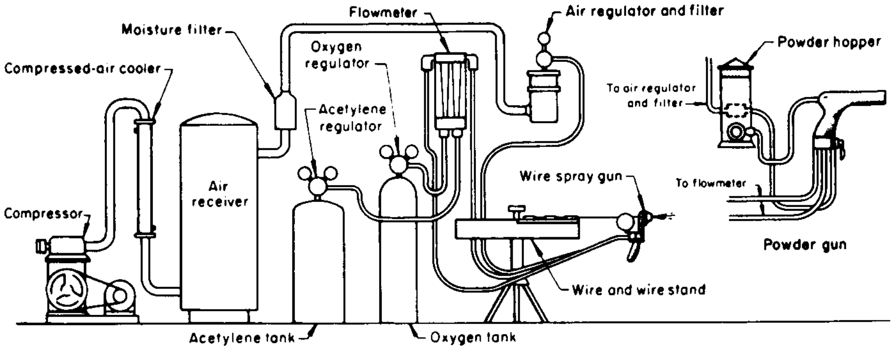


FIGURE 50. SKETCH OF METALLIZING INSTALLATION FOR FUSED COATINGS (REF. 310)

When the surface has been intentionally roughened, these irregularities serve as anchorages for the deposited particles. The particles then serve as anchorages for subsequent particles and, in this manner, an adherent and cohesive deposit is built up. Along with this mechanical bonding there is some point-to-point fusion of particles and some oxide-to-oxide bonding, but the primary bonding mechanism is mechanical.

Fusing the Sprayed Deposit. After spraying, nickel-base alloys can be fused to form a dense, uniform coating that is metallurgically bonded to the workpiece. Fusing should start immediately after completing the spraying operation before the heat developed during spraying is lost.

Fusing of small parts is often done in a controlled-atmosphere furnace. The high cost of equipment, however, and the limitations on the size and shape of parts restrict this method to high-production operations. The most common method of fusing is with an oxyacetylene torch or with oxypropane multiple-hole torches using a neutral or reducing flame, as shown in Figure 51.

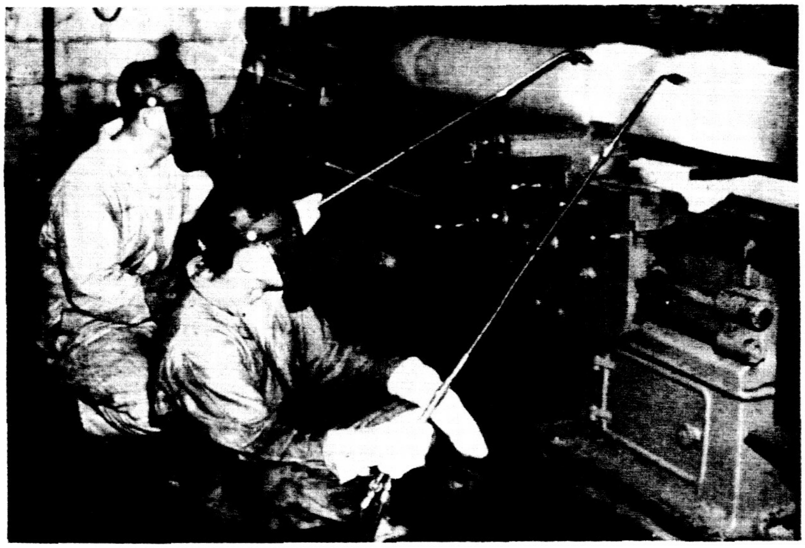


FIGURE 51. FUSING OF A COATING ON A NICHROME ROLL 9 INCHES IN DIAMETER AND 20 FEET LONG (REF. 308)

Finishing the Fused Deposit. Since most of the nickel materials sprayed are applied for hard-surfacing purposes, grinding is usually the most economical method of finishing. Dry grinding may be suitable for certain operations, but the heat generated and rapid-wheel wear make close-tolerance work difficult. Wet grinding with silicon carbide grinding wheels produce close-tolerance parts, fine finishes, and economical stock-removal rates. Where necessary, grinding can be followed by superfinishing that involves a machine-lapping operation under controlled conditions, using abrasive stones as fine as 1000 grit. Finishes as smooth as 2 rms microinches can be obtained and the operation controlled to produce either highly reflective surfaces or frosty mat finishes, as required.

Properties of Nickel and Nickel-Alloy-Fused Coatings. The characteristics of sprayed nickel-alloy deposits chemically resemble the derivative wire or powder, but their physical properties are quite different. The structure is not homogeneous, and cohesion is achieved through mechanical and metallurgical bonding. The tensile strength may be relatively high for the low-melting metals, but the ductility is low in all instances, and all spray deposits are porous to some extent.

The thickness of the coating and the extent of diffusion of the sprayed metal influence the hardness of fused-sprayed coatings.

For an accurate hardness reading, the coating thickness should be approximately ten times the depth of penetration. Typical hardness readings for a few nickel-alloy compositions are given in Table XLVII.

TABLE XLVII. TYPICAL COMPOSITION AND HARDNESS OF NICKEL ALLOYS USED FOR FUSED-SPRAYED COATINGS (REF. 312)

Per Cent									Hardness, Rockwell C
Ni	Cr	W	Mo	Cu	B	Si	Fe	C	
93	--	--	--	--	1.9	3.5	--	--	35-44
82	10	--	--	--	2.5	2.5	2.5	0.2	30-40
76	13	--	--	--	3.0	Si + Fe + C, 9 max			45-50
70	17	--	--	--	3.5	4.0	4	1	56-64
67	16	--	3	3	4.0	4.0	2.5	0.5	59-62
66	13	16	--	--	4.0	Si + Fe + C, 10 max			50-55

Corrosion rates vary widely with differences in temperature, degree of aeration, velocity, and metal-ion concentration of the reagent. Therefore, laboratory data can only serve as a rough guide to actual service conditions. Table XLVIII was compiled from literature and limited tests run in the laboratory.

NICKEL ALUMINIDE COATINGS

There is a need for a self-bonding material that can be sprayed from a metallizing gun and would eliminate the fusion step. Such a self-bonding material is nickel aluminide, which has been described by J. Alan Sheppard (Ref. 313). Nickel aluminide is a nickel-clad aluminum powder that, when sprayed, produces, by means of an exothermic reaction, self-bonding coatings of intermetallic nickel aluminide. These coatings can be nonporous and are resistant to high-temperature (up to 1760 C) in gaseous environments. Some engineering data on nickel aluminide are given in Table XLIX. A picture of the composite powder particle is shown in Figure 52.

With the formation of nickel aluminide, the exothermic reaction causes additional heating of the materials and results in a metallurgical bond with the substrate. Such bases as aluminum, nitrided or hardened steels, niobium, tantalum, titanium, or graphite can be coated without prior machining or shot blasting. It is only necessary to remove the surface film to achieve bond strengths approximately 20 per cent stronger than those available from molybdenum wire (see Table L), and the degree of reliability is much less dependent on operator technique. Coatings of nickel aluminide provide an

TABLE XLVIII. CORROSION RESISTANCE OF NICKEL ALLOYS USED FOR FUSED-SPRAYED COATINGS (REF. 310)

Reagent	Corrosion Resistance of Coating A(a, b)	Corrosion Resistance of Coating B(a)	Corrosion Resistance of Coating C(a)
Nitric acid, up to 2 per cent	F	--	F-G
Nitric acid, up to 4 per cent	--	--	F
Hydrochloric acid, up to 2 per cent	G	G	G
Hydrochloric acid, up to 5 per cent	F	G	G
Phosphoric acid, all concentrations	G	G	G
Sulfuric acid, up to 75 per cent	G	G	G
Sulfuric acid, up to 96 per cent	F	F	F
Sulfuric acid, 150 F, up to 25 per cent	--	--	F
Sulfuric acid, 150 F, above 25 per cent	--	--	G
Hydrofluoric acid, all concentrations	F-G	F-G	F-G
Fresh water, all types	G	G	G
Mine water, neutral and acid	G	G	G
Boiler water, distilled water	G	G	G
Sea water, brackish water – stagnant	F	F	F
Sea water, brackish water – flowing	G	G	G
Caustic solutions, all concentrations	G	G	G
Neutral salts, general	G	G	G
Acid salts	F	F	F
Anhydrous ammonia	G	G	G
Ammonium hydroxide	G	G	G

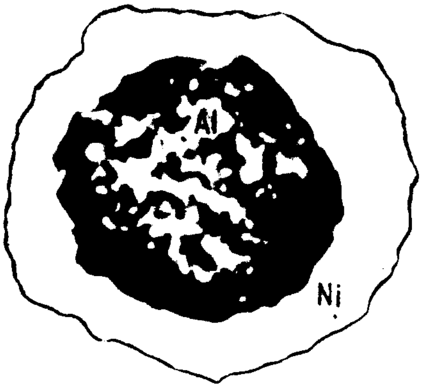
(a) Powder composition prior to coating
A = 10Cr, 2.5B, 2.5Si, 2.5Fe, 0.15C, Bal Ni
B = 17Cr, 3.5B, 4.0Si, 4.0Fe, 1.0C, Bal Ni
C = 16Cr, 4.0B, 4.0Si, 3Mo, 3Cu, 2.5Fe, 0.5C, Bal Ni.

(b) Corrosion resistance
F = fair – indicates a corrosion rate of less than 0.030 inches per year
G = good – indicates a corrosion rate of less than 0.005 inches per year.

TABLE XLIX. PROPERTIES OF NICKEL ALUMINIDE (REF. 313)

Specific gravity	6.0
Typical hardness	Brinell 137 (Rockwell B 75 approx)
Average particle hardness	In plasma-spray coatings - KHN$^{(a)}$ 320 to 685
	In airless-flame coatings - KHN$^{(a)}$ 290 to 554
Typical tensile strength	In plasma-spray coatings - 25,000 lb/in.^{2}
	In airless-flame coatings - 5,200-5,600 lb/in.^{2}
Electrical resistivity	220 x 10^{-6} ohm-cm (at 24 C)
Thermal expansion	12.6 x 10^{-6}/C (26 to 550 C)
Magnetic properties	Slightly magnetic
Thermal-shock resistance	Excellent
Oxidation resistance	Very good
Erosion resistance	Good
Chemical composition	Approx equal parts Ni, Al, and Ni_3Al, with some miscellaneous oxides

(a) 50-g load.



500X

FIGURE 52. PARTICLE OF NICKEL ALUMINIDE POWDER SHOWING THE NICKEL-CLAD ALUMINUM CORE BEFORE SPRAYING (REF. 313)

ideally rough substrate for bonding other flame-sprayed materials. Sprayed metals and ceramics up to 0.1 inch thick bond to many bases with an undercoat of 0.004 to 0.006 inch of nickel aluminide. It is not self-bonding to tungsten and there may be other materials to which it will not self-bond reliably, but special techniques may prove successful with such materials.

TABLE L. TYPICAL BOND STRENGTHS OF HIGH-MOLYBDENUM AND NICKEL ALUMINIDE COATINGS (REF. 313)

		Nickel Aluminide, lb/in.^{2}	
	Molybdenum, lb/in.^{2}	Airless Flame	Plasma Flame
In Relation to Various Base Materials			
Steel	2,150	2,750	3,000
Steel, hardened	2,150	2,750	3,000
Stainless steel	--	2,750	3,000
Nitrided steel	--	2,750	3,000
Grey cast iron	2,250	2,750	3,000
Aluminum	2,150	2,750	3,000
Titanium	--	2,750	3,000
In Relation to Various Sprayed Coatings			
Mild steel	2,150	2,750	--
18/8 stainless steel	2,250	2,750	--
Aluminum	1,640	2,200	--

As a refractory material, nickel aluminide can be considered for use by itself to produce coatings from 0.010 to 0.030 inch thick. At about 1520 C the coatings exhibit sweating, or minor melting, although the structure of the coating remains intact. The sweating continues up to a temperature of about 1650 C, when it stops with no further melting to 1760 C.

PLASMA SPRAYING

Plasma spraying is a recent development in the metallizing field and has been gaining wider use as a method for producing surface coatings of refractory materials. The outstanding advantage of the plasma torch over conventional metal-spraying equipment is that a very high-temperature plasma can melt all known materials. While the state of development of plasma spraying is still in its infancy and most investigations have been conducted on the spraying of high-melting-point materials, nevertheless some nickel and nickel alloys have been plasma sprayed.

The plasma torch is a gas-stabilized electric-arc device. A pressurized plasma-forming gas flows into a plenum chamber where it is ionized by an arc formed between a tungsten electrode and a water-cooled copper or brass nozzle that serves as the other electrode. As the ionized gas flows into the reduced section of the nozzle it becomes highly concentrated resulting in corresponding increase in temperature. There is also a thermal-pinch effect that tends to concentrate the current in the central region of the plasma. Self-induced magnetic fields constrict the plasma further and produce localized temperatures of over 15,000 F.

There are two types of plasma torches, nontransferred arc and transferred arc. In the nontransferred arc, both electrodes are part of the torch so that the arc is wholly contained within the torch, and most of the energy is directed to heating the gas. This is the type of torch used for spraying. The transferred-arc torch uses an external workpiece for one of the electrodes, and it is used primarily in metal cutting. With the nontransferred arc a number of plasma-forming gases may be used: argon, helium, nitrogen, or hydrogen. The nitrogen and hydrogen gases have the best heat-transfer characteristics. A schematic drawing of a typical nontransferred-arc type of plasma-spraying gun is shown in Figure 53.

Nickel-base materials for plasma spraying are generally in powder form and of the same composition as those listed in Table XLVI for fused metallized coatings. Nickel-base materials have been investigated for use as refractory, hard-facing and corrosion-resistant coatings by Ingham and Sheparad (Ref. 311). The properties of plasma-sprayed nickel-base coatings are listed in Table LI.

Powder-feed mechanisms are an important consideration where high-quality deposits are required. The injection of powder into the plasma stream as well as particle size are very critical. Powder size, injection angle, and particle velocity must all be closely controlled to assure high-quality reproducible deposits.

Although the techniques employed in the plasma method of metal spraying do not vary radically from those of the oxyacetylene method in plasma spraying, increased attention must be given to the effect of process variables on the quality of the work and preservation of the substrate.

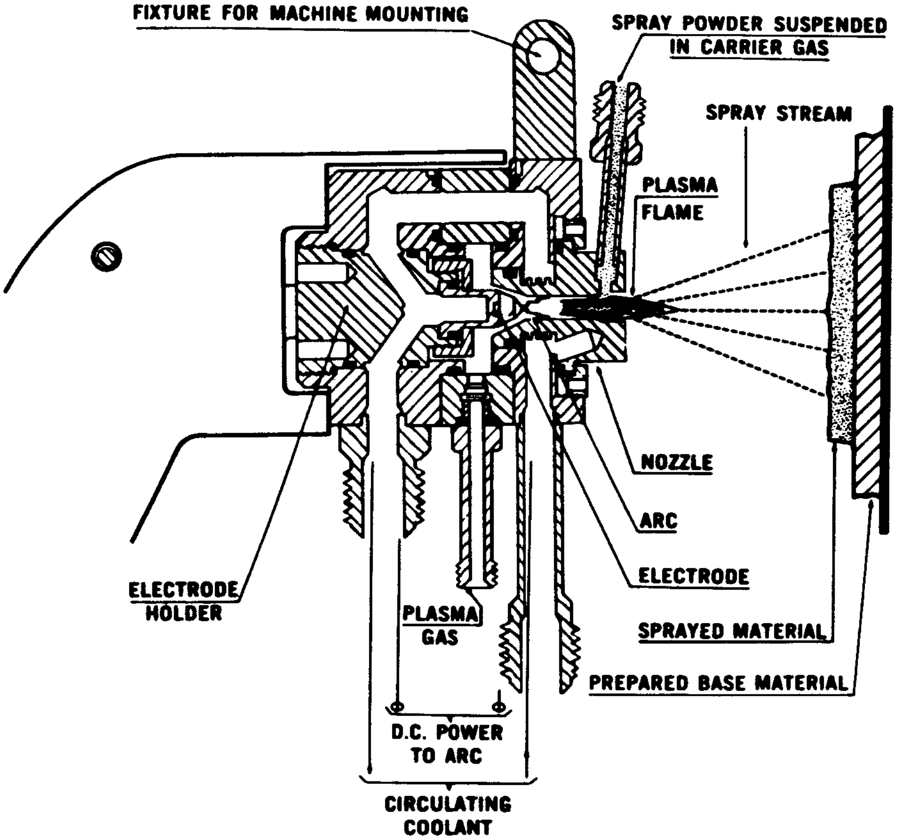


FIGURE 53. CROSS SECTION OF A PLASMA-FLAME SPRAY GUN (REF. 311)

TABLE LI. PROPERTIES OF PLASMA-SPRAYED NICKEL-BASE COATINGS (REF. 311)

Material	Melting Point, F	Thermal Expansion $\times 10^{-6}$/F	As-Sprayed Texture	Porosity	Bonding Properties	Corrosion and Oxidation Resistance[(a)]			Finishing Characteristics[(b)]	Hardness	Coating Density, g/cc	Silica-Slurry Wear Rate, 0.001 in./hr	Bond Tensile Strength (Mil of Steel Base), psi
						Chemicals	Molten Metals or Glass	High-Temperature Oxidation					
Nickel aluminide	2750	7	Medium	Very low	Self-bonding	--	A	D	M, fair G, excellent	R_B 75	6.0	17.5	3,000
99.9^+ Ni (coarse)	2650	7.39-9.04 212-1652 F	Medium	Medium	Good	C	--	--	M, fair G, good	--	--	--	--
99.9^+ Ni (fine)	2650	7.39-9.04 212-1652 F	Fine	Low	Good	C	--	--	M, fair G, good	--	--	--	--
80Ni-20Cr	2550	9.8 158-1832 F	Fine	Very low	Excellent	C	--	--	M, fair G, good	--	--	--	--
Ni-10Cr-2.5B	1850 1950	Approx 6	Medium	Very low	Very good	B	B	--	M,G, very good	R_C 30-35 (fused)	8.2 (fused)	--	--
Ni-17Cr-2.5B	1850 1950	Approx 6	Fine to medium	Very low	Very good	B	B	--	G, very good	R_C 62 (fused)	7.8 (fused)	3.2 (fused)	--
Ni-16Cr-3B	1850 1950	Approx 6	Medium	Very low	Very good	B	B	--	G, very good	R_C 60-62 (fused)	7.9 (fused)	--	--
Ni-11C-2.5B-35 WC	1850 1950	--	Fine	Very low	Very good	--	--	--	G, very good	--	8.8 (fused)	--	--

(a) A – coating usually used as sprayed
B – fuse coating for optimum protection of base
C – recommend sealer to provide optimum protection of base
D – recommend undercoat for optimum protection of base.

(b) M – machined
G – grind.

(1) Torch-to-work distance affects powder dwell time, velocity, impact, density, substrate temperature, deposit efficiency, oxidation, particle adhesion, coating bond strength, and stress in the substrate and coating.

(2) The rate of torch traverse affects the degree of oxidation of the coating sublayers and determines the amount of thermal stress in the coating.

(3) A 90-degree torch-to-work angle may not always produce the best results. A slightly offset position of the torch prevents particle bounce back, provides a more desirable directional exhaust, and takes advantage of directional surface roughening for better mechanical bonding.

(4) Oxidation of the coating may be controlled by the use of an inert gas surrounding the torch-impact work area.

(5) The ratio of electrical power to gas flow determines the heat content of the plasma stream, the velocity of the stream, and the melting and deposition potential of the torch. Materials respond differently in the plasma. Gas-flow rate and electrical power must be determined for each special application.

Some of the variables that control the torch are electrical-power input and stability, plasma-forming gas, volume and velocity of the carrier gases, configuration of torch electrodes, externally applied magnetic effects, heat content of the gas, and efficiency of the torch-cooling system.

Because the plasma flame imparts a large amount of heat to the workpiece, rapid dissipation of heat at the point of flame-to-work contact is necessary to prevent overheating and oxidation of the deposits. Most substrate cooling is achieved by internal circulation of cold water, or by an external blast of inert gas impinging on the substrate on both sides of the torch flame.

FLAME PLATING

A third process for spraying nickel and nickel alloys is called flame plating and has been described by McGeary and Koffskey (Ref. 314) and by Doyle and Lambert (Ref. 315). While nickel and nickel alloys are not the most popular flame-plating alloys, nevertheless, nickel coatings can be flame plated successfully. The more commonly used coatings are listed as follows:

WC + 9Co
WC + 13Co
WC + 15Co
$60Al_2O_3 + 40TiO_3$
25WC + 5Mi + mixed W-Cr carbides
$80Cr_3C_2$ + 20Nichrome
$70Cr_3C_2$ + 30Nichrome
$80Cr_2O_3 + 20Al_2O_3$
$99Al_2O_3$.

The flame-plating process utilizes the energy of detonation through the use of a specially constructed gun (Figure 54).

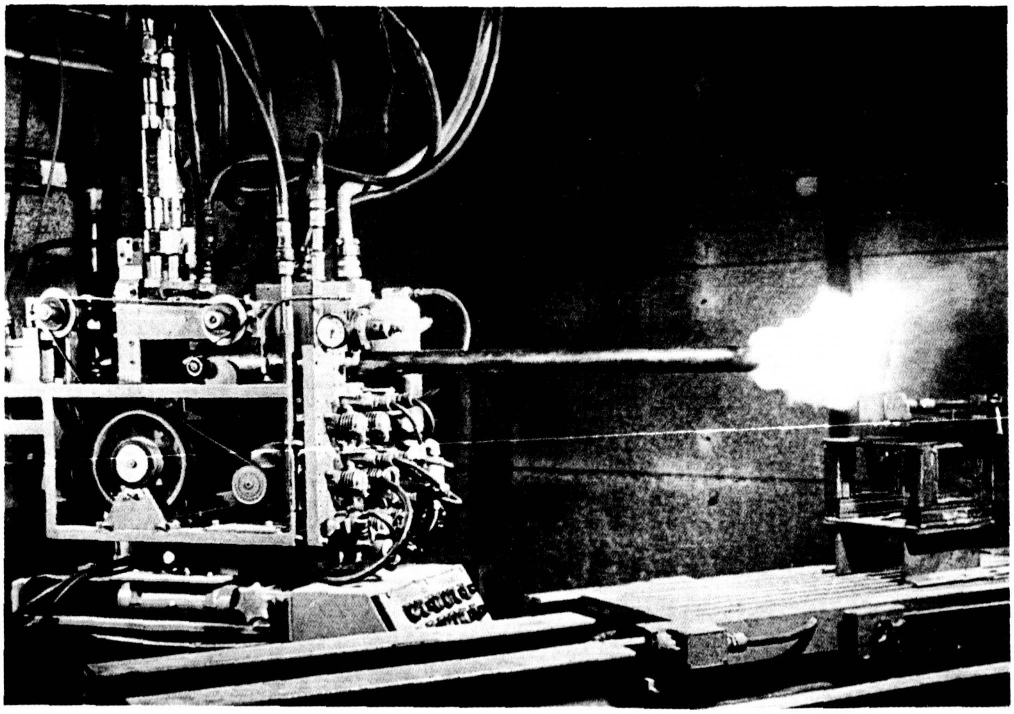


FIGURE 54. FLAME-PLATING GUN (REF. 314)

Metered amounts of oxygen and acetylene, together with a carefully controlled amount of powder, are fed into the chamber of the barrel by means of a regulated sequence of cam-operated valves. When the precise quantities of the mixture of oxygen, acetylene, and nickel powder, suspended in a stream of nitrogen, have been admitted into the firing chamber, a further cam-operated valve opens to admit a stream of nitrogen for the purpose of protecting the valves from detonation. The powder remains suspended in the explosive mixture of oxygen and acetylene until a spark ignites the mixture, producing heat and pressure waves. The confining shape of the barrel concentrates these waves into a small area with a tremendous heat and pressure buildup. When the temperature reaches 3000 C the heat waves begin to move faster than the pressure. This phenomenon is detonation. The process in diagrammatic form is shown in Figure 55.

In a detonation of an oxyacetylene mixture, the shock front velocity reaches 9600 ft/sec, or approximately nine times the speed of sound. The actual velocity of the gas as it leaves the gun is somewhat lower, but it is in the region of 4000 ft/sec.

The flame-plating process takes the advantage of ultrasonic speed to impart a high kinetic energy to the particles of the coating material. The particles are accelerated and reach an exit speed of 2500 ft/sec. The part to be coated is held at a prescribed distance, usually 2 to 4 inches from the end of the gun barrel. At impact, the hot particles of the coating material are further heated by the release of their kinetic energy. Under these conditions, particles of the coating material are flattened and develop a tenacious bond. Detonation is repeated 260 times a minute. Each cycle applies about 0.25 mil of material, and successive particle impacts build up the coating to the desired thickness.

Heat input from the hot gas and powder lasts for only a few milliseconds. Cooling occurs during the much longer period between blasts. Thus, it is possible to maintain the part at a sufficiently low temperature to avoid metallurgical changes and distortion even at the interface.

Before being flame plated, the part may be masked off to restrict the coating to a certain area. Masking can be individually applied or incorporated into a fixture that holds the part in its proper position. To achieve the best possible bond, most surfaces are grit blasted before coating to remove oxides, scale, or other contaminants.

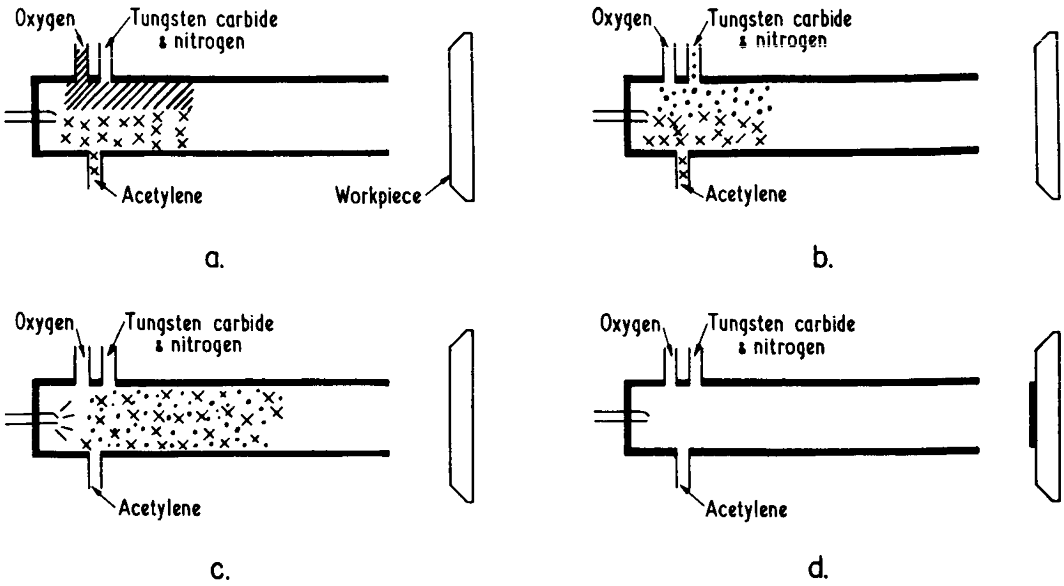


FIGURE 55. DIAGRAMMATIC FORM OF FLAME-PLATING PROCESS (REF. 315)

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.

Generally, any material with a hardness below Rockwell C 60 can be flame plated. Hardness and strength of the material, however, must be sufficient to resist permanent deformation in service, especially if point or line loads are involved. Flame-plated coatings have been successfully applied to critical areas of precision parts made from steel, cast iron, aluminum, copper, brass, bronze, molybdenum, titanium, beryllium, nickel, magnesium, and graphite.

Since the essence of flame plating is the impingement of high-velocity coating particles onto the surface of a part, only areas permitting sufficient access will be coated evenly. This rules out narrow holes, blind cavities, and deep V-shaped grooves.

A flame-plated coating has a higher density than those obtained by other spraying methods. In most instances coating porosity is 1 per cent or less. The thickness of the coating can be controlled precisely over the normal thickness ranges - from 0.0005 to 0.010 inch.

The as-coated surface is usually in the range of 125 to 225 rms microinches, much like the surface of emery paper. The roughness can be reduced to 50 to 70 rms microinches by brush finishing or polishing, and to less than 1 rms microinch by precision diamond grinding and lapping.

Equipment and buildings needed for flame plating are expensive. The high noise level, around 150 decibels, generated by the flame-plating gun requires specially designed, soundproof cubicles. Double walls are 3 feet thick and insulated, and a double door and triple-paned window isolate the operation. Technicians operate the flame-plating gun from a control panel outside the cubicle. Since the large capital investment required cannot be justified by many users, parts to be flame plated are sent to custom-engineering service centers for plating. Linde maintains three production plants for the custom-engineering service.

STATE OF DEVELOPMENT AND POSSIBLE APPLICATIONS

The state of the art of fused metallized coatings is well established and there are many applications where nickel or nickel alloys are used for corrosion and wear-resistant coatings. Table LII indicates the frequency of use of nickel coatings. The information in this table has been abstracted from a questionnaire prepared for the

International Institute of Welding. As can be seen in Table LII, the primary use is for wear-resistant coatings.

TABLE LII. FREQUENCY OF USE OF METALLIZING IN SELECTED APPLICATIONS OF NICKEL COATINGS (REF. 308)

Material	Application:	1[(a)]	2[(b)]
Nickel	Corrosion protection		
0.010 to 0.090 in. thick	Carbon packings	X	
	Pumps	X	
	Rolls	X	
Ni-Cr-Si-B, Ni-Si-B	Wear-resistant coatings		
0.010 to 0.250 in. thick;	Machine parts		X
melting temperature	Pump plungers		X
1850 F to 2050 F	Corrosion wear		X

(a) Used to some extent.

(b) Used a great deal.

More specific applications and potential uses of metallized nickel-base coatings are given in Table LIII.

The state of the art of plasma spraying is still in its infancy. Thus the present applications are rather limited. Table LIV shows possible areas of usefulness for plasma-sprayed nickel and nickel-base alloys. Applications with the greatest potential are in the areas of corrosion resistance and wear resistance. When sealed, nickel-base coatings resist corrosion and also impart added wear resistance to pump plungers, pump rods, hydraulic rams, packing sections of steam-turbine shafts, boat tailshafts, and valves.

TABLE LIII. APPLICATIONS OF METALLIZED NICKEL-BASE COATINGS (REFS. 308, 310)

Material	Application
	Corrosion Protection
Ni-Cr-Si-B	Glass molds
	Heat-treating conveyor rolls
	Pump parts
Ni-Cr	Bonding ceramics
Ni-Al	Slag pots
	Valve heads
	Valve throats
	Heat-treating furnace pots
	Injector nozzles
	Heat-treating conveyor rolls
	Hot-metal forming dies and rolls
	Kiln fixtures
	Wear-Resistant Coatings
Ni-Cr-Si-B	Pump plungers
	Crusher rolls
	Pulverizing equipment
	Paper-core winding mandrels
	Pump sleeves
	Conveyor screws, bearing housings
	Steel-mill roller-guide boxes
	Valve plugs
	Plug gages
	Farm implements
	Mixing-machine blades
	Textile spindels
	Sizing mandrels
	Abrasive wheel molds, plungers
	Diesel-engine valves, stems
Ni-Cr-Si-B	Extrusion rams
	Wire-drawing capstans
	Concrete-block cores
Ni-Cr-Si-B-WC	Oil-well tool joints
	Conveyor screws, housings
	Buffing fixtures
Ni-Cr	Repair of brick-making machine

TABLE LIV. APPLICATIONS OF PLASMA-SPRAYED, NICKEL-BASE COATINGS (REFS. 308, 311)

Material	Melting Point, F	Possible Areas of Usefulness
Nickel aluminide	2750	Self-bonding undercoat, especially in refractory applications; oxidation protection
99.9Ni (coarse)	2650	Corrosion resistant when properly sealed
99.9Ni (fine)	2650	Corrosion resistant when properly sealed
80Ni-20Cr	2550	Resistant to heat, corrosion, and oxidation; for general wear very fine machine finish is needed
Ni-10Cr-2.5B	1850 1950	Good wear and corrosion resistance; fused coating may be machined with ordinary tool steel
Ni-17Cr-2.5B	1850 1950	Very good wear and corrosion resistance, use fused or unfused
Ni-16Cr-3.0B	1850 1950	Very good wear and corrosion resistance, use fused or unfused; used for heavier fused build-up thickness

CONCLUSIONS AND RECOMMENDATIONS

CASTING

The foundry technology for producing castings from air-melted or vacuum-melted nickel-base alloys is fairly well established. Castings of high quality can be produced when sound metallurgical practices are employed to cope with characteristics that may be peculiar to a specific alloy. The main problem is in ascertaining that the foundry has awareness of the characteristics of the alloy being cast and an awareness of the technology and the ability to apply the technology that is required. In this regard, it would be desirable to develop a list of foundries having certified capability to produce castings of high quality from specific alloys. A program for certifying foundries is recommended.

POWDER METALLURGY

Comparatively speaking, the powder metallurgy of pure nickel does not embrace any difficult problems. Powder production, consolidation, and shaping are straightforward matters. However, there are problems associated with the full-scale production of nickel-base alloys, for example, some of the nickel-base superalloys.

One of the principal difficulties arises from the fact that these alloys contain readily oxidized components (aluminum, titanium, and zirconium) that cannot be reduced in hydrogen. Methods for making uncontaminated powder are needed.

Also, development work is needed embracing actual nickel-base-alloy parts production for the purpose of working out practical fabrication procedures. Those would be aimed at supplanting cast alloy routes where a powder route might be more economical or provide improved products. Success would be predicated on the attainment of equal or better mechanical properties, notably high-temperature creep strength and ductility.

ELECTROFORMING

A problem facing organizations utilizing electroforming is the incompleteness of information on effective procedures for control of properties and structural characteristics of electroformed nickel and nickel alloys. It is recognized that properties and structure are influenced by solution composition, pH, temperature, and impurities; and by current density, agitation, and other factors. Quantitative data have been developed for some of these process variables on an individual basis, but variations in others have not been investigated, quantitatively, nor have the interrelated effects of the influential variables been studied. The complexity of controlling a large number of variables independently to achieve good control poses sophisticated and costly monitoring techniques. There is reason for believing that such complexity can be reduced considerably by exercising direct control of the reduction process by monitoring the cathode potential at significant surfaces. It may be possible to control the individual and interrelated effects of many variables such as temperature, current density, solution pH, and metal ion concentration by controlling just the cathode potential during the production of electroformed nickel. A study is recommended for determining the usefulness of cathode potential control for improving the adaptability of electroforming for meeting present-day and future requirements for good reproducibility of specific properties and structure.

Research on alloy electroforming of nickel with cobalt, iron, tungsten, manganese, alumina, and other metals and oxides has been conducted on a piecemeal basis in at least 15 different laboratories, and specific improvements in some properties have been observed for some alloying elements. The property data needed by engineers responsible for material selection are spotty and incomplete. The development of handbook data for alloys of nickel is important for guiding electroforming into appropriate channels of application. The desirable comprehensive aspect of such a development is beyond the capability of any individual industrial organization and is a logical pursuit for Government funding and administration.

Because of the low deposition rate of present-day nickel electroforming, capital equipment costs are relatively high, and control problems are complex. Higher deposition rates of the order of at least 0.1, but preferably 0.5 to 1.0 in./hr, would improve the adaptability of nickel electroforming and increase its usefulness for fabricating complex shapes. Studies directed to development of electroforming at higher rates should be encouraged.

CHEMICAL VAPOR DEPOSITION

The use of nickel carbonyl always involves the problem of toxicity, which cannot be taken lightly. However, safe-handling procedures have been worked out so that the problem can be met.

In deposition from a carbonyl, a variable amount of carbon is always codeposited. In any application for which the carbon would be

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distinctly undesirable, steps would have to be taken to minimize the amount deposited. The approach to this problem should include a program to determine the mechanism of carbon deposition and the nature of the slow step in the process. Methods of inhibiting this reaction would then be sought.

In the deposition of nickel alloys, the rates of deposition of the components must be regulated to obtain the desired composition in the final shape. The effects of the various process variables on the rate of nickel deposition in the regions of kinetic and diffusional control have been well established. Studies should be carried out for any added components. Finally, the effects of one plating gas on the deposition rate from the other should be established.

METAL SPRAYING

Although the metal spraying process is over 60 years old, there still is need for further development in fused-metallized coatings. The metal-spraying processes have developed largely as a result of practical experience, and not enough information has been published to form the basis of specifications or to give answers to the many problems that arise. Many of these problems apply not only to the spraying of nickel and nickel alloys but to other metals as well.

Some of the main problem areas in fused-metallized coatings are:

(1) Preparation of Work Surfaces. The blasting process needs to be examined to determine the best abrasives and procedures.

(2) Spraying Tools. A theoretical study of various nozzle designs is needed. All events that occur during spraying between the pistol nozzle and work surface need study. By means of high-speed color photography, an examination of the transit of metal from the nozzle to the substrate might lead to improved nozzle design.

(3) The Sprayed Deposit. A study of spraying procedure and its effect on the properties of sprayed-metal coatings is needed. This includes the effects of the powder particle size and shape on the properties of the coating, the effects of working distances, and the microstructure of deposits obtained under differing conditions.

(4) After-Treatment of Sprayed Metal. A study of the behavior of various paints and pore sealers in corrosive media is needed. More information is needed on the effects of heat treatment and the dimensional changes that occur in the sprayed deposits and in the substrates.

(5) Choice of Materials for Spraying. It would be desirable to set up standard specifications for sprayed coatings.

Plasma-jet spraying is the most recent advance in the metal-spraying industry, and research in the field of plasma spraying is now producing data concerning fundamentals of the process. The following areas are suggested for further investigation:

(1) Studies of basic plasma-spraying parameters with the objective of developing improved equipment

(2) Determination of the physical properties of powders that influence applicability to the plasma-spray process.

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THE MAKING OF NICKEL AND NICKEL-ALLOY SHAPES BY CASTING, POWDER METALLURGY, ELECTROFORMING, CHEMICAL VAPOR DEPOSITION, AND METAL SPRAYING

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