NASA-SP-5086

TECHNOLOGY UTILIZATION

SHAPING OF PRECIPITATION-HARDENING STAINLESS STEELS BY CASTING AND POWDER-METALLURGY

An AEC/NASA HANDBOOK

NATIONAL AERONAUTICS
AND
SPACE ADMINISTRATION

Printed in USA. Price $3.00. Available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U. S. Department of Commerce, Springfield, Virginia 22151.

USAEC Division of Technical Information Extension, Oak Ridge, Tennessee

NASA-SP-5086
METALS, CERAMICS, AND
MATERIALS (TID-4500)

NASA-GEORGE C. MARSHALL SPACE FLIGHT CENTER

SHAPING OF PRECIPITATION-HARDENING STAINLESS STEELS BY CASTING AND POWDER-METALLURGY

By

J. G. Kura, V. D. Barth, and H. O. McIntire

Prepared for

Manufacturing Engineering Laboratory

In Cooperation with

Technology Utilization Office

Under the Supervision of

Redstone Scientific Information Center
U. S. Army Missile Command
Redstone Arsenal, Alabama
MSFC Order No. H-76715
Report No. RSIC-491

Subcontracted to

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio
Contract No. DA-01-021-AMC-11651(Z)

ABSTRACT

The state of the art of making shapes of precipitation-hardenable stainless steels by methods other than machining from wrought materials is reviewed. The methods reviewed are casting and powder metallurgy. Of these methods, only casting is extensively practiced. Powder metallurgy is a promising procedure for 17-4 PH stainless, but techniques are not well advanced for handling the grades that contain titanium or aluminum such as PH 15-7 Mo and A-286.

FOREWORD

Precipitation-hardening stainless steels are potentially useful wherever corrosion resistance and high strength at high temperatures are needed. They were developed initially to meet urgent requirements in World War II, but new alloys and methods of processing have since been introduced to assist engineers concerned with missiles and space vehicles and with various applications in the field of nuclear science and technology.

The Atomic Energy Commission and National Aeronautics and Space Administration have established a cooperative program to make available information, describing the technology resulting from their research and development efforts, which may have commercial application in American industry. This publication is one of the many resulting from the cooperative effort of these agencies to transfer technology to private industry.

This survey is based on information contained in a series of reports originally prepared by Battelle Memorial Institute for the Manufacturing Engineering Laboratory of the George C. Marshall Space Flight Center. The original information has been updated and revised in writing the current, seven volume survey. These volumes were prepared under a contract with the NASA Office of Technology Utilization which was monitored by the Redstone Scientific Information Center.

PREFACE

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

The making of shapes of precipitation-hardening grades of stainless steel by casting and by powder metallurgy is reviewed in this report. The information on methods of handling these useful grades of stainless steel is scattered among a variety of sources. The sources of information include the open literature, unpublished reports on Government contracts, and personal contacts with leading authorities in the respective fields. It is the purpose of this report to present the state of the art in making parts from these grades by methods other than machining from wrought materials. This report covers casting and powder-metallurgy methods. Other methods considered were metal spraying, electroforming and chemical vapor deposition. It was found that these latter methods have not been used for making shapes of precipitation-hardenable grades of steel and therefore no mention is made of them in the text of this report.

This report brings together information from a variety of sources for the purpose of making it available to those who can use it in industry, the military and the aerospace industry.

TABLE OF CONTENTS

Page

TABLE OF CONTENTS
(Continued)

LIST OF ILLUSTRATIONS

LIST OF TABLES

SHAPING OF PRECIPITATION-HARDENING STAINLESS STEELS BY CASTING AND POWDER-METALLURGY

SUMMARY

CASTING

Ten casting alloys constitute the family of precipitation-hardenable stainless steels. The 17-4 PH alloy is cast in sand molds, in investment molds, or by the centrifugal process. The A-286 and 14-4 PH alloy are cast in investment molds. Sand and investment molds are used to cast the AM 355 alloy. The PH-55A, PH-55B, PH-55C, PH-55D, CB-7Cu, and CD-4MCu alloys are cast in sand molds. All of the alloys are air melted with the exception of the A-286 alloy which is vacuum melted.

For casting in sand molds or by the centrifugal process, there is essentially no top limit on the weight of the casting other than the restriction imposed by available molding equipment. Investment castings weighing up to 400 pounds have been produced, but they are usually under 20 pounds in weight. The normal dimensional tolerance on sand castings is about 1/16 inch per foot, but can approach the tolerance of ±0.005 inch per inch which is typical of investment castings. Optimum conditions give a finish of 100 to 250 microinches rms on castings made in green sand molds and a finish of 10 to 85 microinches rms on investment castings. The usual finish on green sand castings is 250 to 1000 microinches rms and 80 to 125 on investment castings.

The 17-4 PH alloy is melted in an indirect arc furnace or in an induction furnace. For good control of composition, a master alloy is used for remelting. Castings made from a master alloy in the form of bar stock have better ductility than castings made from a shotted master alloy. Heats are deoxidized with an addition of calcium manganese silicon. Deoxidation by addition of aluminum is effective but a residual of 0.15 to 0.20 per cent aluminum is detrimental to ductility. An excessive pickup of carbon or nitrogen during melting causes loss in response to heat treatment because of stabilization of the austenite phase. In such cases, a solution treatment at a temperature as low as 1400 F instead of the normal temperature of 1900 F makes the austenite phase less stable and aging response is regained.

Because the 17-4 PH alloy is susceptible to microshrinkage, ductility decreases as the size of the cast section increases. The strength is unaffected. Ductility is also affected adversely if copper segregates at the grain boundaries as a result of the copper content being too high, the pouring temperature being too low, or the cooling of the casting being too slow during solidification. The latter two operations can also result in the formation of acicular delta ferrite in the structure which cannot be changed by heat treatment and causes low ductility. If welding is anticipated, underbead cracking can be minimized by keeping the copper content near the lower limit of the specification, particularly for castings produced in sand molds or by the centrifugal process. Susceptibility to hot tearing is another problem in designs having changes in section size or corners. The 14-4 PH alloy has the same general foundry characteristics as the 17-4 PH alloy.

The foundry characteristics of the AM 355 alloy are similar to the 17-4 PH alloy. A cover of argon gas is suggested when melting in an indirect arc furnace to minimize the pickup of carbon. Optimum properties after heat treatment are obtained with a carbon plus nitrogen content in the range of 0.18 to 0.24 per cent. At higher levels the response to heat treatment is lost because of stabilization of the austenite phase. A high pouring temperature and slow cooling of the casting during solidification promote formation of delta ferrite, but the effect of the delta ferrite on the properties of the casting is not yet well defined.

Some segregation occurs in AM 355 alloy castings. Homogenization treatments at high temperatures do not enhance the properties. Properties are improved by preceding the 1750 F solution-anneal treatment with an "equalizing" treatment at 1475 F. In order that maximum hardening response occurs upon aging, the solution-anneal treatment must be followed by a sub-room-temperature treatment to transform the austenite. Because the AM 355 alloy undergoes a significant change in volume during heat treatment, inspection of the dimensions should be done after the aging treatment.

For consistent properties at a higher level, particularly ductility, the A-286 alloy must be vacuum melted from certified vacuum-melted master heats. Crucibles of alumina, magnesia, or zirconia are satisfactorily inert after they are cured by several wash heats. Alumina or zirconia facing material is preferred in investment molds (poured at a preheat of up to 1900 F) because a silica facing may react to form surface defects in the casting. The production cycle for vacuum casting a 12-pound charge is 6 minutes. The cost is about one-half more than that for melting and casting in air. Melting and casting under vacuum results in fewer inclusions in the casting and fewer misruns, particularly in complex shapes with thin sections. A solution anneal at 2000 F precedes a solution anneal at 1650 F which is followed by a 13-hour aging treatment at 1300 F.

Conventional stainless steel melting and sand casting practices are used with the PH-55A, PH-55B, PH-55C, PH-55D, CB-7Cu, and CD-4MCu alloys. The first three alloys are weldable, but should be heat treated after being welded. The last three alloys are the only ones of this group that are not useful at elevated temperatures. The PH-55D alloy is used for its high abrasion resistance as well as its corrosion resistance, but its brittleness is similar to tool steel after it is age hardened. The last two alloys make strong, corrosion-resistant castings; both of them are weldable, but the CD-4MCu alloy should be heat treated after it is welded.

POWDER METALLURGY

Production of parts from stainless steel powders, particularly Types 304, 316L and 410, is growing rapidly. However, there has been little experience with the precipitation-hardening grades of stainless, probably because of disappointing results in the early attempts to use them in the form of powders. The precipitation-hardening stainless powders are harder than conventional stainless steels and high compacting pressures are required to develop the green strengths needed for handling. The martensitic and semiaustenitic grades are very sensitive to nitrogen content. High nitrogen content results in stabilization of austenite and loss of hardening response. Nitrogen can be absorbed during melting, during atomization of the molten alloy, and during sintering. Measures can be taken that will avoid appreciable nitrogen pickup, identified as the cause of erratic heat-treating response. Recently there has been renewed interest in the precipitation-hardening grades and at least two companies expect to begin production soon of parts from 17-4 PH powder. 17-4 PH is a grade of stainless steel that contains no aluminum or titanium and therefore, the powders are less difficult to handle without contamination than other precipitation-hardening stainless powders.

PH 15-7 Mo powders have been pressed into billets and hot extruded with reduction ratios of about 16:1. Properties of the extruded product were reported to be similar to those of the conventional wrought material.

Sintering procedures for 17-4 PH powders are fairly well established. A dry-hydrogen or vacuum atmosphere and temperatures of 2200 to 2300 F appear to be satisfactory. Additional study is needed on sintering of the precipitation-hardening grades containing aluminum and/or titanium.

INTRODUCTION

The precipitation-hardenable grades of stainless steel have been a comparatively recent development. They were introduced early in the 1950's for use in the wrought form, but later in the decade their application for use in castings became commonplace. They provide a combination of corrosion and oxidation resistance with higher mechanical strength than can be obtained with conventional grades of stainless steel or with low-alloy steels.

PRODUCTION BY CASTING

There are few precipitation-hardenable stainless steels currently available as casting alloys. Table I lists the alloys and their composition. Alloy A-286 is the only one that is vacuum melted; the rest are all air melted.

Alloys 17-4 PH, 14-4 PH, AM-355, A-286, PH-35A, PH-55B, and PH-55C have useful strength at temperatures ranging from 700 to about 1400 F. The PH-55D, CB-7Cu, and CD-4MCu (also known as 25-5PH) alloys are used for their good strength and corrosion and abrasion resistance at essentially room temperature.

The following sections describe the practice that is used to melt and cast the alloys, and the practice that is used in heat treating the castings. Mechanical properties are presented to illustrate the potential of the alloys and to reveal the effect of certain practices on the properties.

ALLOYS USED AT ELEVATED TEMPERATURES

17-4 PH Alloy.

General Characteristics. The 17-4 PH alloy is a heat-resistant material that has good ductility and good strength up to 600 F. Its resistance to corrosion is better than that of stainless steel Type 410 and is comparable with that of Types 302 and 304. It is characterized by a relatively low aging temperature which helps to

TABLE I. COMPOSITIONS OF PRECIPITATION-HARDENABLE STAINLESS STEEL CASTING ALLOYS

Alloy	Melting Atmosphere	Casting	Composition, %										Developer	Reference
			Carbon	Manganese	Silicon	Chromium	Nickel	Molybdenum	Copper	Nitrogen	Iron	Other		
17-4PH	Air	Sand and investment	0.06(a)	0.70(a)	0.50-1.00	15.5-16.7	3.6-4.6		2.8-3.5	0.05(a)	Bal	0.15-0.40 CB + Ta, 0.04P(a), 0.03S(a)	Armco Steel Corporation	1, 2 3, 4
14-4PH	Air	Investment	0.06(a)	0.7(a)	0.5-1.0	13.5-14.7	3.75-4.75	2.0-2.75	3.0-3.5	0.05(a)	Bal	0.15-0.35 Cb + Ta, 0.02P(a, b), 0.025S(a)	Ditto	5
AM 355	Air	Sand and investment	0.08-0.15	0.40-1.10	0.75(a)	14.5-15.5	3.5-4.5	2.0-2.6		0.05-0.11	Bal	0.15-0.25 C + N, 0.04P(a), 0.03S(a)	Allegheny Ludlum Steel Corporation	6
A-286	Vacuum	Investment	0.08(a)	2.0(a)	1.0(a)	13.5-16.0	24.0-27.0	1.0-1.5		0.1	Bal	0.003-0.01B, 0.1-0.5V, 0.025P(a), 0.025S(a), 0.35Al(a), 1.9-2.3Ti	Ditto	7, 8
PH-55A	Air	Sand	0.05(a)	1(a)	3-3.75	19.5-20.5	8.5-9.5	3.75-4.25			Bal		Copper Alloy Corporation	9, 10
PH-55B	Air	Sand	0.05(a)	1(a)	1.25-1.75	19.5-20.5	8.5-9.5	4.75-5.25	3.25-3.75		Bal		Ditto	9, 11
PH-55C	Air	Sand	0.05(a)	1(a)	3.25-3.75	19.5-20.5	8.5-9.5	3.75-4.25	2.75-3.25		Bal		"	9
PH-55D	Air	Sand	0.05(a)	1(a)	3-5	18-21	9-12	3.75-4.25		0.06-0.15	Bal	0.75-1.25Cb	"	9
CB-7Cu	Air	Sand	0.05(a)	1.0(a)	1.0(a)	15.5-17.0	3.5-4.5		2.3-3.3		Bal		Alloy Casting Institute	12
CD-4MCu	Air	Sand	0.04(a)	1.0(a)	1.0(a)	25-27	4.75-6.0	1.75-2.25	2.75-3.25		Bal	0.04P(a); 0.04S(a)	Ditto	12

(a) Denotes maximum.

avoid distortion during heat treatment. Castings of 17-4 PH alloy can be machined after a solution-anneal heat treatment and then age hardened with little danger of dimensional changes. The contraction that occurs as a result of aging is 0.0004 to 0.0006 inch per inch (Ref. 13).

Castability. Castings of this air-melted alloy are produced in sand molds, investment molds, and by centrifugal casting. For sand or centrifugal castings, the copper content is 2.5 to 3.2 per cent, instead of the 2.8 to 3.5 per cent shown in Table I, to improve the weldability.

Castability of the alloy is good (Refs. 14, 15). For example, a casting measuring 21 inches in length and 6 inches in width was run satisfactorily in a heated, ceramic investment mold when the section was as thin as 0.080 inch (Ref. 16). However, 17-4 PH castings are subject to hot tearing. Therefore, casting design should avoid heavy X- or T-sections, abrupt changes in section size, and sharp corners (Ref. 17). The alloy is also subject to microshrinkage which decreases the ductility of the casting while having little effect on the strength (Refs. 15, 18, 19). Proper gating and risering to establish directional thermal gradients during solidification minimizes the development of microshrinkage.

Castings of the 17-4 PH alloy weighing up to 450 pounds have been produced in sand molds (Ref. 17). Although the capability exists for making investment molds for castings weighing up to 400 pounds (Ref. 20), the usual weight of investment castings is under 20 pounds. Some examples of investment castings are shown in Figure 1.

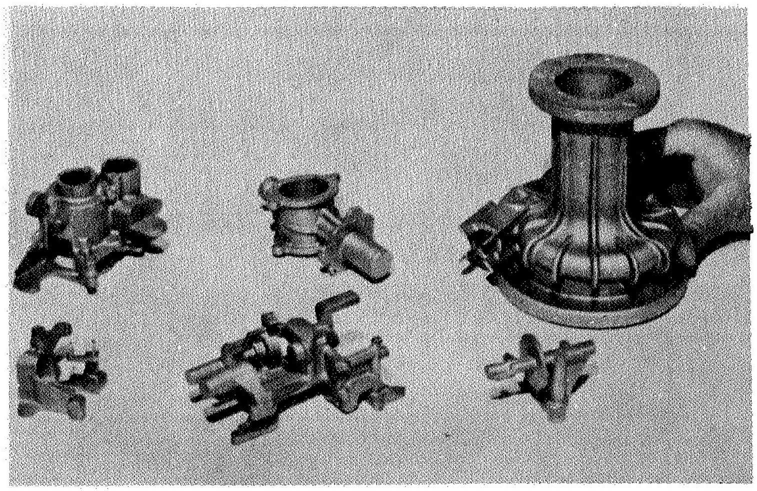

FIGURE 1. INVESTMENT-CAST PARTS OF 17-4 PH FOR THE FUEL-CONTROL SYSTEM OF A JET PLANE (REF. 2)

Under optimum conditions, green sand casting can approach very closely the dimensional tolerances of investment casting, i.e., ±0.005 inch per inch (Ref. 21). Normally the tolerance on green sand castings is about 1/16 inch per foot. Under optimum conditions, the finish on green sand castings is 100 to 250 microinches rms as compared with 10 to 85 microinches rms for investment castings (Ref. 21). Usually, the finish on green sand castings ranges from 250 to 1000 microinches rms (Ref. 21) and 80 to 125 for investment castings (Refs. 22, 23). These tolerances and finishes also apply to the other alloys discussed later.

Heat Treatment. Interest has increased in 17-4 PH castings since fabricators have discovered the cause for earlier erratic response to heat treatment. The composition of the alloy is balanced so that solution annealing for 1/4 to 1 hour at 1875 to 1925 F produces a martensitic matrix upon cooling in air or in oil to below about 270 F. The transformation to martensite is not complete until the temperature drops to 90 F (Ref. 3). A small percentage of delta ferrite is usually present in the as-cast structure and is unaffected by the heat treatment. Maximum hardness in this condition is 36 Rockwell C. Upon aging, the copper-rich constituents precipitate. Optimum strength and ductility are obtained by aging for about 1 hour in the temperature range of 900 to 925 F. Hardness after aging is 40 to 45 Rockwell C (Ref. 24). The heat treatment and minimum properties are covered by a number of Aerospace Material Specifications. The information is summarized in Table II.

Effect of Practice on Properties. When castings have insufficient ductility, the cause is usually the presence of acicular delta ferrite (Refs. 2, 24). Even when the compositional balance is correct, high pouring temperatures and slow cooling rates can promote the formation of delta ferrite as well as a coarse-grain structure. A small amount of delta ferrite, present in globular form, is not detrimental. The form of the delta ferrite is not changed by heat treatment (Ref. 18). A coarse-grain structure affects ductility adversely (Ref. 24).

Low hardness usually indicates that an excessive amount of austenite was retained after solution annealing. This condition can occur from accidental carburizing or nitriding during heat treatment. Carbon and nitrogen stabilize the austenite phase during heat treatment. Stabilized austenite can also be caused by pickup of carbon or nitrogen during melting and casting. For example, this problem was solved in one instance when an induction-melting furnace was used to avoid the carbon and nitrogen pickup that was being experienced in a carbon-arc

TABLE II. SUMMARY OF 17-4 PH ALLOY AEROSPACE MATERIAL SPECIFICATIONS

Aerospace Material Specification	5342 (Ref. 25)		5343 (Ref. 26)		5344 (Ref. 27)		5355A (Ref. 28)	5398A (Ref. 29)
Type of Mold	Investment		Investment		Investment		Investment	Sand or Centrifugal
Homogenization Treatment	2100 F for 1-1/2 hr, air cool to below 90 F		2100 F for 1-1/2 hr, air cool to below 90 F		2100 F for 1-1/2 hr, air cool to below 90 F		2100 F for 1-1/2 hr cool to below 70 F	2100 F for 1-1/2 hr, cool to below 90 F
Solution-Anneal Treatment	1900 F for 1/2 hr per 1/2 inch of section, oil quench to below 90 F		1900 F for 1/2 hr per 1/2 inch of section, oil quench to below 90 F		1900 F for 1/2 hr per 1/2 inch of section, oil quench to below 90 F		1900 F for 1 hr per inch of section (1/2 hr minimum), cool to below 70 F	1925 F for 1 hr per inch of section (1/2 hr minimum) cool to below 90 F
Maximum Hardness	-		-		-		36 R_C	363 BHN
Aging Treatment	1085 to 1115 F for 1-1/2 hr, air cool		985 to 1015 F for 1-1/2 hr, air cool		900 to 925 F for 1-1/2 hr, air cool		925 F for 1-1/2 hr, air cool	925 F for 1-1/2 hr, air cool
Specimen	Cast bar(a)	Casting(a)	Cast bar(a)	Casting(a)	Cast bar(a)	Casting(a, b)	Cast bar or casting(a, b)	Cast bar or casting(b, c)
Tensile Strength, ksi	130	130	150	150	180	180	180	180
Yield Strength (0.2% Offset), ksi	120	120	130	130	160	160	150	150
Elongation, percent in 4D	8	6	6	4	6	4	6	6
Reduction of Area, per cent	15	15	15	12	15	12	15	12
Minimum Hardness	33R_C	33R_C	38R_C	38R_C	40R_C	40R_C	40R_C	375BHN
Maximum Size of Master Heat, lb	7000(d)		7000(d)		7000(d)		7000(d)	15,000(e)

(a) Tensile specimen is to have standard proportions with a 0.25-inch reduced section.
(b) If the cast-to-size tensile specimens fail to meet requirements, specimens may be machined from the castings.
(c) Unless the purchaser permits the use of cast-to-size tensile specimens, the specimens are to be machined from standard keel-block castings.
(d) Castings may be poured directly from a master heat or from a remelt of a master heat. Foundry returns may be used only in the preparation of a refined master heat.
(e) No restriction specified on the preparation other than that it is a single charge not exceeding 15,000 pounds.

furnace (Ref. 18). In another instance, an investment mold that had not been fired sufficiently to eliminate completely the residue in the cavity was the source for carbon pickup during casting (Ref. 3). Although carbon does improve the fluidity of the alloy, it should not be used for this purpose (Ref. 1).

When the composition has been accidentally altered so that the austenite phase is stabilized, the casting will not develop its potential strength after the aging treatment. In such a case, response to heat treatment may be obtained by solution annealing at temperatures below 1900 F. At these lower solution-annealing temperatures (as low as 1400 F), less carbide is taken into solution and thus the austenite that is formed is less stable and will transform to martensite upon cooling (Ref. 30). Because a change in the heat-treatment schedule can often restore hardening capacity in an imbalanced alloy, composition alone should not be a basis for rejection of castings (Ref. 1).

Silicon improves the fluidity and also protects the chromium and columbium from preferential oxidation during melting (Ref. 1). Excessive amounts of delta ferrite are formed if the silicon content is too high.

Columbium is desirable in the 17-4 PH alloy because it inhibits overaging (Ref. 30). It also permits overaging to be done intentionally at higher temperatures to achieve higher ductility without excessive lowering of the tensile strength. However, the columbium content as well as the copper, manganese, phosphorus, and sulfur contents should be kept low because they contribute to dendritic segregation (Refs. 1, 2). Segregation of copper at the grain boundaries can occur if the copper content is too high, the pouring temperature is too high, or cooling of the casting is too slow (Ref. 30). Segregation can impart low ductility (Ref. 18) and cause underbead cracking during welding (Ref. 3). If welding is anticipated, the copper content should be kept to the low side of the specified range. This lower range of copper content results in 5 to 10 ksi loss in ultimate tensile strength and 15 to 25 ksi loss in yield strength.

In a case where segregation of copper was suspected as the cause for low ductility, homogenization for 2 hours at 2100 F prior to solution annealing and aging gave the following improvement (Ref. 18):

	Tensile Strength, psi	Yield Strength (0.2% Offset), psi	Elongation, %	Reduction in Area, %
Not Homogenized	192,000	171,000	4	7
Homogenized	194,000	174,000	10	33

In another instance, up to 80 per cent of a casting lot was rejected because inspection by the magnetic-particle method indicated the presence of cracks (Ref. 31). No indications were found when the castings were homogenized at 2150 F. Subsequent investigation showed that the indications were related to copper segregation.

Unfortunately the 17-4 PH alloy scales serverly in air 2100 F or higher (Ref. 24). Most "neutral" salt baths and endothermic atmospheres tend to carburize the alloy. Exothermic gas is a satisfactory heat-treating atmosphere if kept neutral by adjusting its moisture content (Ref. 2). The adherent oxide film produced on the castings cannot be removed by sand blasting, but can be removed by a sodium hydride bath. A double solution anneal at 1900 F in air is recommended to improve the ductility instead of the high-temperature homogenizing treatment (Ref. 2). Scaling at 1900 F is moderate; at 2000 F it is heavy.

Distortion in large castings or in those having intricate form may be minimized by solution annealing at a lower temperature, such as 1700 F (Ref. 1). The slightly lower tensile and yield strengths that result may still be adequate for the intended applications.

Producers of master alloys provide compositions that can assure castings will meet the specified minimum properties (Ref. 19). Control of the composition lies in the foundry practice. The data in Table III indicate the gains and losses that may be expected in the alloying elements when they are melted in an indirect arc furnace and in an induction furnace (Ref. 30).

TABLE III. RECOMMENDED SPECIFICATION AND REMELT ANALYSES

Element	Specification, %	Specifications for Stock to Be Remelted, %	
		Indirect Arc Furnace	Induction Furnace
Carbon	0.06 max	0.04 max	0.05 max
Nitrogen	0.05 max	0.035 max	0.05 max
Silicon	0.60-0.90	0.70-1.00	0.70-1.00
Manganese	0.30-0.70	0.40-0.70	0.40-0.70
Chromium	15.5-16.75	15.7-16.8	15.7-16.8
Nitrogen	3.25-4.25	3.25-4.25	3.25-4.25
Columbium + Tantalum	0.15-0.40	0.25-0.40	0.25-0.40
Copper	3.00-4.00	3.00-4.00	3.00-4.00

Heats are usually deoxidized with an addition of calcium-manganese silicon. Aluminum can be used but should be restricted to 3/4 to 1-1/2 pounds per ton because a residual of 0.15 to 0.20 per cent aluminum is detrimental to ductility (Ref. 3).

The effect of section thickness on the tensile properties is shown in Table IV (Ref. 17). As the section size increased from 1 to 4 inches, the strength remained essentially constant, but the ductility decreased somewhat.

TABLE IV. EFFECT OF SECTION THICKNESS ON TENSILE PROPERTIES

	Section Thickness, inches(a)		
	1	1-1/2	4
Tensile Strength, ksi	148	145	144
Yield Strength (0.2% Offset), ksi	131	131	128
Elongation, %	19.0	18.5	13.5
Reduction of Area, %	53.3	52.0	36.5
Brinell Hardness Number	302	302	302

(a) Heat treated to a minimum tensile strength of 130 ksi.

For purposes of quality control, adequate ductility cannot be deduced from hardness tests, X-ray inspection, or chemical analysis. In one instance (Ref. 18), foundries were qualified by testing integrally cast test bars, two to a mold, and the casting from the first three lots. An example of correlation between the test bars and castings made in investment molds is presented below:

	Remelt Stock	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset), ksi	Elongation, %
Casting	Bar	199	176	12
Test Bar	Bar	200	178	11
Casting	Shot	188	170	2
Test Bar	Shot	193	170	3

The data show that a correlation does exist between the test bar and the casting and that shot is an unsatisfactory remelt stock as evidenced by the low ductility that was obtained in the casting and the test bar.

When a foundry demonstrates that satisfactory properties can be obtained in test bars and that a correlation exists between test bars and castings, then test bars are used as the basis for acceptance of castings. However, a random sample of 2 per cent of the castings is tested to validate the correlation.

14-4 PH Alloy.

General Characteristics. As shown in Table I, the composition of the 14-4 PH alloy is very similar to the 17-4 PH alloy except that the chromium content is a little lower, the copper content is a little higher, and molybdenum has been added. As a result of the content of the latter two elements, the 14-4 PH alloy has useful strength at temperatures up to 1000 F (Ref. 5) whereas the 17-4 PH alloy is restricted to 600 F (Ref. 27).

The castability and weldability of the 14-4 PH alloy were not found in the literature. It may be assumed that these characteristics are comparable with those of the 17-4 PH alloy.

Heat Treatment and Properties. The requirements for investment castings of this air-melted alloy are covered by AMS 5340 (Ref. 5) and are summarized as follows. Castings may be poured directly from a master heat or a remelt of a master heat. The size of the master heat is not to exceed 7000 pounds. Foundry returns may be used only for preparing a refined master heat.

If a homogenization heat treatment is permitted, the castings are heated for 1-1/2 hours at 2100 F and cooled to below 90 F. This is followed by a solution-anneal treatment. If the homogenization heat treatment is not permitted, the castings may be given the solution heat treatment twice. Solution annealing consists of heating at 1900 F for 1 hour per inch of cross section and cooling as required to below 90 F. A neutral or slightly reducing atmosphere should be used in the homogenizing and solution-annealing heat treatments. Castings are normally supplied in the solution-annealed condition and are to have a hardness not higher than 36 Rockwell C.

After aging at 1100 F for 4 hours and air cooling, the requirements for minimum tensile properties are as follows:

	Separately Cast Test Specimens	Specimens Cut From Castings
Tensile Strength, ksi	150	140
Yield Strength (0.2% Offset), ksi	130	130
Elongation, per cent in 4D	10	6
Reduction of Area, per cent	25	15
Hardness	$30R_C$	$30R_C$

Because the hardness may be lower after aging as compared with the hardness after solution annealing, it appears that the specified treatment results in overaging. The reason for overaging is probably to achieve improved ductility and to make the alloy more stable at elevated temperatures.

AM 355 Alloy.

General Characteristics. Like the 17-4 PH alloy, the AM 355 alloy has excellent properties for long-term applications at temperatures up to at least 700 F and for short-term applications up to at least 900 F (Ref. 7). The alloy is air melted and has good castability in sand or investment molds (Refs. 14, 32, 33). The castings are corrosion resistant and are weldable. Castings may be softened by heat treatment (as described later in this section) to make them machinable and then heat treated for hardening and strengthening.

Because the composition of the AM 355 alloy is critical, it is good practice to use certified master heats for remelting. If an indirect arc furnace is used, a cover of argon gas is used to minimize carbon pickup (Ref. 33). The charge is melted rapidly, usually deoxidized with calcium manganese silicon, and poured at about 2950 F. When casting in investment shell molds, the molds are preheated to a temperature of about 1900 F. Figure 2 shows examples of investment castings of AM 355 for use on a wing inboard panel of an airplane.

Heat Treatment. The composition of the AM 355 alloy is adjusted to produce an as-cast structure that is austenite with up to 5 per cent delta ferrite, and has a considerable amount of precipitated carbide as a result of the slow cooling of the casting in the mold. Solution annealing redissolves the carbides and stable austenite forms when the casting is cooled to room temperature. The austenite is transformed to martensite by cooling to below room temperature. Aging then tempers the martensite and precipitates carbides to achieve hardening.

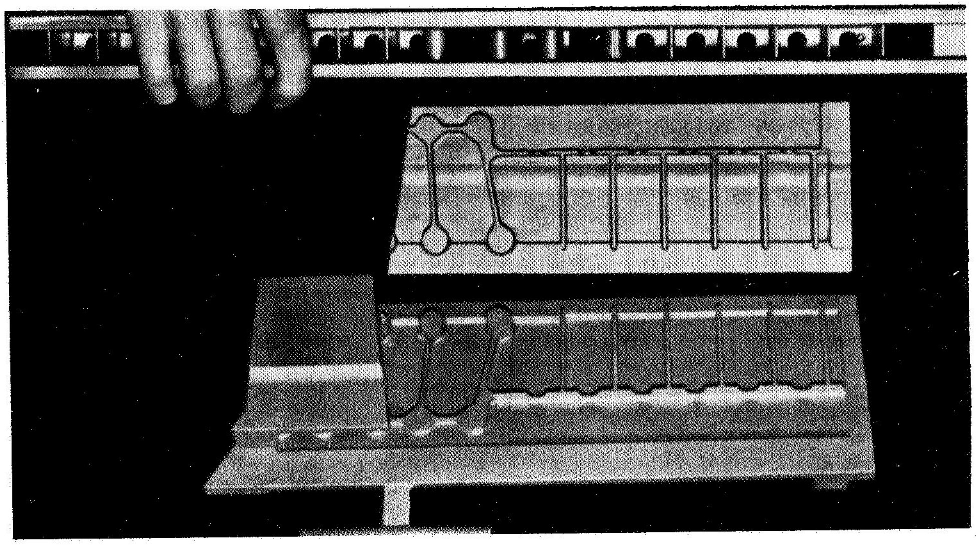

FIGURE 2. EDGE-MEMBER INVESTMENT CASTINGS OF AM 355 FOR USE ON A WING INBOARD PANEL OF THE NORTH AMERICAN B-70 AIRPLANE (REF. 7)

Note the integrally cast test bar.

The heat treatment and minimum properties are covered by AMS 5359 (Ref. 6) and AMS 5368 (Ref. 34). The information is summarized in Table V.

These two specifications indicate that castings are usually supplied in the solution-annealed condition. It is also to be noted that AMS 5368 requires the nitrogen content for investment castings to be in the range of 0.05 to 0.13 per cent. The tighter range of 0.05 to 0.11 per cent given in Table I is required for sand castings by AMS 5359.

Effect of Practice on Properties. High pouring temperatures and slow cooling of the castings during solidification promote the formation of delta ferrite. The influence of the delta ferrite on the properties of the casting has not yet been well defined.

Carbon and nitrogen are very effective in stabilizing the austenite. Hardening of the alloy is dependent upon the formation of martensite after solution annealing and prior to aging, and is also dependent upon precipitation of carbides upon aging. Therefore, control of the carbon and nitrogen content is very important. For a given heat-treatment cycle, the strength is higher and the ductility is lower as the content of carbon plus nitrogen is increased. Optimum properties are

TABLE V. SUMMARY OF AM 355 ALLOY AEROSPACE MATERIAL SPECIFICATIONS

Aerospace Material Specification	5359	5368
Type of Mold	Sand	Investment
Austenite Conditioning Treatment	200 F for 1 hour per inch of section, rapid air cool if under 1 inch, oil or water quench if over 1 inch(a).	1900 F for 1 hour per inch of section, rapid air cool if under 1 inch, oil or water quench if over 1 inch.
Maximum Hardness	$39R_C$	$39R_C$
Solution Anneal	1725 to 1850 F for 1 hour per inch of section, water quench, cool to at least -100 F for 3 hours minimum, warm in air to room temperature.	1725 F to 1850 F for 1 hour per inch of section, water quench, cool to at least -100 F for 3 hours minimum, warm in air to room temperature.
Aging Treatment	850 F for 3 hours minimum, air cool	850 F for 3 hours minimum, air cool
Properties:(b)		
Tensile Strength, ksi	200	200
Yield Strength (0.2 % Offset), ksi	150	150
Elongation, per cent in 4D	8	8
Minimum Hardness	$40R_C$	$40R_C$
Maximum Size of Master Heat, lb	5000(c)	7000(d)

(a) If specified by the customer, this treatment may be followed by heating at 1400 F for 3 hours, air cooling to room temperature or lower temperature, reheating at 1050 F for 3 hours, and air cooling.

(b) For the investment castings, the tensile specimen is to have standard proportions with a 0.25-inch reduced section. For the sand castings, if the cast-to-size tensile specimens are not permitted, the specimens are to be machined from standard keel-block castings. For investment or sand castings, if the separately cast tensile specimens fail to meet requirements, specimens may be machined from castings.

(c) No restriction specified on the preparation of the melt other than it is a single charge not exceeding 5000 pounds.

(d) Castings may be poured directly from a master heat or from a remelt of a master heat. Foundry returns may be used only in the preparation of a refined master heat.

obtainable when the carbon-plus-nitrogen content is in the range of about 0.18 to 0.24 per cent (Ref. 33). If the content is too high, an appreciable amount of the austenite will resist transformation to martensite after solution annealing. Although a small amount of retained austenite (5 to 20 per cent) is not detrimental to the properties, a large amount results in poor ductility. The retained austenite is stable during aging treatments or subsequent thermal cycling even if the casting is under stressed conditions.

Some segregation occurs as a result of coring during solidification of the castings. Coring is the presence of a variable composition between the center and surface of dendrite arms or grains. Homogenization treatment at 2000 F prior to solution annealing and aging does not improve the properties appreciably. Apparently the benefit of high-temperature homogenization of the structure is offset by the coarse austenitic grains that are formed. The properties may also suffer as a result of the surface of the casting becoming damaged by pickup of carbon or nitrogen. These elements stabilize the austenite excessively so that the response to heat treatment is poor. To avoid this type of damage during treatments at high temperature, good control must be maintained over the atmosphere that is being used. Adequate control of the atmosphere is difficult and costly.

Fortunately homogenization can be achieved by "equalizing" the castings for several hours in the range of 1375 F to 1475 F prior to solution annealing and aging. A typical heat-treating schedule is as follows (Ref. 33):

- Equalize at 1475 F for 3 hours and air cool
- Solution anneal at 1750 F for 1/2 hour and oil quench
- Cool to -100 F for 3 hours for maximum transformation to martensite
- Age at 1000 F for 3 hours and air cool.

The above heat-treating schedule is conducted in air. Such a schedule produces reliable minimum yield strength of 145,000 psi and elongation of 10 per cent. An extension of the homogenization time at 1475 F to 16 hours has been shown to be detrimental to ductility. Solution annealing at higher temperatures coarsened the grains and stabilized the austenite with the result that response to hardening was diminished and poorer properties were obtained. Aging at 850 F as compared with 1000 F gave the following average tensile properties (Ref. 33):

Aging Temperature, F	Ultimate Tensile Strength, ksi	0.2 Per Cent Yield Strength, ksi	Elongation in 1 Inch, %	Reduction in Area, %	Hardness, RC	Carbon + Nitrogen, %
850	227	160	12	19	51	0.20
850	221	166	14	34	48	0.17
1000	168	150	17	47	40	0.17

A comparison of the data for the bars aged at 850 F shows the potent effect that carbon and nitrogen have on the properties. Aging at 1000 F gave a hardness ranging from 38 to 41 Rockwell C. In this range it was possible to do finish machining on the casting.

If machining is difficult or extensive, the casting can be heat treated to a hardness of 35 Rockwell C by aging for 3 hours at 1050 to 1100 F immediately after the equalizing treatment (Ref. 33). After machining, the normal heat-treating schedule is followed without repeating the equalizing step.

The AM 355 alloy undergoes an increase in volume when the austenite transforms to martensite (Ref. 35). The volume change can be a significant amount. Therefore, final dimensional inspection should be done after the aging treatment. Also, if the volume change causes warpage, holding fixtures should be used during heat treatment (Ref. 7). Integrally cast test bars are heat treated and tensile tested to qualify the castings (Ref. 7).

A-286 Alloy.

General Characteristics. The A-286 alloy is used for structural parts requiring corrosion resistance and good strength at temperatures up to 1200 F (Ref. 8). It is vacuum-induction remelted from vacuum-melted master heats. Satisfactory crucible materials are high-purity alumina, magnesia, or zirconia that are well cured with wash heats. If the alloy were melted in air, there would be an uncontrollable loss of titanium, aluminum, and boron as well as dirty metal from the oxide and nitride inclusions of these elements. Even if the alloy were to be melted under an argon blanket, enough air could diffuse through the blanket to partially lose the effectiveness of these particular alloying elements.

Melting and Casting Practice. The data presented below compare the room-temperature tensile properties of investment castings made from air melts and vacuum melts (Ref. 36). The heat-treatment schedule for these bars was: solution anneal at 2000 F for

10 minutes and oil quench, then solution anneal at 1650 F for 2 hours and air cool rapidly, followed by aging at 1300 F for 16 hours and air cool.

	Tensile Strength, ksi	Yield Strength (0.2% Offset), ksi	Elongation, %	Reduction of Area, %
Air Melted (11 bars, 7 heats)				
Range	81.3-140.7	52.6-120.6	2-13	5-34
Average	108.3	85.8	6	14
Vacuum Melted (4 bars, 2 heats)				
Range	105.1-125.1	54.9-68.4	15-18	20-29
Average	115.3	63	17	23

It is apparent that the properties are much more consistent and the ductility is much higher for the vacuum-melted castings.

Casting must be produced in an investment mold because this is the only type of mold that is free of volatiles for casting under vacuum. The pour is usually made in molds at their preheat temperature which may range up to 1900 F. Alumina or zirconia is preferred as the facing material in the mold cavity because the melt is apt to react with a silica facing and result in surface defects in the casting (Ref. 37).

The shell investment mold imposes no restriction on size other than the available facilities for making large molds. Vacuum furnaces exist with a capacity of up to 500 pounds (Ref. 37). Investment castings have been made that weigh up to 400 pounds (Ref. 20); however, they usually weigh under 20 pounds. To prevent bulging of large molds, they are backed with refractory material during preheating and castings are poured with the backing in place.

The production cycle for vacuum casting a 12-pound charge is 6 minutes (Refs. 7, 37). The cost is about one-half more than that for melting and casting in air (Ref. 38). Although casting under vacuum is more expensive, it should minimize rejects attributed to oxide inclusions or misruns. The reason for this is that when casting under vacuum, there is no atmosphere to create oxides or develop back pressure in the mold cavity. Examples of complex shapes produced by vacuum casting are shown in Figure 3.

Heat Treatment. The A-286 alloy castings are solution annealed at 2000 F for 1 to 2 hours and oil quenched. This is followed by an intermediate solution anneal at 1650 F for 1-1/2 hours per inch

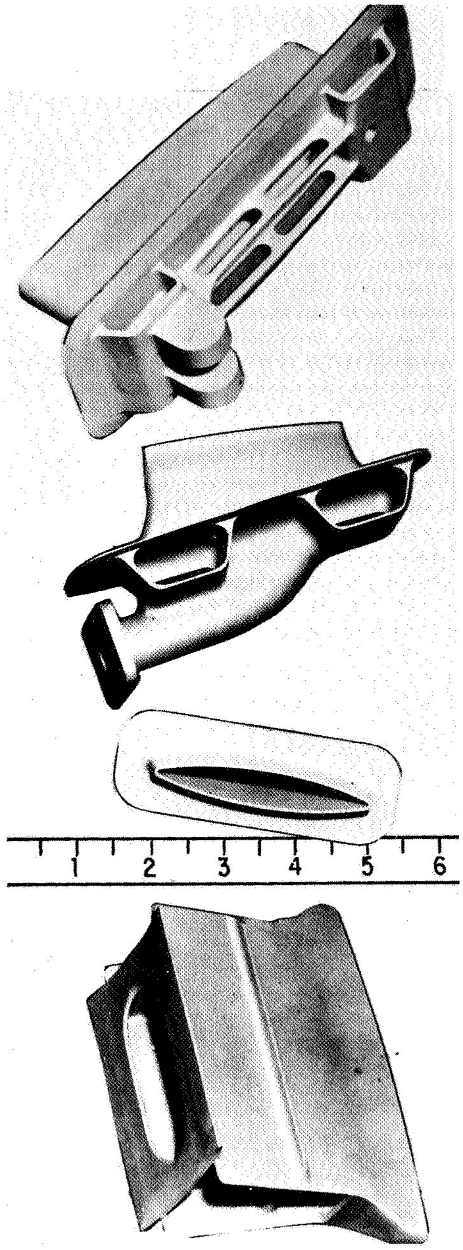

FIGURE 3. COMPLEX INVESTMENT CASTINGS OF A-286 PRODUCED BY MELTING AND CASTING UNDER VACUUM (REF. 37)

of section thickness with rapid cooling in air to room temperature. Aging is at 1325 F for 16 hours with air cooling to room temperature (Ref. 36). The alloy is austenitic and hardening occurs as a result of precipitation of Ni_3Ti phase in the austenite during aging.

Typical mechanical properties are as follows (Ref. 8):

Tensile strength, psi	106,000
Yield strength (0.2% offset), psi	85,400
Elongation, %	10.5
Reduction of area, %	17
Rupture stress at 100-hr life, psi	
At 1100 F	82,000
At 1200 F	66,000
At 1300 F	50,000.

PH-55A, PH-55B, and PH-55C Alloys (Ref. 9).

General Characteristics. These three air-melted alloys have good resistance to corrosion. They have high strength, and resist abrasion, erosion, and stress corrosion. The PH-55A alloy resists pitting corrosion better than Type 316 stainless steel. The PH-55B alloy is suitable for applications involving mechanical shock. The PH-55C alloy has better resistance to corrosion than the PH-55A alloy and slightly less resistance to mechanical shock than the PH-55A alloy. In addition to these characteristics at essentially room temperature, these three alloys are useful where resistance to softening is desired after extended exposure to temperatures up to 1400 F.

The alloys are readily cast in sand molds. Castings are weldable, but should be solution annealed after welding to obtain uniform mechanical properties and maximum corrosion resistance. Machining is done on castings in the solution-annealed condition.

Heat Treatment and Typical Properties. Heat treatment consists of a solution anneal at 2050 F for 1 hour per inch of section thickness, a water quench to room temperature, aging at 900 F for 8 hours, and furnace or air cooling. The structure after solution annealing is austenitic with discontinuous ferrite grains. After aging, a finely dispersed sigma phase is formed in the ferrite grains.

Table VI presents some mechanical properties of the three PH-55 alloys at room temperature. Tensile properties at elevated temperatures are given in Table VII. The resistance to softening after long exposures to elevated temperatures is shown in Table VIII.

TABLE VI. SOME TYPICAL ROOM-TEMPERATURE PROPERTIES OF PH-55 ALLOYS

Property	Alloy PH-55A	 PH-55B	 PH-55C
Solution-Annealed Hardness, BHN	241-285	217-235	269-331
Aged Hardness, BHN	311-388	248-302	363-477
Charpy Impact Toughness (Keyhole), ft-lb	4	35	3
Tensile Strength, psi	139,000	139,000	185,000
Yield Strength, psi	106,000	105,000	150,000
Elongation, %	8	21	2
Reduction of Area, %	6	18	2

TABLE VII. TENSILE PROPERTIES AT ELEVATED TEMPERATURES OF PH-55 ALLOYS

Property	Alloy PH-55A	 PH-55B	 PH-55C
At 900 F			
Tensile Strength, psi	102,700	83,300	108,500
Yield Strength, psi	58,000	57,500	57,500
Elongation, %	9	11	6
Reduction of Area, %	13	9	9
At 1300 F			
Tensile Strength, psi	75,400	63,500	50,100
Yield Strength, psi	36,000	32,500	33,000
Elongation, %	29	22	20
Reduction of Area, %	42	24	38

TABLE VIII. HARDNESS OF PH-55 ALLOYS AFTER EXPOSURE TO ELEVATED TEMPERATURES

Aging Treatment		Hardness, BHN		
Time, hr	Temp, F	PH-55A	PH-55B	PH-55C
8	900	352	262	387
50	900	382	293	418
50	1200	340	248	387
50	1400	402	293	430

ALLOYS NOT USED AT ELEVATED TEMPERATURES (PH-55D, CB-7Cu, AND CD-4MCu)

General Characteristics. The PH-55D, CB-7Cu, and CD-4MCu alloys are all air melted and cast in sand molds. Conventional stainless steel melting and casting practices are employed. These alloys are normally not used at elevated temperatures. The corrosion resistance of the PH-55D alloy is very similar to that of the PH-55A alloy which was discussed earlier in this report (Ref. 9).

The CB-7Cu and CD-4MCu alloys are strong and resistant to stress-corrosion attack. These characteristics make them suitable for such service as pump impellers and blades in the chemical, petroleum, food processing, metallurgical, and pulp and paper industries (Ref. 12). A structural application for the CB-7Cu alloy is shown in Figure 4. The CB-7Cu alloy is cheaper than the CD-4MCu alloy and can be hardened to over 400 Brinell by aging at low temperatures, but the CD-4MCu alloy has superior resistance to corrosion.

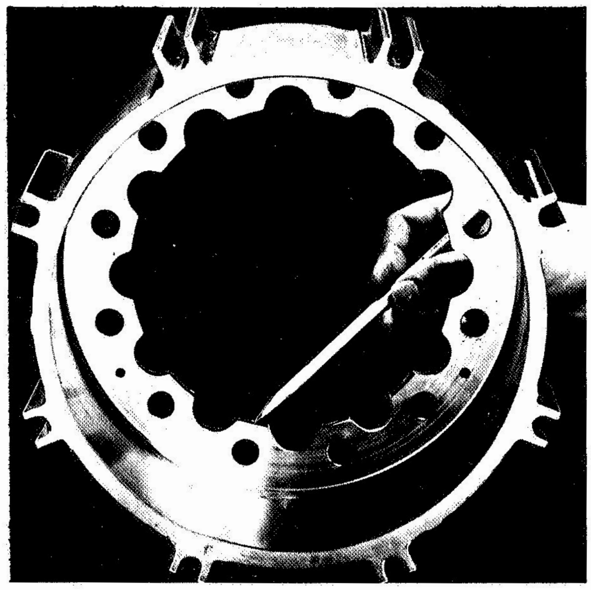

FIGURE 4. TORQUE TUBE CASTING OF CB-7Cu ALLOY FOR THE BOEING 707 BRAKE ASSEMBLY

The casting is heat treated to a tensile strength of 162,000 psi and is subject to landing impact and 15,000 ft-lb torque.

Courtesy of Alloy Casting Institute.

Heat Treatment and Typical Properties. The PH-55D alloy is solution annealed at 2050 F (Ref. 9). In this condition it has a hardness range of 286 to 418 Brinell. After aging at 900 F, the hardness is 418 to 600 Brinell which imparts high abrasion resistance. Its brittleness is similar to that of tool steel.

Heat treatment of the CB-7Cu alloy consists of solution annealing at 1900 F and air cooling or oil quenching to below 270 F to form a martensitic structure containing spheroidized ferrite. Precipitation hardening occurs upon aging. The best combination of properties is obtained by aging at 1000 F for 1 hour.

The CD-4MCu alloy is solution annealed at 2050 F for at least 2 hours, furnace cooled to 1750 F, and then oil quenched. In this condition the structure is ferritic with some retained austenite. Aging at 900 F hardens the castings by precipitation of copper and sigma phase within the ferrite.

Although the CB-7Cu and CD-4MCu alloys are weldable, the CD-4MCu alloy should be given the standard heat treatment after being welded.

Some mechanical properties of CB-7Cu and CD-4MCu alloy castings are presented in Table IX (Ref. 12).

TABLE IX. TYPICAL MECHANICAL PROPERTIES OF CAST CB-7Cu AND CD-4MCu

Alloy	Aging Temp, F	Tensile Strength, psi	Yield Strength, psi	Elongation, %	Hardness, BHN	Charpy Impact (Keyhole), ft-lb
CB-7Cu	None	140,000	113,000	14	315	-
	900	180,000	156,000	3	418	23
	1000	165,000	152,000	14	367	23
	1050	148,000	141,000	17	333	26
CD-4MCu	None	110,000	85,000	25	260	37
	900	140,000	110,000	18	305	12

POWDER METALLURGY PRODUCTS

The common grades of stainless steel are regularly produced in the form of powders and are made into pressed and sintered parts when corrosion or oxidation resistance is required. By far the most extensively used stainless powders are Types 304, 316L, and 410 although virtually any grade can be obtained in powder form if desired (Ref. 39).

Except for some experimental work with PH 15-7 Mo in making extrusion billets, no published information was found that related to the powder metallurgy of precipitation-hardenable stainless steels. However, private communications disclosed that two companies presently plan to start production of specific parts from 17-4 PH stainless steel powder (Ref. 39). Nominal compositions of the precipitation-hardenable stainless steels that have been used experimentally in powder form, i.e., Types 17-4 PH, PH 15-7 Mo, and A-286, are listed in Table X (Ref. 40).

POWDER PRODUCTION

In recent years, the dominant method for manufacturing stainless steel powders has been the atomization of the molten alloy, replacing the earlier and more costly method involving carbide sensitization of scrap, followed by electrochemical disintegration (Ref. 41). Sensitization was induced by heating in the temperature range of 1200 to 1300 F which caused precipitation of chromium carbides at grain boundaries. This depleted the areas adjacent to the grain boundaries of chromium and permitted intergranular attack and separation of the grains.

Atomization, aside from being the more economical method, can be used for production of stainless steel grades that contain strong carbide formers such as niobium, tantalum, molybdenum, and titanium. Electrochemical disintegration was not effective with stainless steels containing these elements because they inhibit carbide sensitization. Steel containing aluminum or titanium can also be atomized, thus making it possible to produce the precipitation-hardening grades that contain these elements, for example, PH 15-7 Mo and A-286. It appears, however, that production of these particular precipitation-hardenable steels is still in the development stage and that adequately low oxygen and nitrogen contents can only be obtained through use of argon as an atomizing medium. 17-4 PH stainless steel, on the other hand, contains 2.8 to 3.5 per cent copper rather than titanium or aluminum to provide precipitation hardening. Consequently, this alloy is less sensitive to oxidation during melting, atomizing, and subsequent sintering. The principal precautions required with 17-4 PH pertain to keeping the nitrogen content low. This can be done by using low-nitrogen melting stock, melting out of contact with air (slag or argon blanket), and use of dry hydrogen (-40 F dew point) or vacuum for

TABLE X. NOMINAL COMPOSITIONS OF CAST PRECIPITATION-HARDENABLE STAINLESS STEELS (REF. 40)

Type	Composition, per cent										
	Classification	Carbon[a]	Manganese	Silicon[a]	Chromium	Nitrogen	Molybdenum	Copper	Aluminum	Titanium	Other[b]
17-4 PH	Martensitic	0.06	0.070[a]	1.0	15.5-16.7	3.6-4.6	--	2.8-3.5	--	--	0.05N[a] 0.15-0.40 Cb+Ta
PH 15-7 Mo	Semiaustenitic	0.09	1.0[a]	1.0	14.0-16.0	6.5-7.75	2.0-3.0	--	1.5	--	--
A-286	Austenitic	0.08	1.0-2.0	1.0	13.5-16.0	24.0-27.0	1.0-1.5	--	0.35[a]	1.9-2.3	0.10-0.50

(a) Denotes maximum value.

(b) Balance iron.

sintering. Atomization can be accomplished with a high-pressure water jet (Ref. 39). Thus, it should be possible to produce powders from 17-4 PH alloy on a commercial basis at no greater cost than that for the more common grades of stainless steel, Types 304 and 316L. Nitrogen content must be kept low because nitrogen is a powerful austenite stabilizer and, if present in amounts greater than about 0.05 per cent it interferes with the hardening process.

The atomization process has been reviewed by Watkinson (Ref. 42) and by Parikh, et al. (Ref. 43), and therefore will not be discussed here in detail. The gas-atomization process used by Parikh for atomizing PH 15-7 Mo alloy is illustrated in Figure 5. Particle size of the powder and chemical compositions at various stages of processing are shown in Table XI. These data show that there was appreciable oxygen pickup during atomization and a still further increase during sintering in argon. The nitrogen content increased appreciably during melting and there was a further increase during atomization. It is interesting

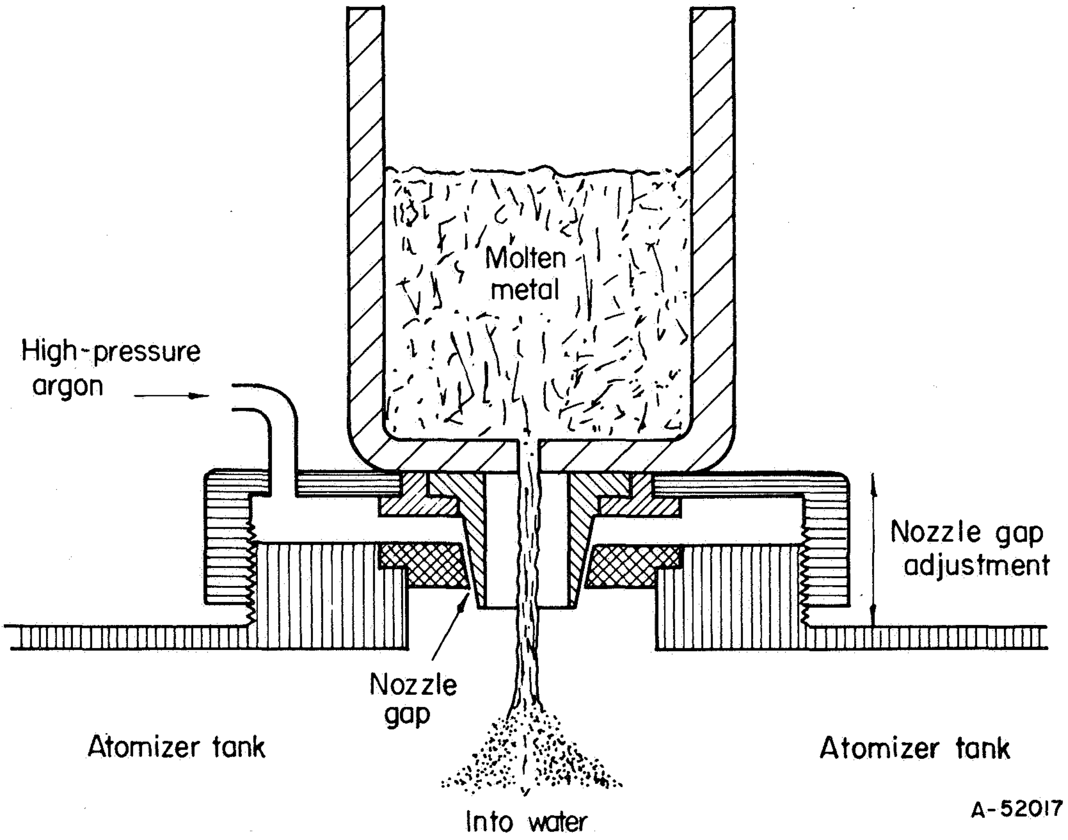

FIGURE 5. GAS-ATOMIZATION TECHNIQUE (REF. 43)

TABLE XI. INFLUENCE OF MELTING, ATOMIZATION, AND SINTERING ON CHEMICAL COMPOSITION OF PH 15-7 Mo ALLOY (REF. 44)

Alloy	Composition, per cent									
	Carbon	Manganese	Silicon	Chromium	Nickel	Molybdenum	Aluminum	Oxygen	Nitrogen	Sulfur
Specification	0.09 max	1.00 max	1.00 max	14.00/ 16.00	6.50/ 7.75	2.00/ 3.00	0.75/ 1.50	-- --	-- --	0.030 max
Melt Stock	0.07	<0.20	0.19	15.70	7.00	2.55	1.05	0.004	0.005	0.030
As Melted	0.03	--	--	15.64	7.41	2.65	0.86	0.022	0.065	--
-80 Mesh Powder(a)	0.02	--	--	15.54	7.40	2.70	0.76	0.149	0.085	--
As Sintered(b)	0.02	--	--	15.52	7.54	2.00	0.80	0.200	0.004	--

(a)

Mesh	Per Cent
-80 + 140	32.0
-140 + 200	22.0
-200 + 270	20.2
-270 + 325	6.3
-325	19.5

Apparent density: 3.93 g/cc

Flowability (Hall): 19.2 sec

(b) In argon.

to note that most of the nitrogen was eliminated during sintering in argon. More careful melting and atomization practices probably would yield lower gas contents in the powder.

That PH 15-7 Mo stainless is sensitive to nitrogen content was shown by the work of Toaz, et al. (Ref. 45). For making experimental extrusion billets of this alloy, powder was prepared by atomization with nitrogen. After pressing, sintering, hot extruding 16 to 1, and heat treating, it was found that the specimens did not harden satisfactorily. Examination of the microstructures showed them to be essentially austenitic. Nitrogen contents of some of the specimens and the hardnesses attained are shown in Table XII. It was concluded that the poor heat-treatment response resulted from the high nitrogen levels. Metallurgists at Armco Steel Corporation suggested that the nitrogen should not exceed 0.02 per cent and that the total nitrogen plus carbon content should not exceed 0.09 per cent for best results.

TABLE XII. GAS ANALYSES AND HARDNESSES OF PH 15-7 Mo ALLOY IN THE RH 950 CONDITION(a) (REF. 45)

		Analysis, per cent			
Extrusion	Type	Nitrogen	Carbon	N_2 + C	R_C Hardness(b)
177	Powder	0.098	0.030	0.128	18
178	Powder	0.11	0.036	0.146	18
179	Powder	0.060	0.060	0.120	25
180	Powder	0.062	0.028	0.090	36
191	Wrought	0.012	0.075	0.087	48
182	Wrought	0.020	0.075	0.095	47

(a) Solution treat 1950 F ± 25 F, 1 hr, water quench*; Austenite Condition 1750 F ± 15 F, 10 min, air cool to room temperature*; Transform -100 F ± 10 F, 8 hr. Precipitation harden 950 F ± 10 F, 1 hr, air cool to room temperature*.

*Performed in dry-argon atmosphere.

(b) Grain size was ASTM 8.

PH 15-7 Mo powder has been made in pilot quantities with less than 0.05 per cent oxygen and about 0.002 per cent nitrogen (Ref. 43). Melting was done under an argon blanket and atomization was carried out with a high-pressure jet of argon.

Although no data are available to support this contention, it would seem that A-286 stainless steel should not be sensitive to nitrogen content, since it is an austenitic grade of stainless.

Table XIII shows the characteristic chemical analyses and screen analyses obtained by a commercial powder producer for two 180-pound lots of PH 15-7 Mo powder. Lot 1 was atomized with nitrogen and Lot 2 was atomized with argon (Ref. 45). The nitrogen content for the lot made with nitrogen was reported to be 0.044 per cent. Unfortunately, no value was reported for the nitrogen in the lot atomized with argon. In both instances, the powder was essentially all minus 50 mesh.

TABLE XIII. CHEMICAL COMPOSITION AND SCREEN ANALYSIS OF TWO LOTS OF PH 15-7 Mo ALLOY POWDERS FROM A COMMERCIAL SOURCE (REF. 45)

	Analysis, per cent		
Element	Nominal(a)	Lot No. 1(b)	Lot No. 2(c)
Carbon	0.09 max	0.08	0.04
Silicon	1.00 max	0.81	0.56
Manganese	1.00 max	0.76	0.62
Sulfur	0.03 max	0.019	0.020
Phosphorus	0.04 max	0.017	0.019
Chromium	14.0-16.0	14.82	15.13
Nickel	6.5-7.75	7.15	7.39
Molybdenum	2.0-3.0	2.53	2.62
Iron	Balance	Balance	Balance
Aluminum	0.75-1.5	1.27	1.43
Nitrogen	--	0.044	--
Mesh Size			
+50		1.8	2.9
-50 to +100		16.1	25.9
-100 to +140		20.3	23.8
-140 to +200		29.1	23.0
-200 to +325		30.7	21.1
-325		2.0	3.3

(a) For vacuum melted, wrought, or cast material.

(b) Atomized in N_2.

(c) Atomized in A.

METHODS OF CONSOLIDATION

Cold-Die Pressing. The conventional method of consolidating metal powders involves cold-die pressing, sintering, and, if high densities are required, repressing and resintering. Little specific information is available about the processing characteristics of the precipitation-hardenable stainless steels. It is known that the powders tend to be harder than pure-metal powders, even harder than conventional stainless steel powders (Ref. 39). This characteristic makes it necessary to use high compacting pressures to achieve a given green density or green strength. Figure 6 shows the density-pressure relationship for three particle size ranges of PH 15-7 Mo powder (Ref. 44). The data show slightly higher compactibility for the finer powders with 75 per cent of theoretical density being achieved at pressures of about 20 tsi. Data for minus 80 mesh powder (Ref. 43) obtained in the same study are plotted in Figure 7 along with data for minus 80 mesh Udimet 700 and Inconel 713C powders. These data indicated 75 per cent of full density resulted for the PH 15-7 Mo powder at a pressure of about 35 tsi. In these compactibility tests, the dies were 3/8 inch in diameter with the compacts being about 1/2 inch high. The die walls were lubricated with a thin film of stearic acid to facilitate ejection of the green compacts. The high compacting pressures and abrasiveness of the powders would make the use of carbide dies important for quantity production of parts. Lubricants, such as stearic acid or zinc stearate, are normally used with stainless steel powders and probably could be used with the precipitation-hardenable grades if care is used to eliminate the lubricant, preferably in a chamber separate from the sintering furnace.

Unfortunately no green strength data have been reported for precipitation-hardenable grades of stainless. Ordinarily there is no problem in handling cold-pressed metal compacts at densities of 75 per cent. Pressed at 30 tsi, molding grades of Type 316 stainless steel powder yield a density of about 6.1 g/cc (78 per cent theoretical) with a green transverse strength of about 600 lb/sq in. (Ref. 46). Type 410 stainless steel which is a softer ferritic steel will have a density of about 6.3 g/cc (81 per cent theoretical) and a strength of about 1450 lb/sq in. when pressed under the same conditions.

Other Methods of Consolidation. Depending upon the specific part being made, a variety of methods of compaction used for other metal powders are applicable to the precipitation-hardenable grades of stainless. These methods include hot pressing, hydrostatic pressing, and possibly slip casting. Hot pressing should be applicable for larger parts where the cost and low production rate could be justified. Pressing would need to be done in inert atmosphere or vacuum to avoid contamination. Dies for hot pressing are usually machined graphite and hot graphite could contaminate stainless steel. Hydrostatic pressing appears to have its greatest application in the production of billets to be extruded or rolled. Toaz (Ref. 45) used hydrostatic compaction for making extrusion billets. Billets 3 inches in diameter by 6 inches long were obtained with a hydrostatic pressure of 40,000 psi. Green

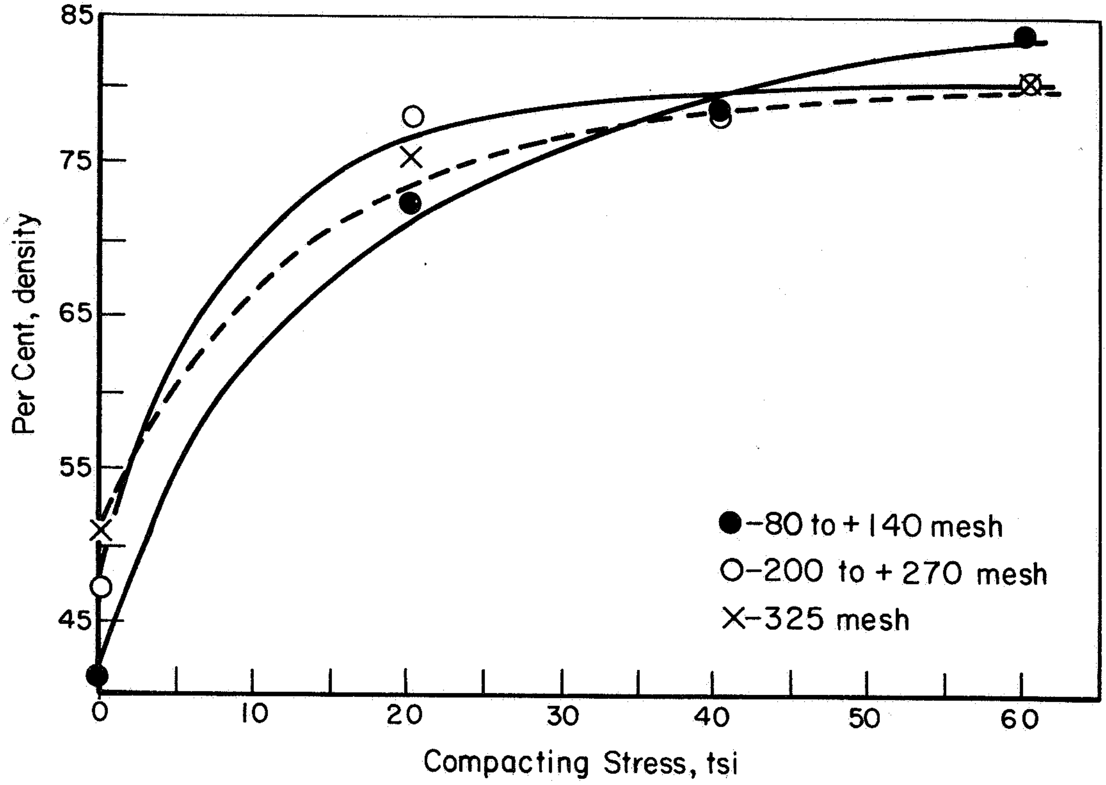

FIGURE 6. COMPACTIBILITY TESTS ON PH 15-7 Mo POWDERS (REF. 44)

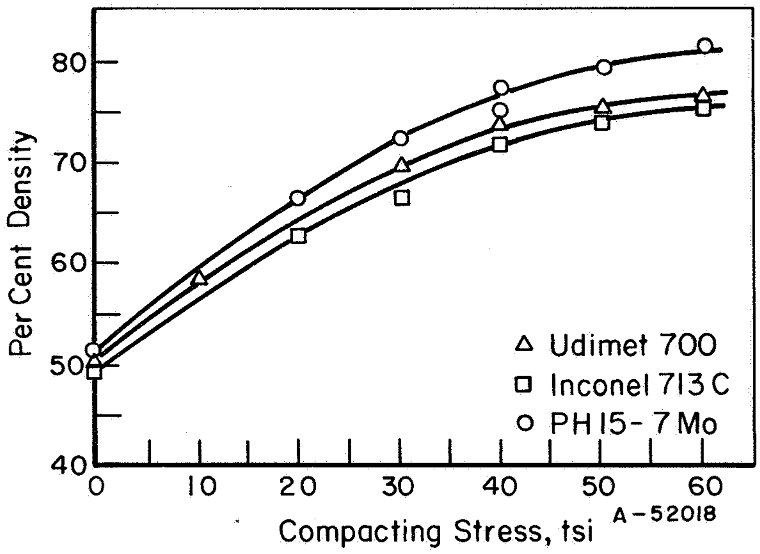

FIGURE 7. COMPACTIBILITY OF MINUS 80 MESH POWDERS; PH 15-7 Mo COMPARED WITH TWO OTHER ALLOYS (REF. 43)

densities were 65.9 to 66.8 per cent of the theoretical density based on a wrought density of 7.80 g/cc.

Slip casting offers considerable promise as a method for forming certain thin-walled stainless steel parts including 17-4 PH stainless. It is not known whether or not other grades of precipitation-hardenable stainless would be contaminated excessively by the deflocculants (usually ammonium or sodium alginates) employed in forming the slip. Reference 47 presents a description of the slip-casting process. Slip-cast Type 316 stainless powder was sintered to a density of 7.87 g/cc and had a strength of 70,000 to 80,000 psi with 55 to 57 per cent elongation. Unfortunately, the sintering conditions were not reported. Figure 8 illustrates the steps required for making parts by slip casting (Ref. 45). An attempt was made to slip cast and sinter A-286 powder on a recent Air Force project (Ref. 48). The alloy was atomized with argon and the resulting powder contained an excessive amount of oxygen. An indication was obtained that even with high oxygen contents, addition of 0.3 per cent ZrB_2 to the powder improved its sinterability.

SINTERING

Sintering procedures for the common grades of stainless steel are well developed. There are several references that describe the requirements for furnace-design atmospheres and temperatures (Refs. 49-51). However, there is no published information specifically covering the precipitation-hardenable stainless steels. It seems probable that 17-4 PH stainless can be handled in the same manner as other grades of stainless; that is, sinter either in hydrogen with dew point less than -40 F or in a vacuum of about 10^{-5} torr. Dissociated ammonia is used frequently for the common grades of stainless because of its lower cost, possibly at some sacrifice in quality of the parts. Dissociated ammonia is not likely to be satisfactory for either 17-4 PH or PH 15-7 Mo because of the danger of nitrogen pickup. Types 316, 304, and 347 stainless steels are normally sintered at 2200 to 2300 F in pucker-type or hump-type muffle furnaces since hydrogen is lighter than air and these furnaces have a raised hot zone it is easier to maintain a protective atmosphere. Production rates can run up to 1000 lb/hr in the larger sizes of furnaces (Ref. 51).

The oxides on PH 15-7 Mo steel powders may cause difficulty in sintering. In the work reported by Farrel (Ref. 43), sintering and densification proceeded for Udimet 700 in vacuum at about 2350 F, at about 2450 F for Inco 713C, but a temperature of 2700 F was required to produce much sintering with PH 15-7 Mo, both in argon and in

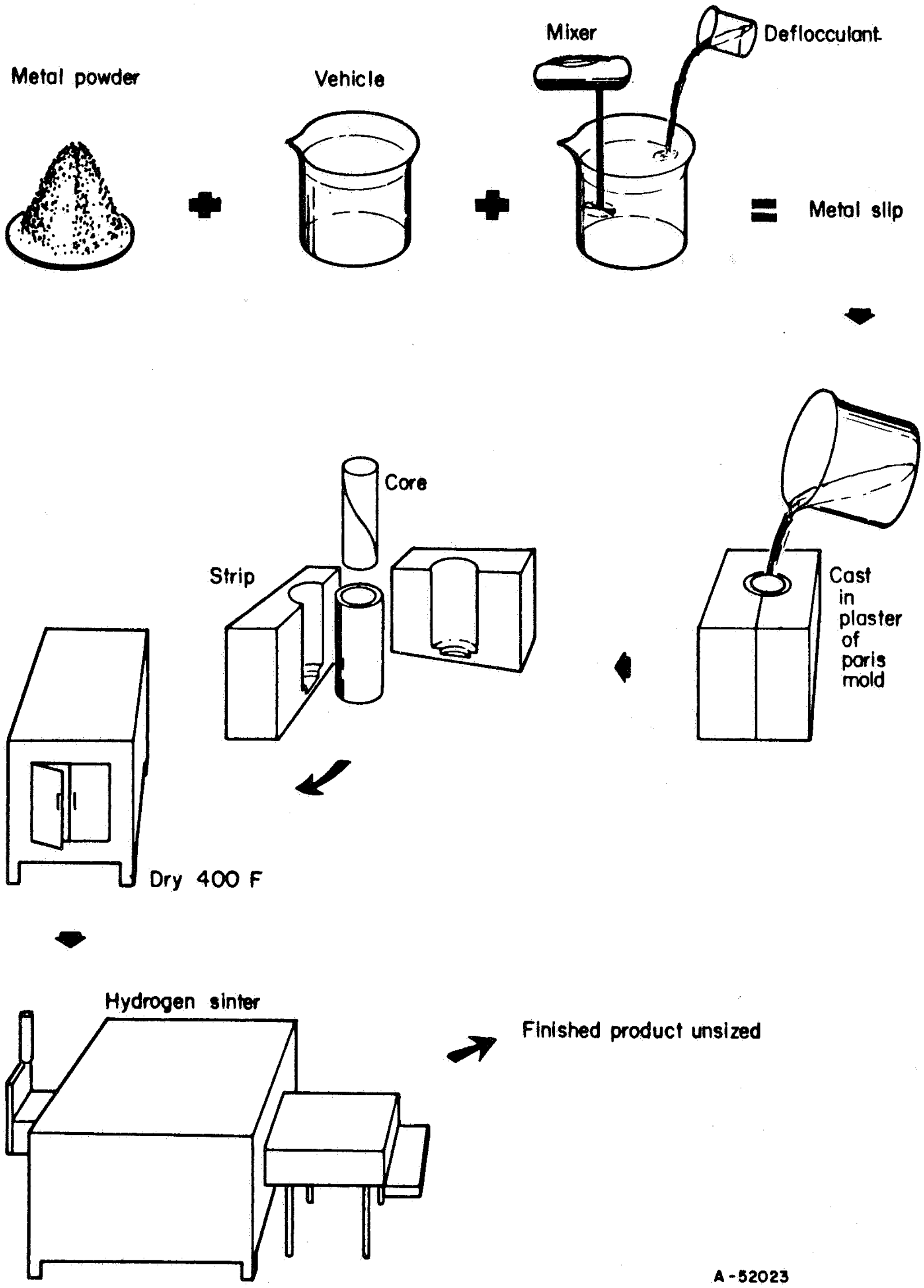

FIGURE 8. SLIP CASTING (REF. 45)

vacuum. These results are plotted in Figure 9. Figure 10 shows the effects of time at temperature on density of PH 15-7 Mo compacts.

PROPERTIES

There is little, if any, information available on the properties of pressed and sintered precipitation-hardenable stainless steels. Information available on sintered and extruded materials suggests that a fine-grained product can be produced that would approach the mechanical properties of cast and wrought material (Ref. 44). There are reasons to believe that pressed and sintered 17-4 PH stainless steel powders can have good corrosion resistance combined with greater hardness and strength than those of Type 316 or Type 410 grades.

CONCLUSIONS AND RECOMMENDATIONS

CASTING

The foundry technology for producing castings from air-melted or vacuum-melted precipitation-hardenable stainless steel 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 probably 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

The same basic production advantages characteristic of fabrication by the consolidation and shaping of metal powders in general, are applicable to powders of precipitation-hardenable stainless steel compositions. Some problems, however, are inherent in these hardenable compositions.

Currently, the greatest problem lies in the difficulty in obtaining high-quality powder. Some of the precipitation-hardenable powders are of such a composition as to be readily contaminated during production with nitrogen or oxygen. This is notably the case for compositions containing the more reactive elements, aluminum and/or

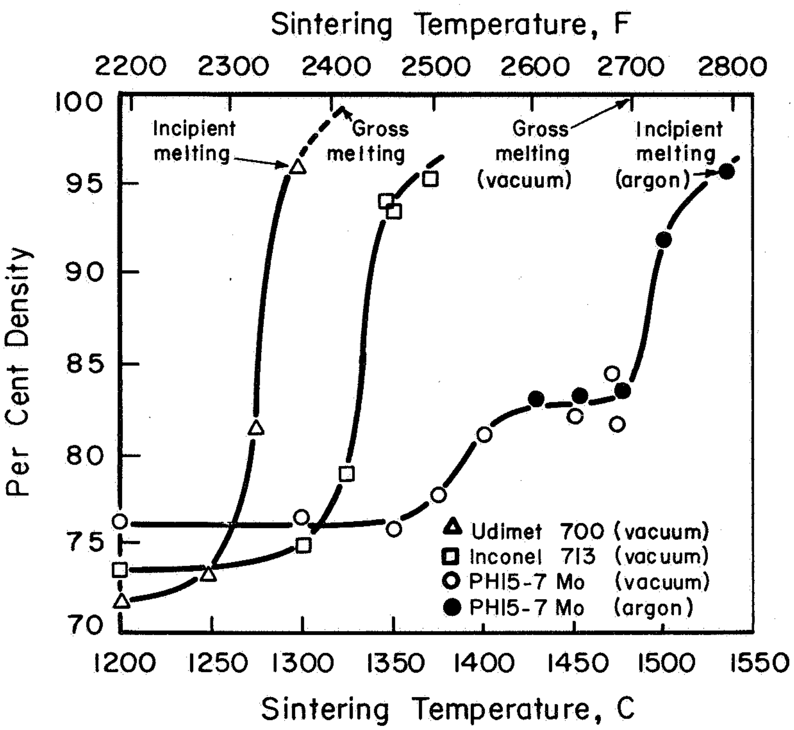

FIGURE 9. INFLUENCE OF SINTERING TEMPERATURE ON THE DENSITIES OF 1-INCH-DIAMETER COMPACTS HELD AT TEMPERATURE IN VACUUM (~0.1μ) OR IN ARGON FOR 1 HOUR (REF. 43)

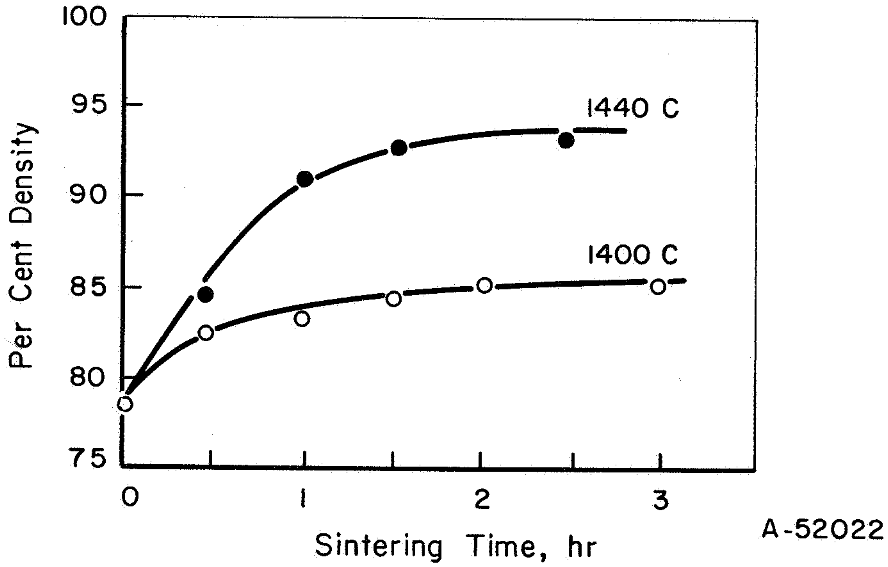

FIGURE 10. EFFECT OF SINTERING TIME IN ARGON AT TEMPERATURE ON THE DENSITY OF PH 15-7 Mo POWDER COMPACTS (REF. 43)

titanium. Therefore, improved methods are needed for making the powders without excessive contamination.

A second general problem area is concerned with sintering conditions. Sintering procedures have not been thoroughly investigated. Because some of the precipitation-hardenable alloys contain the reactive additives, aluminum and titanium, very low dew points seem desirable to avoid oxidation of these elements. Furthermore, dissociated ammonia is frequently used for sintering powders of the common grades of stainless steels. Dissociated ammonia does not appear satisfactory for sintering the precipitation-hardenable stainless steel powders because of nitrogen contamination and associated loss of heat-treating response. Vacuum may also be used although this is less economical in general. There has been at least one investigation in which a precipitation-hardenable stainless powder could not be sintered in vacuum, but was sintered with partial success in argon. The problem then is the specification of sintering atmospheres for specific steel compositions, since the various alloys will not all respond in the same manner to a single furnace condition. Sintering-atmosphere studies should include thermodynamic studies, sintering schedules, and sintering equipment.

Powder metallurgy of precipitation-hardenable stainless steels would benefit from studies of mechanical properties, corrosion tests, and in-service studies on finished hardware.

REFERENCES

1. "Armco Precipitation Hardening Stainless Steels – Casting of Armco 17-4 PH", Armco Product Data S-13, 4 pp (RSIC 0056).

2. Dvorak, R. J., and Fritz, J. C., "How to Process 17-4 PH Stainless Steel Castings", Metal Progress, 174-179 (August, 1964) (RSIC 0039).

3. Cole, S. W., "Heat Treatment for Cast 17-4 PH", Industrial Heating, 29 (10), 1856-1860 (October, 1962) (RSIC 0041).

4. A Practical Guide to Investment Casting, Pamphlet, Arwood Corporation, Brooklyn, N. Y., 47 pp (1962) (RSIC 0038).

5. "AMS 5340, Steel Castings, Investment, Corrosion Resistant, 14Cr-4.2Ni-2.3Mo-3.2Cu-0.25Cb + Ta", Society of Automotive Engineers, Inc., New York, N. Y., 4 pp (January 31, 1964) (RSIC 0623).

6. "AMS 5359, Steel Castings, Sand, Corrosion, and Heat Resistant, 15Cr-4Ni-2.3Mo-0.1N", Society of Automotive Engineers, Inc., New York, N. Y., 4 pp (January 15, 1961) (RSIC 0624).

7. Wilcox, R. J., and Martini, R. W., "Investment Castings for Advanced Aircraft", Metal Progress, 82 (3) 90-96 (September, 1962) (RSIC 0093).

8. "Vacuum Melted Investment Casting Alloys", Precision Metal Molding, 22, 57-58 (September, 1964) (RSIC 0062).

9. Mott, N. S., "Four New Stainless Alloys", The Iron Age, 183 (16), 118-21 (April 16, 1959) (RSIC 0054).

10. "Cooper PH-55A (Precipitation-Hardenable Stainless Steel) SS-109", Engineering Alloys Digest, Inc., Upper Montclair, N. J., Alloy Digest, 2 pp (November, 1960) (RSIC 0151).

11. "Copper PH-55B (Precipitation-Hardening Stainless Steel SS-117", Engineering Alloys Digest, Inc., Upper Montclair, N. J., Alloy Digest, 2 pp (May, 1961) (RSIC 0152).

12. "Stainless Casting Alloys", Product Engineering, 67-71 (July 6, 1964) (RSIC 0089).

13. "Armco Precipitation Hardening Stainless Steels – Armco 17-4 PH Bar and Wire", Armco Product Data Bulletin, 10 pp (April 1, 1957) (RSIC 0052).

14. "Investment Castable Air Melted Alloys", Precision Metal Molding, 22 (149) 31-32 (April, 1964) (RSIC 0087).

15. Boynton, Kenneth G., "Castability Ratings for Investment-Casting Alloys", American Machinist, 108 (15), 95-97 (July 20, 1964) (RSIC 0090).

16. "Problems in Designing Castings With Thin Sections", Metal Progress, 82 (5) 84-88 (November, 1962) (RSIC 0049).

17. Ekey, D. C., and Black, E. V., "17-4 PH Castings – Strong and Corrosion Resistant", Materials in Design Engineering, 46, 105-107 (July, 1957) (RSIC 0055).

18. Roser, W. R., and Kuschell, K. E., "High-Quality Aircraft Castings", Foundry, 88, 82-85 (April, 1960) (RSIC 0044).

19. Roser, W. R., "High Strength Structural Steel Castings for Aerospace Applications", Modern Castings, 40, 109-12 (August, 1961) (RSIC 0040).

20. "A Designers' Guide to Investment Casting. I, The Steps in Making Investment Castings", Precision Metal Molding, 21, 45-46 (January, 1963) (RSIC 0092).

21. Peckner, D., "Guide to Ferrous Castings", Materials in Design Engineering, 127-42 (October, 1960) (RSIC 0153).

22. "Try This Method for Fast Casting", Canadian Metalworking, 25 (9) 37-38 (September, 1962) (RSIC 0075).

23. Flinn, R. A., "Casting, Chapter 5", Nonferrous Metals Book Issue, Machine Design, 35 (22) 91-95 (September 19, 1963) (RSIC 0131).

24. James, W. P., "Heat Treatment of Cast Stainless Steels, Part 2", Precision Metal Molding, 19, 39-41 (January, 1961) (RSIC 0047).

25. "AMS 5342, Steel Castings, Investment, Corrosion Resistant, 16Cr-4.0Ni-3.1Cu, 130,000 psi, Premium Quality", Society of Automotive Engineers, Inc., New York, N. Y., 3 pp (July 15, 1963) (RSIC 0621).

26. "AMS 5343, Steel Castings, Investment, Corrosion Resistant, 16Cr-4.0Ni-3.1Cu, 150,000 psi, Premium Quality", Society of Automotive Engineers, Inc., New York, N. Y., 3 pp (July 15, 1963) (RSIC 0620).

27. "AMS 5344, Steel Castings, Investment, Corrosion Resistant, 16Cr-4.0Ni-3.1Cu, 180,000 psi Premium Quality", Society of Automotive Engineers, Inc., New York, N. Y., 3 pp (July 15, 1963) (RSIC 0622).

28. "AMS 5355A, Steel Castings, Investment, Corrosion Resistant, 16Cr-4Ni-3.1Cu", Society of Automotive Engineers, Inc., New York, N. Y., 3 pp (June 30, 1960) (RSIC 0619).

29. "AMS 5398A, Steel Castings, Sand and Centrifugal, Corrosion Resistant, 16Cr-4Ni-2.9Cu, Society of Automotive Engineers, Inc., New York, N. Y., 4 pp (July 15, 1961) (RSIC 0618).

30. Iverson, F. K., "The Effect of Aging Time and Temperature on Mechanical Properties of 17-4 PH", Precision Metal Molding, 17, 25-26 (June, 1959) (RSIC 0042).

31. Lampson, F. K., "Metallurgical Considerations for Precipitation Hardening Steels Up to 1200 F", SAE Transactions, 66, 565-73 (1958) (RSIC 0097).

32. James, W. P., "Heat Treatment of Cast Stainless Steels", Precision Metal Molding, 19, 56-57 (March, 1961) (RSIC 0046).

33. Waindle, R. F., "Heat Treating Alloy AM-355 for Optimum Properties", Foundry, 115-23 (February, 1962) (RSIC 0094).

34. "AMS 5368, Steel Castings, Investment, Corrosion Resistant, 15Cr-4Ni-2.3Mo-0.1N", Society of Automotive Engineers, Inc., New York, N. Y., 3 pp (January 15, 1961) (RSIC 0625).

35. Boyle, J. H., "Production of Integrally Cast Airfoil Components for Small Gas Turbine Engines", presented before American Society of Mechanical Engineers, Houston, Texas, 15 pp (March, 1962) (RSIC 0120).

36. Popp, H. G., "Materials Property Data Compilation – A-286 Forgings and Castings, Part III", General Electric Company, Fourth Quarterly Report, Contract No. AF 33(657-8017, pp C. 3. 1-C. 3. 7 (February, 1963) (RSIC 0154).

37. Rapoport, Dan, "Recent Developments in Vacuum Melting and Pouring", Foundry, 54-57 (November, 1963) (RSIC 0035).

38. Iverson, F. Kenneth, "Bright Future Seen for Vacuum Investment Castings", Iron Age, 178 (3), 120-22 (July 19, 1956) (RSIC 0114).

39. Private communication from Athan Stosuy, Hoeganaes Sponge Iron Corporation, Riverton, N. Y., September 21, 1965 (RSIC 1279).

40. James, W. P., "Heat Treatment of Cast Stainless Steels (Part 1)", Precision Metal Molding, December, 1960 (RSIC 1273).

41. Sands, R. L., and Watkinson, J. F., "Sintered Stainless Steel. I, The Influence of Alloy Composition Upon Compacting and Sintering Behavior", Powder Metallurgy, 5, p 85 (1960) (RSIC 1275).

42. Watkinson, J. F., "Atomization of Metal and Alloy Powders", Powder Metallurgy, 1/2, p 13 (1958) (RSIC 1269).

43. Farrell, K., "Sintering of Atomized Super Alloys and a Hardenable Stainless Steel", International Journal Powder Metallurgy, 1 (3), p 26 (1965) (RSIC 1276).

44. Parikh, N. M., Farrell, K., and Spachner, S. A., "Final Report on Improved Production of Powder Metallurgy Items", IIT Technical Report AFML-TR-65-103 on Air Force Contract A F 33(657)-9140, March, 1965 (RSIC 1274).

45. Toaz, M. W., Davies, G. F., and Johnson, R. D., "Production of Extrusion Billets of High Temperature Alloys by Powder Metallurgy", Clevite Final Report AMC TR 60-7-764 on Air Force Contract AF 33(600)-39327, December, 1960 (RSIC 1100).

46. Private communication from E. R. Andreotti, Burgess-Norton Manufacturing Corporation, Geneva, Illinois, September 22, 1965 (RSIC 1278).

47. Hausner, Henry H., and Poster, Arnold R., "Slip Casting Technique Widens Powder Metallurgy", Steel, February 15, 1960 (RSIC 1277).

48. Fischer, G. W., et al, "Complex Shapes by Slip Casting", General Electric Company Final Report AFM-TR-60-257 on Air Force Contract AF 33(615)-1366, August, 1965, (RSIC 1290).

49. Kalish, Herbert S., and Magza, E. N., "An Evaluation of Dissociated Ammonia and Hydrogen Atmospheres for Sintering Stainless Steels", Journal of Metals, p 304 (February, 1955) (RSIC 1271).

50. Ruediger, Bernt A., "Heat Treating and Sintering Stainless in an Atmosphere, Metal Progress, 79 (2) p 101 (February, 1961) (RSIC 1272).

51. Batten, W. L., "Developments Extend Market for Stainless Powders", The Iron Age, 186, p 64 (September 1, 1960) (RSIC 1270).

APPROVAL

SHAPING OF PRECIPITATION-HARDENING STAINLESS STEELS BY CASTING AND POWDER-METALLURGY

By J. G. Kura, V. D. Barth, and H. O. McIntire

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entire entirety, has been determined to be unclassified.

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

W. A. Wilson

W. A. WILSON
Chief, Methods Development Branch

J P Orr

J. P. ORR
Chief, Manufacturing Research and
Technology Division

for WERNER R. KUERS
Director, Manufacturing Engineering
Laboratory