RI 8556

Bureau of Mines Report of Investigations/1981

Consolidation of an Iron-Base Superalloy by Powder Metallurgy Techniques

By J. F. McIlwain and L. A. Neumeier

UNITED STATES DEPARTMENT OF THE INTERIOR

Report of Investigations 8556

Consolidation of an Iron-Base Superalloy by Powder Metallurgy Techniques

By J. F. McIlwain and L. A. Neumeier

UNITED STATES DEPARTMENT OF THE INTERIOR
James G. Watt, Secretary

BUREAU OF MINES

This publication has been cataloged as follows:

McIlwain, J F

Consolidation of an iron-base superalloy by powder metallurgy techniques.

(Report of investigations ; 8556)
Bibliography: p. 22.
Supt. of Docs. no.: I 28.23:8556.

1. Powder metallurgy. 2. Iron alloys. 3. Compacting. I. Neumeier, L. A., joint author. II. Title. III. Series: United States. Bureau of Mines. Report of investigations ; 8556.

TN23.U43 [TN697.17] 622s [672.3'7] 80-607851

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS--Continued

TABLES

CONSOLIDATION OF AN IRON-BASE SUPERALLOY BY POWDER METALLURGY TECHNIQUES

by

J. F. McIlwain[1] and L. A. Neumeier[2]

ABSTRACT

As part of its goal to minimize the requirements for critical materials, the Federal Bureau of Mines has investigated the consolidation of an iron-base superalloy (20 pct Cr, 5 pct each Ni and Mn, 1 pct each C, W, Mo, and Cb, and the balance Fe) and its modifications by powder metallurgy (P/M) techniques. Vacuum-atomized, prealloyed powder was used. Consolidation was by sintering of cold-pressed alloy powder, or by forging plus rolling or extrusion of canned powder.

Several commercial lubricants were evaluated in the pressing operation. At 50-tsi compacting pressure with up to 3 wt-pct lubricant, green strengths did not exceed 1,000 psi. Powder sintered just below the solidus temperature attained a tensile strength of 69,000 psi and 4 pct elongation. Liquid-phase sintering produced higher densities, lower tensile strengths, and nil ductility.

Canned powder was forged and rolled at 1,200° C, yielding a 100-hr rupture strength of 9,800 psi at 815° C and a room-temperature tensile strength of >130,000 psi at 6 pct elongation. Heat treatment of modified P/M iron-base alloy containing 0.63 pct C resulted in a 100-hr rupture strength of 17,000 psi. Oxidation resistance at 805° to 815° C of the forged and rolled P/M iron-base alloy was similar to that of the cast iron-base alloy and superior to conventional stainless steels.

INTRODUCTION

One aim of metallurgy research within the Bureau of Mines is the minimizing of requirements for critical metals currently used in alloys which helps to foster the overall goal of maintaining an adequate supply of minerals and metals to meet national economic and strategic needs. In particular are those metals that must be imported and whose future supply is uncertain. Chromium and cobalt are of special concern since their principal origins are in

---

[1]Metallurgist.
[2]Supervisory metallurgist.
Both authors are with Rolla Research Center, Bureau of Mines, Rolla, Mo.

politically unstable regions of Africa. Nickel is another critical metal upon which the United States depends largely on imports for its supply.

Alloys having superior elevated-temperature strength and corrosion resistance, yet lesser contents of critical elements such as chromium, cobalt, and nickel are increasingly needed for a variety of minerals-processing equipment, as well as transportation, energy conversion, and other systems. Development of iron-base alloys inherently lower in nickel, chromium, or cobalt, with higher operating temperatures, would help fulfill this need.

One such cast alloy, designated CRM-6D, has been developed by the Chrysler Corp.[3] for use in automotive gas turbines (7-8).[4] It is designed for operation at temperatures of up to 815° C, above that normally encountered by stainless steels, and in the lower range of the nickel- or cobalt-base alloys. Alloy composition consists of about 65 pct Fe, 20 pct Cr, 5 pct each Ni and Mn for austenite stabilization, plus C, W, Mo, and Cb at about 1 pct each for solid-solution and carbide strengthening. Silicon and boron are also present in small amounts as beneficial residuals. Because of its lower nickel content, absence of cobalt, and superior properties, this alloy was selected as a basis for research into means of further conserving these elements. The objective of this work was the conservation of alloy constituent metals by the overall reduction of material usage through powder metallurgy (P/M) techniques. The P/M approach inherently conserves materials lost in gating and risering systems of castings. Also, material is saved because of reduced machining often required with near-net-shape P/M parts. Related research had as its objective the reduction of chromium usage in this alloy by partial substitution of aluminum. Description of this work, which involved the preparation of powder by the mechanical alloying method (5), is beyond the scope of this report.

The present report describes procedures for the consolidation of the iron-base alloy CRM-6D and modifications of it by P/M techniques, and presents data on oxidation resistance and mechanical properties at 815° C relative to those of the cast alloy. Included for reference are data for AISI type 316 stainless steel, one of the strongest and most common of the stainless steels.

ACKNOWLEDGMENT

The authors wish to thank the Chrysler Corp. for donating the starting CRM-6D material and for helpful discussions and information regarding its work with this alloy.

---

[3]Reference to specific equipment (or trade names or manufacturers) does not imply endorsement by the Bureau of Mines.

[4]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

MATERIALS AND EXPERIMENTAL PROCEDURE

Powder Characterization

About 180 lb of CRM-6D rondelles (pellets) were supplied by Chrysler Corp. for the research. The rondelles were sent for custom vacuum-atomization, which yielded 142 lb of minus 80-mesh prealloyed powder.

Chemical analyses of the rondelles used as melt stock and the atomized powder appear in table 1. Chemical analyses of melt stock, powder, and subsequently consolidated material employed standard techniques: chromium by oxidation-reduction titration; Ni, Mn, and Mo by atomic absorption spectroscopy; W, Cb, and Si by gravimetric methods; boron by a colorimetric method; nitrogen by a volumetric method; and carbon and oxygen by Leco combustion.

TABLE 1. - Chemical composition of CRM-6D rondelles and vacuum-atomized powder, weight-percent

Element	Rondelles	Powder	Element	Rondelles	Powder
Iron..........	Balance	Balance	Molybdenum...	0.98	0.97
Chromium......	22.5	21.2	Columbium....	.93	1.23
Nickel........	5.4	5.2	Silicon......	.48	.37
Manganese.....	5.4	4.6	Boron........	.003	.003
Carbon........	1.0	1.09	Nitrogen.....	.029	.035
Tungsten......	.92	.94	Oxygen.......	NA	0.015- .032

NA Not available.

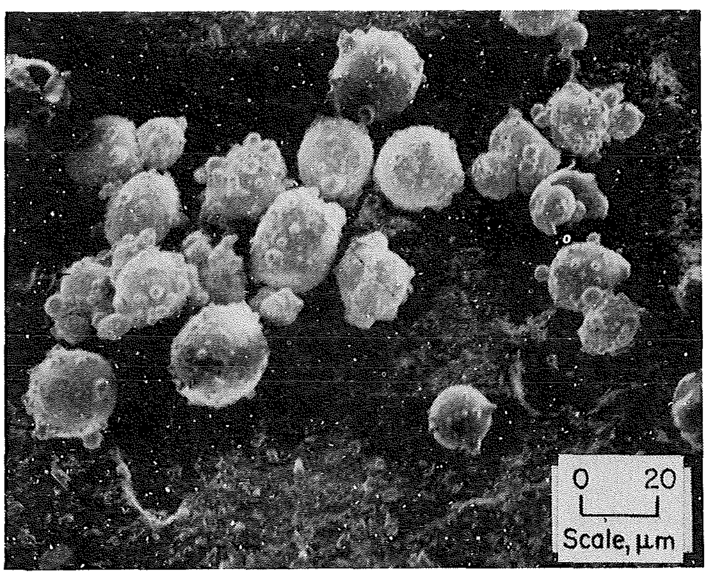

FIGURE 1. - Scanning electron micrograph of prealloyed, vacuum-atomized CRM-6D powder. Sample area represents a relatively large particle size fraction.

The sieve analysis and other physical properties of the powder are listed in table 2. Screening was by mechanical vibration followed by hand shaking. Flow rate was measured using a Hall flowmeter funnel. Apparent density was determined using a 25-cm^3 cup filled by freely flowing powder. Tap-density measurements were made by simultaneously filling and vibrating this cup. All densities are related to the value of 7.86 g/cm^3, the density of investment-cast CRM-6D. This value is subsequently referred to as the "theoretical" density, although it is an experimental, rather than a calculated quantity. Knoop microhardness measurements were made on powder samples mounted and polished for metallography. Metallographic specimens were mounted, polished, and etched according to conventional procedures. Either a glyceregia etch (25 ml glycerin, 20 ml HCl, 10 ml HNO_3, and 15 ml H_2O) or a chrome regia electroetch (25 ml HCl, 25 ml of 10 pct aqueous CrO_3, 50 ml glycerin, and 50 ml alcohol) was used. Figures 1 and 2 show, respectively, the magnified, exterior particle morphology and the interior microstructure of the atomized powder.

TABLE 2. - Properties of CRM-6D powder

(minus 80-mesh fraction)

Property	Quantity
Powder retained, by screen size, wt-pct:	
Plus 80 mesh..........................	0.1
Minus 80 plus 120 mesh................	14.4
Minus 120 plus 170 mesh...............	12.8
Minus 170 plus 230 mesh...............	19.3
Minus 230 plus 325 mesh...............	20.2
Minus 325 mesh........................	33.2
Total.............................	100.0
Flow (50 g)........................sec..	18.3
Apparent density................g/cm^3..	4.29
Tap density.....................g/cm^3..	5.11
Microhardness (Knoop, 100 g)............	337

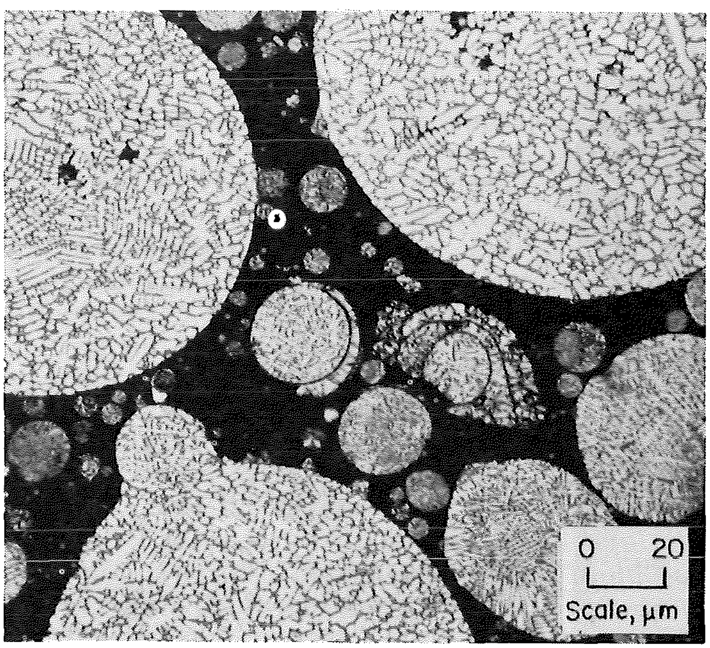

FIGURE 2. - Microstructure of atomized CRM-6D powder. Glyceregia etch.

Two quantities of lower carbon CRM powder were also prepared. The melt stock was prepared at the Rolla Research Center in the form of 50-lb ingots, which were subsequently vacuum-atomized by an outside company. Chemical analyses of these alloy powders, designated CL (low carbon) and CM (medium carbon), appear in table 3. Figure 3 shows the microstructures of the CL and CM powders. These powders were used both to prepare intermediate (0.5 to 0.9 pct) carbon alloys for this study and for other consolidation studies not reported here.

TABLE 3. - Chemical composition of lower carbon CRM prealloyed powder,[1] weight-percent

Element	CL	CM	Element	CL	CM
Iron........	Balance	Balance	Molybdenum..	0.90	0.98
Carbon......	0.02	0.51	Columbium...	.89	.60
Chromium....	21.9	21.8	Silicon.....	.85	.94
Nickel......	5.3	5.3	Boron.......	.012	.018
Manganese...	2.7	4.1	Oxygen......	.14	.09
Tungsten....	1.5	1.5	Nitrogen....	.009	.007

[1]Vacuum-atomized.

Powder Consolidation

Consolidation was performed by conventional pressing and sintering, or by hot-forging plus rolling or extrusion of canned powders. Attempts to consolidate by isostatically pressing to 60,000 psi at room temperature were unsuccessful.

Pressing and Sintering

Cylindrical compressibility compacts, rectangular green strength bars and tensile specimens were compacted on a 50-ton vertical press with double-acting dies. Die cavities were lubricated with zinc stearate in a

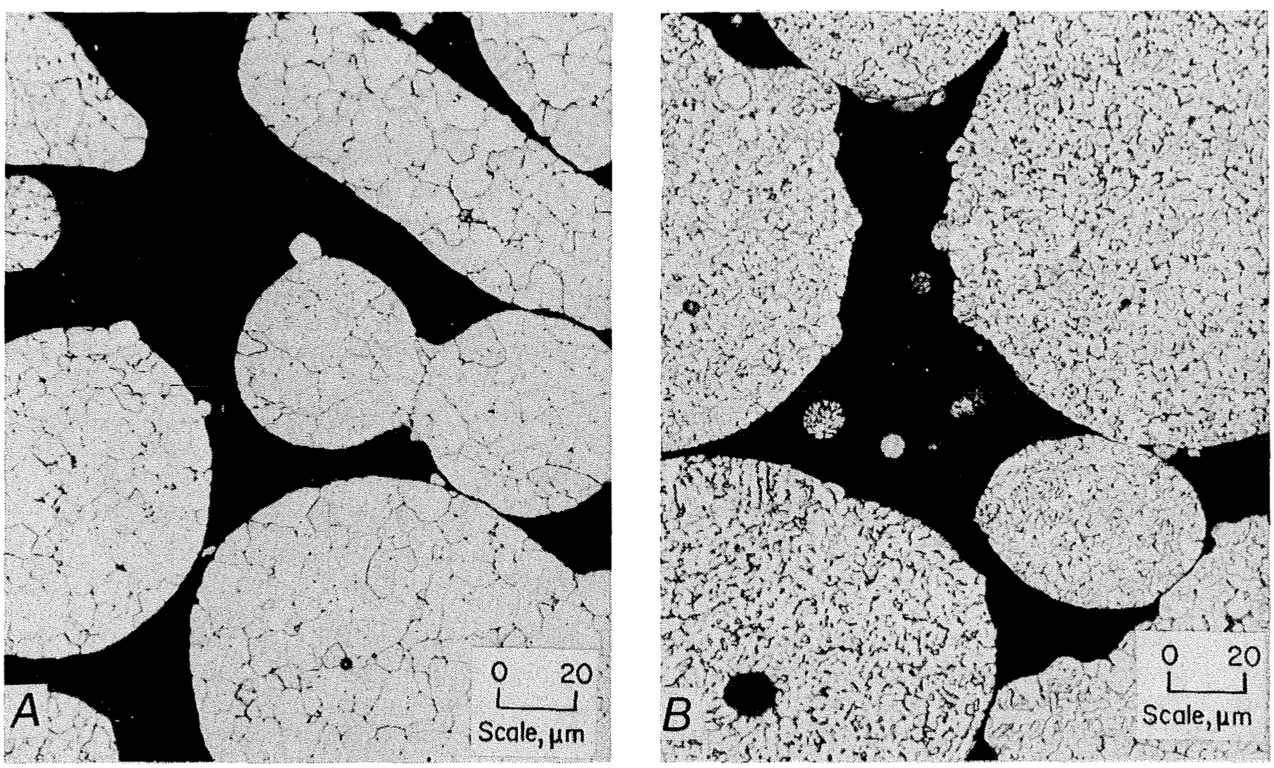

FIGURE 3. - Microstructures of atomized low-carbon (*A*) and medium-carbon (*B*) alloy powders. Glyceregia etch.

trichloroethane carrier. Several commercial lubricants were evaluated. These had been blended with the CRM powder in a double-cone blender. Green strengths of the pressed bars, measured according to ASTM Standard B312-64 (4), served to evaluate the lubricants. Green densities were calculated from specimen weight and measured volume.

Sintering was conducted in a hydrogen furnace at a gas flow rate of approximately 0.08 ft^3/min. Specimens rested on alumina plates inside iron boats. Because of relativly high dew points in the furnace (-23° to -6° C), the specimens were covered with tantalum foil under a titanium lid. Graphite powder sealed the base of the lid. The foil did not contact the specimens. No correlation between dew point and specimen oxygen content was found. Temperatures inside the cover were measured with a Pt/Pt-10 pct Rh thermocouple. The sintering was for a 1- or 2-hr duration at temperature, following a 1/2-hr lubricant burnoff period.

Extrusion

Powder was sealed into AISI type 304 stainless steel cans 1.75 in ID by 2.12 in deep with 0.07-in walls. Cans were sealed in air or under vacuum ($\leq 10^{-4}$ mm Hg) without heating. Extrusions were performed at 1,200° C at a ratio of 9.2:1, followed by light swaging at 1,200° C for straightening. The extrusions were performed at the Bureau's Albany Research Center, Albany, Oreg. Test specimens were machined from the extruded rod.

Forging and Rolling

CRM powder without lubricant was loaded into AISI type 304 stainless steel cans and vibrated to a tap density of 67 to 69 pct. The can dimensions were 2.75 in ID by 2.5 in deep with 0.063-in or 0.12-in walls. Initially, the cans were sealed by welding in air with no outgassing treatment. Subsequent data indicated that a higher density resulted from vacuum outgassing at about 350° C, then sealing under vacuum ($\leq 10^{-4}$ mm Hg).

Forging and rolling were performed on cans heated to 1,200° C. They were upset-forged to consecutive reductions-in-height of 32, 32, and 18 pct, with intermediate reheating at 1,200° C. The billets were further reduced by cross-rolling in three passes of about 19, 15, and 15 pct, again with intermediate reheats. An optical pyrometer measurement indicated a surface temperature of ~1,000° C after rolling. The rolled "pancakes" measured approximately 5 by 6 by 0.6 inch. Test specimens were machined from the pancakes. The stainless steel sheath was removed mechanically from the cut coupons.

Cast Material

Two 10-lb ingots were cast from plus 80-mesh CRM powder and solid pieces of the atomization furnace "skull." The molten alloy was cast into iron molds at 1,500° C under helium. The tapered, square-cross-section ingots measured 2 to 2.5 in wide by 6.3 in high. Tensile and oxidation specimens were machined from pieces cut parallel to the ingot axis.

Mechanical and Physical Testing

Room-temperature tensile tests were performed on the P/M specimens. The sintered specimen configuration followed ASTM Standard E8-69 (2). Forged coupons were initially machined to threaded-shoulder specimens having a 0.19-in center diameter with a 1.1-in reduced section. Subsequently, the hot-consolidated coupons, both forged and extruded, were machined to a "buttonhead" configuration, which required less machining time and facilitated mounting of specimens in the test machines. These had the same gage diameter, but with a 1.4-in reduced section. Specimen lengths and diameters before and after testing were measured with a traveling stage microscope. Tensile tests were performed on a universal tensile machine operating at a constant crosshead speed of 0.02 in/min.

Stress-rupture tests were performed on constant-load creep rupture machines at elevated temperature in still air. Tests were performed on forged or cast threaded-end specimens, and on forged or extruded buttonhead specimens of the same configurations specified above. No stress-rupture tests were performed on sintered material.

Densities were measured by water-immersion after oil-impregnation, following ASTM Standard B328-73 (3). Measurements were made on sintered specimens after mechanical testing, or on pieces cut from rolled or extruded material adjacent to where tensile coupons were sectioned.

Oxidation Testing

Cylincrical oxidation test pieces 0.3 in diam by 0.88 in long were machined from cast or hot-rolled coupons, and polished to a surface roughness of <20 μin (rms). Specimens rested in individual porcelain crucibles set in a pot furnace. Furnace temperature was measured with a Pt/Pt-10 pct Rh thermocouple suspended in a crucible at a sample position. Specimens were removed at intervals of 16, 40, or 63 hr for light brushing to remove loose scale, and were subsequently weighed.

Other

Heat treatments to effect microstructural changes were performed on glass-slurry-coated coupons in air to 1,290° C, and on uncoated coupons in helium to 1,280° C or in hydrogen to 1,315° C. Examination of microstructures was performed with an optical metallograph or a scanning electron microscope (SEM). X-ray diffraction analyses of carbides, extracted electrolytically in an HCl solution or chemically with HF, were made with the help of a Debye-Sherrer powder camera. Larger carbides were also examined in place with an SEM energy dispersive X-ray (EDX) unit.

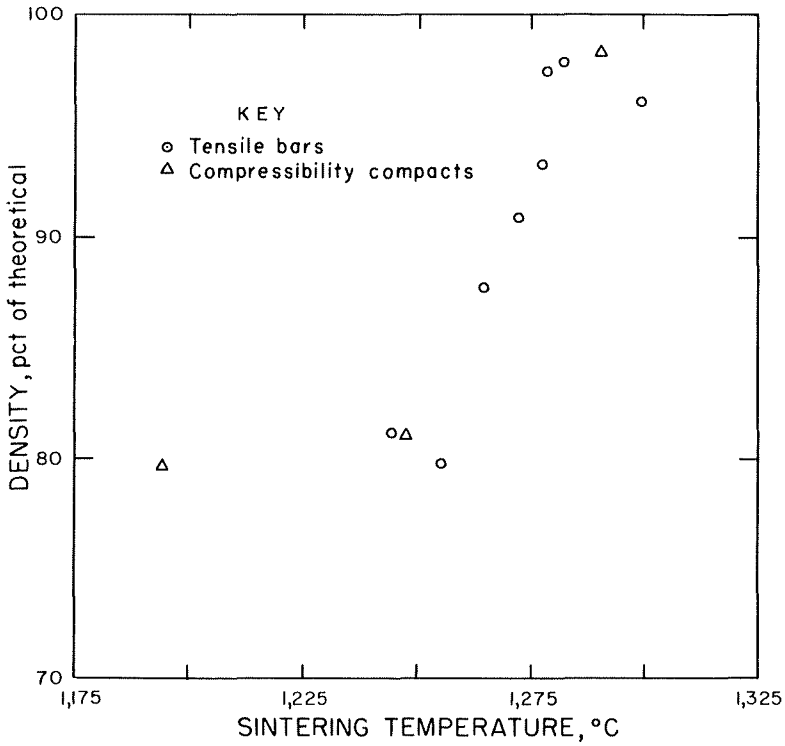

FIGURE 4. - Density of sintered compacts as a function of sintering temperature. Data are for cylindrical compressibility specimens and tensile bars, sintered for 1 hr in H_2 after a 1/2-hr binder burnoff at 450° C. Theoretical density is 7.86 g/cm^3. Starting (green) corrected densities were 78.9 and 75.3 pct for the compressibility and tensile specimens, respectively.

RESULTS

Pressed and Sintered Material

As is normally the case with highly alloyed powder, the atomized CRM powder did not cold press well. With additions of up to 3 wt-pct, none of the lubricants generated compacts with green strengths in excess of 1,000 psi. The data appear in table 4; included are the corrected densities $\rho_c = \rho_{meas}(1-f_{lubricant})$. All data are for transverse rupture bars pressed at 100,000 psi (50 tsi). The lubricants are listed in order of decreasing maximum green strength. Except for those of zinc stearate, the values shown are the maximum obtained with respect to compacting pressure. It will be noted that, in each case, the density decreases with increasing lubricant fraction.

TABLE 4. - Comparison of lubricants used with CRM-6D powder compacts[1]

Lubricant	Amount, pct	Green density (ρ_c),[2] g/cm^3	Green strength, psi
Polymist A-12.........	3.0	5.79	790
Do.................	2.0	6.09	430
Do.................	1.0	6.14	135
Acrawax C............	3.0	6.01	302
Do.................	2.0	6.15	118
Do.................	1.0	6.17	7
Nopcowax DS22.........	2.0	5.80	82
Do.................	1.0	5.91	33
Zinc stearate.........	3.0	5.97	91
Do.................	2.0	6.07	36
Do.................	1.0	([3])	([3])

[1]Compacted at 50 tsi.
[2]Corrected for lubricant additions.
[3]Did not compact.

On the basis of having maximum green strength, powder mixtures with Polymist A-12 (hereafter referred to as Polymist) were selected for the bulk of the sintering studies. Sintering trials were performed over the temperature range 1,200° to 1,300° C. Densification was minimal below 1,250° C (fig. 4), then rose rapidly, reaching 98 pct at 1,285° C. No apparent differences in sintered densities were seen between compressibility and tensile specimens, even though their respective green densities had been 79 and 75 pct, respectively. Microstructures of compacts sintered at 1,250° or at 1,275° C (fig. 5) illustrate the effect of incipient melting on the densification. Between the two temperatures, incipient melting has commenced (fig. 5B), filling the interstices with a continuous second phase. The limited sintering at 1,250° C (fig. 5A) is evident; note that the solid-phase microstructure is little changed from that of the atomized powder. Further sintering trials indicated that incipient melting of the alloy begins at ~1,265° C).

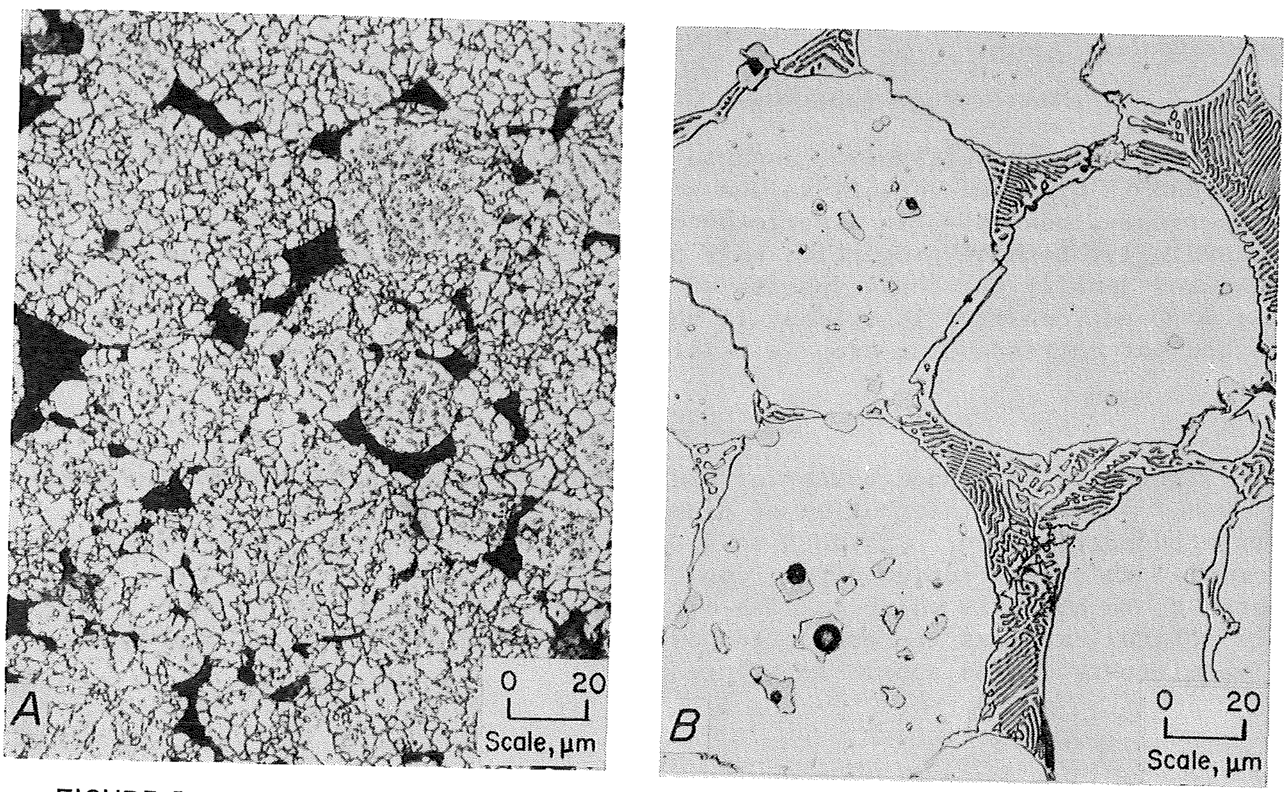

FIGURE 5. - Microstructures of compacts sintered below (*A*) and above (*B*) the solidus temperature of ~1,265° C. Chrome regia electroetch.

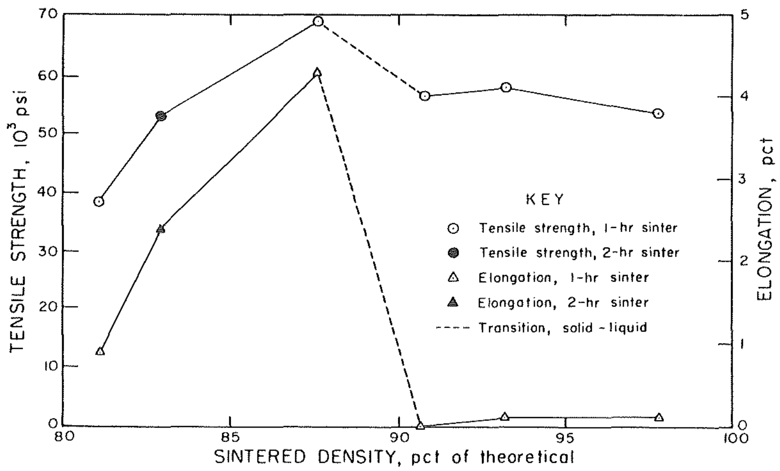

FIGURE 6. - Room-temperature tensile strength and elongation for solid-phase (<90 pct) and liquid-phase (>90 pct) sintered powder.

Powder blends with 0 to 2 pct Polymist were sintered at 1,200°, 1,250°, and 1,300° C. Maximum densities occurred at 1 to 1.5 pct Polymist for all three sintering temperatures. To optimize green strength and sintered density in subsequent work, a blend with 1.5 pct Polymist was selected for tensile strength studies.

Specimens were sintered for 1 or 2 hr at nominal specimen temperatures of 1,250° or 1,275° C; that is, within the solid- and liquid-phase regions, respectively. Because of temperature gradients in the furnace, a spread in sintered density values was obtained for the same nominal furnace temperature, resulting in a corresponding variation in tensile properties. Accordingly, tensile properties have been plotted in figure 6 as a function of measured density for these specimens. The transition to liquid-phase sintering (>90 pct density) is evident in the elongation curve; a somewhat less marked but corresponding drop in tensile strength is also observed.

Extruded Material

Extrusion of canned powder of the CRM-6D alloy produced a very fine-grain size and a denser distribution of finer precipitates that primarily occupy grain boundary sites. Grains 3 to 4 µm wide by 4 to 6 µm long were oriented parallel to the extrusion direction. The proportion of precipitate was estimated to be about 10 vol-pct. The air-sealed powder densified to 99.7 pct of theoretical, whereas the evacuated powder compacted to 99.4 pct. Representative microstructures appear in figure 7.

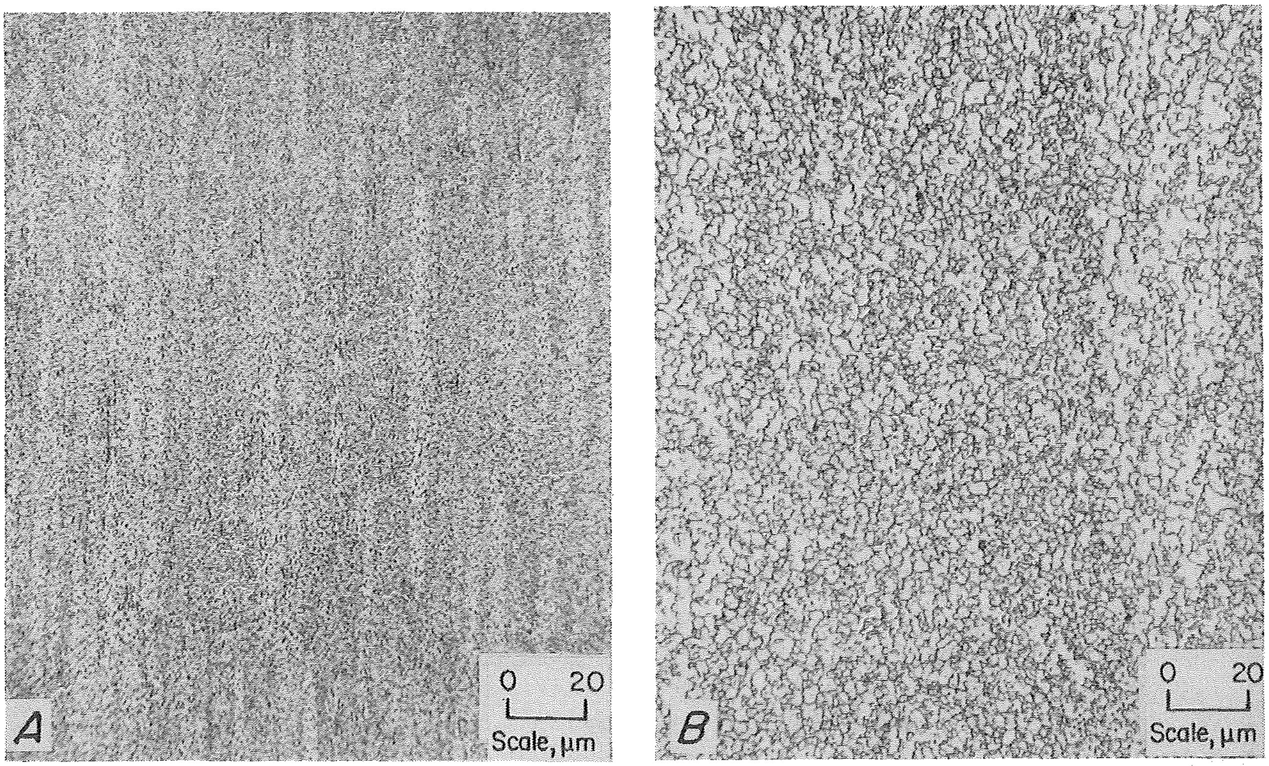

FIGURE 7. - Microstructure of CRM-6D extruded at 1,200° C. *A*, Lower magnification showing elongated regions of larger grains and fewer precipitates; *B*, higher magnification showing nearly equiaxed grains and predominantly grain boundary precipitates. Both views parallel to extrusion direction. Glyceregia etch.

Room-temperature tensile and 760° C stress-rupture data appear in table 5. Two specimens each of the air-canned or vacuum-canned powder were tested.

TABLE 5. - Mechanical properties of extruded CRM-6D prealloyed powder

Property	Canning atmosphere	
	Air	Vacuum
ROOM-TEMPERATURE TENSILE PROPERTIES		
Yield strength[1]....................psi..	78,000	79,000
Do[1]..............................psi..	80,000	81,000
Tensile strength....................psi..	139,000	145,000
Do..............................psi..	137,000	142,000
Elongation[2].......................pct..	10	14
Do..............................pct..	9	9
Reduction of area..................pct..	14	13
Do..............................pct..	12	11
STRESS-RUPTURE PROPERTIES, 760° C		
Time to rupture at 20,000 psi........hr..	4.0	6.2
Do..............................hr..	4.4	4.3
Elongation.........................pct..	44	44
Do..............................pct..	43	50
Reduction of area..................pct..	41	52
Do..............................pct..	56	50

[1]0.2-pct offset method.
[2]Based on a 1.4-in reduced section.

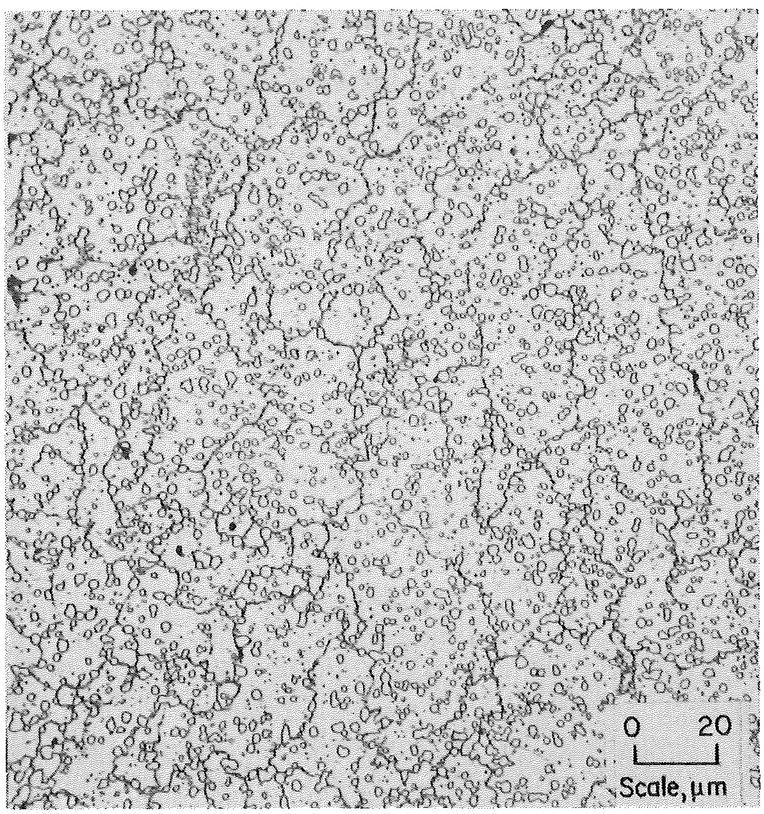

FIGURE 8. - Microstructure of P/M CRM-6D alloy forged and rolled at 1,200° C. Precipitates are $M_{23}C_6$ (M = Fe, Cr, Ni, Mn) and CbC. Glyceregia etch.

Forged and Rolled Material

Forging consolidated the canned CRM-6D powder to between 94 and 98 pct dense. With the air-sealed powder, subsequent rolling narrowed the range to between 96 and 98 pct dense. Vacuum sealing resulted in densities that were 98 to 99+ pct of theoretical density. The microstructure (fig. 8) consists of essentially equiaxed grains ranging from 15 to 19 µm in diameter. Coarse (1 to 3 µm) and fine (<0.5 µm) spherical precipitates appear, both intragranularly and intergranularly. Volume fractions were estimated from particle counts (number per unit area) and mean diameters. The coarse and fine precipitates accounted for 17 and 1 vol-pct, respectively. They were identified by X-ray examination of the extracted

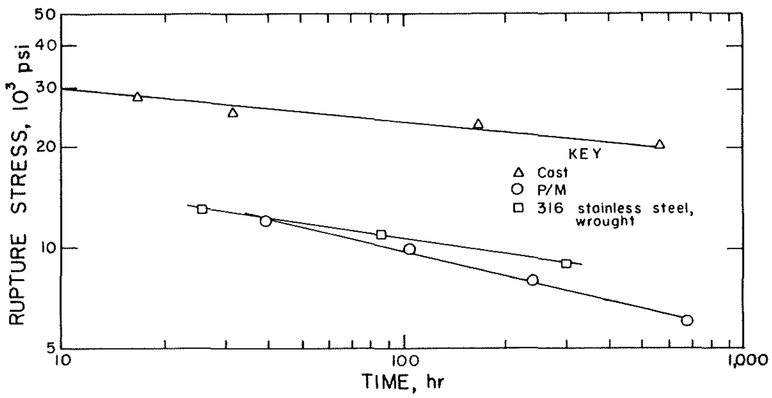

FIGURE 9. - Stress-rupture data for cast CRM-6D, P/M CRM-6D, and AISI 316 stainless steel. Tests performed in the range of 800° to 820° C in air.

particles as $M_{23}C_6$ and CbC, where M = Cr, Fe, Ni, and possibly Mn. No distinctions between coarse and fine particles could be made with these measurements; however, SEM-EDX analysis of the coarse carbides showed iron and chromium as the major components. EDX analysis of the fine precipitates was not feasible. Tungsten and molybdenum appeared as segregates in the EDX area scans, but no X-ray patterns of their carbides were detected. The forged-and-rolled material was nonmagnetic, signifying an austenitic matrix.

Tensile test results for two nominally equal specimens of the forged-plus-rolled CRM-6D are given in table 6. The room-temperature properties are superior to those published for cast CRM-6D (7). At 815° C, however, the reverse is true. Rupture strengths of the P/M alloy fell below those of the cast CRM by about 14,000 psi. Data for as-cast and as-consolidated P/M CRM-6D appear in figure 9. Also shown are data for wrought type 316 stainless steel, which were obtained both for reference values and to provide a check of the apparatus. These values fell within the range given in the literature for 316 stainless steel (1).

TABLE 6. - Room-temperature mechanical properties of forged-plus-rolled P/M CRM-6D powder

Specimen	Yield strength,[1] psi	Tensile strength, psi	Elongation, pct	Reduction of area, pct
P/M............	84,000	147,000	[2]13	7
Do...........	87,000	136,000	[2]6	7
Cast (7).......	65,000	105,000	3	NA

NA Not available.
[1]0.2-pct offset method.
[2]Based on a 1.1-in reduced section.

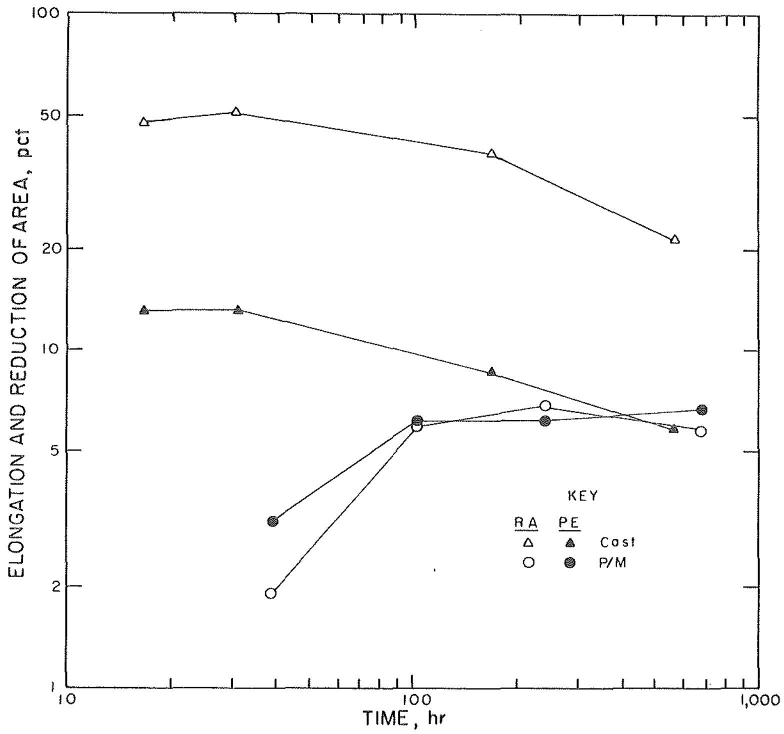

FIGURE 10. - Percent elongation (PE) and reduction-of-area (RA) data for stress-rupture tests of cast and P/M CRM-6D alloys. Tests performed in the range of 800° to 820° C in air.

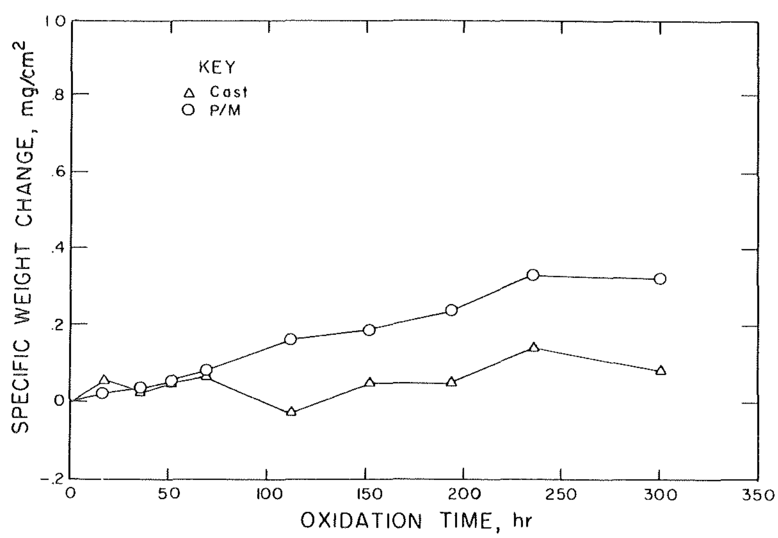

FIGURE 11. - Oxidation data for cast and P/M CRM-6D alloy. Test performed at 815° C in still air. Specimens were lightly brushed prior to weighing to remove loose scale.

The ductility behavior, illustrated in figure 10, differed for the two materials. The cast alloy necked and generally decreased in ductility at longer times. The P/M values changed little from those at room temperature (table 6), with the exception of the 40-hr data, which appear to be anomalously low. Oxidation of fracture surfaces obscured fine details under the SEM; however, the P/M fractures appeared to be intergranular; whereas, the cast had both intergranular and transgranular or ductile features.

The oxidation resistance of the cast and P/M alloys was not significantly different. Figures 11 and 12 show the results of two similar tests at 805° and 815° C. The specific weight changes for the P/M specimens after 300-hr exposures were 0.33 and 0.32 mg/cm^2, while those for the cast CRM-6D varied substantially more, ranging from 0.09 to 0.52 mg/cm^2. Averaging these two curves, however, gives results nearly equal to those for the P/M alloy. Included in the second test for reference were specimens of the 316 stainless steel. Their oxidation behavior appears in figure 12.

MATERIAL AND PROCESS VARIATIONS

Heat treatments (1 hr) of consolidated CRM-6D and CM established solidus temperatures of ~1,265° C

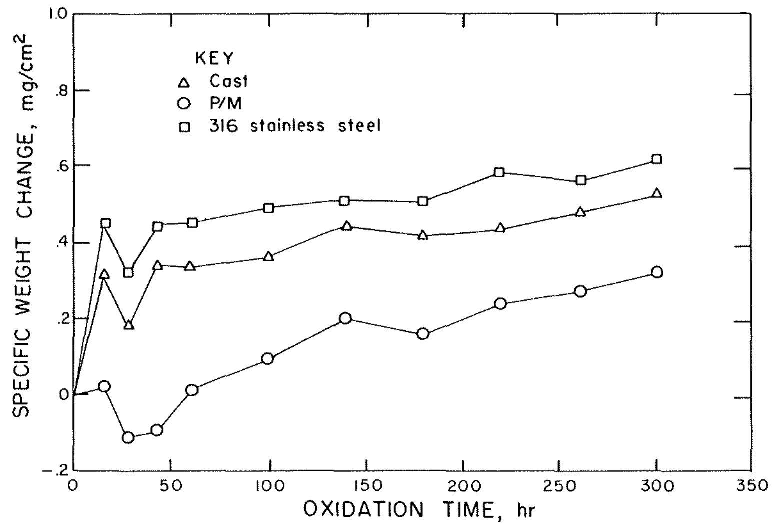

FIGURE 12. - Oxidation of cast and P/M CRM-6D alloy and AISI 316 stainless steel at 805° C.

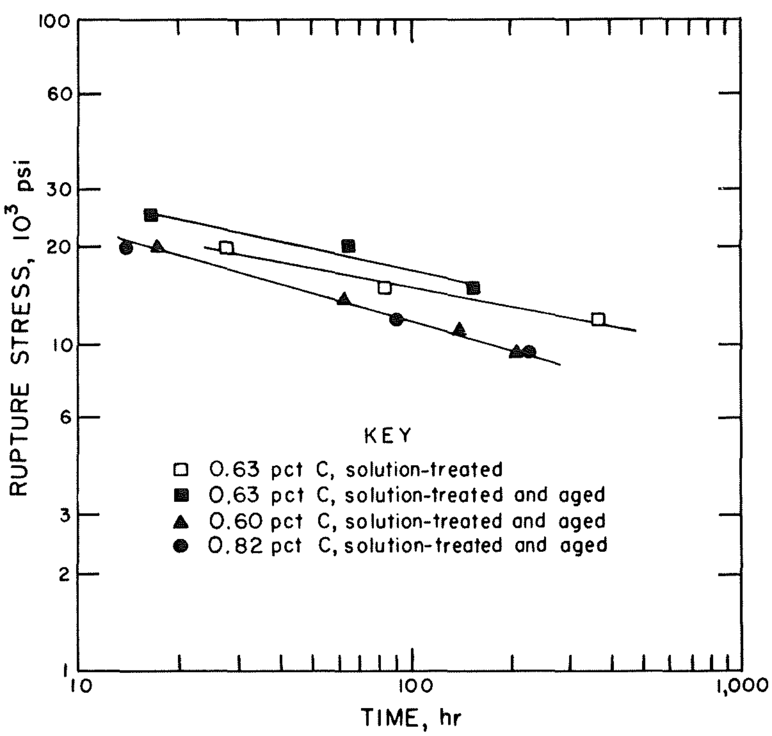

FIGURE 13. - Stress-rupture data for intermediate-carbon CRM alloys at 815° C in air.

and between 1,285° and 1,307° C, respectively, based on metallographic observations. Likewise, apparent solutioning of the coarse $M_{23}C_6$ carbides occurred at ~1,275° C in the CRM-6D. (It was not established whether the carbides dissolved and remained in solution or reprecipitated in the intergranular phase.) Intermediate-carbon compositions were therefore selected so that solution-treatments could be performed both above and below the solidus. A 0.63-pct C alloy was prepared from a blend of CL and graphite powders. The blend was canned and consolidated as described previously. Coupons of the consolidated powder were solution-treated for 40 to 60 min at 1,282° to 1,296° C, then water-quenched. After machining, half of these were aged 24 hr at 650° C in helium. Stress-rupture testing was performed at 815° C.

Following these tests, two blends of nominally 0.6 and 0.9 pct C were prepared from CM and graphite powders using the same consolidation procedures. Solution-treatments for the nominally 0.6 and 0.9 pct C specimens were 2 hr at 1,273° and 1,260° C, respectively, followed by water-quenching. Aging of machined specimens was 24 hr at 650° C. Chemical analyses of the two quenched-and-aged alloys gave carbon levels of 0.60 and 0.82 pct, respectively. Specimens were stress-rupture tested at 815° C and tensile tested at room temperature.

As seen in figure 13, aging increased the rupture strength of the solution-treated 0.63 pct C alloy by about 2,000 to 3,000 psi over the range tested. Ductility was not significantly affected by the aging, although it was relatively low (≤4 pct elongation) for both the as-solution-treated and the as-aged conditions. The two other alloys displayed equivalent stress-time dependences, but fell about 5,000 psi below the value for the 0.63 pct C alloy. These were also generally less ductile. Microstructures of the three alloys appear in figure 14. Note that the alloys have been stressed to rupture at 815° C. The black "holes" in figures 14A and 14B are inclusions that fell out during sample preparation. Transverse grain sizes for figures 14A, 14B, and 14C are 72, 84, and 100 µm, respectively. No measurement was made for figure 14D because of poor grain-boundary definition.

Sufficient material was not available for tensile testing of the 0.63 pct C alloy. Data for the 0.60 and 0.82 pct C alloys appear in table 7. As expected, the strength increases and ductility decreases with increased carbon. Although not strictly comparable because of the lack of heat treatment, the P/M CRM-6D data from table 6 generally fall in line with the values for the lower carbon alloys.

TABLE 7. - Room-temperature mechanical properties of hot-consolidated and heat-treated intermediate-carbon CRM powder

Specimen	Yield strength,[1] psi	Tensile strength, psi	Elongation,[2] pct	Reduction of area, pct
0.60 pct C....	75,000	125,000	19	16
	72,000	125,000	22	18
0.82 pct C....	87,000	140,000	10	11
	92,000	135,000	8	10

[1]0.2-pct offset method.
[2]Based on 1.4-in reduced section.

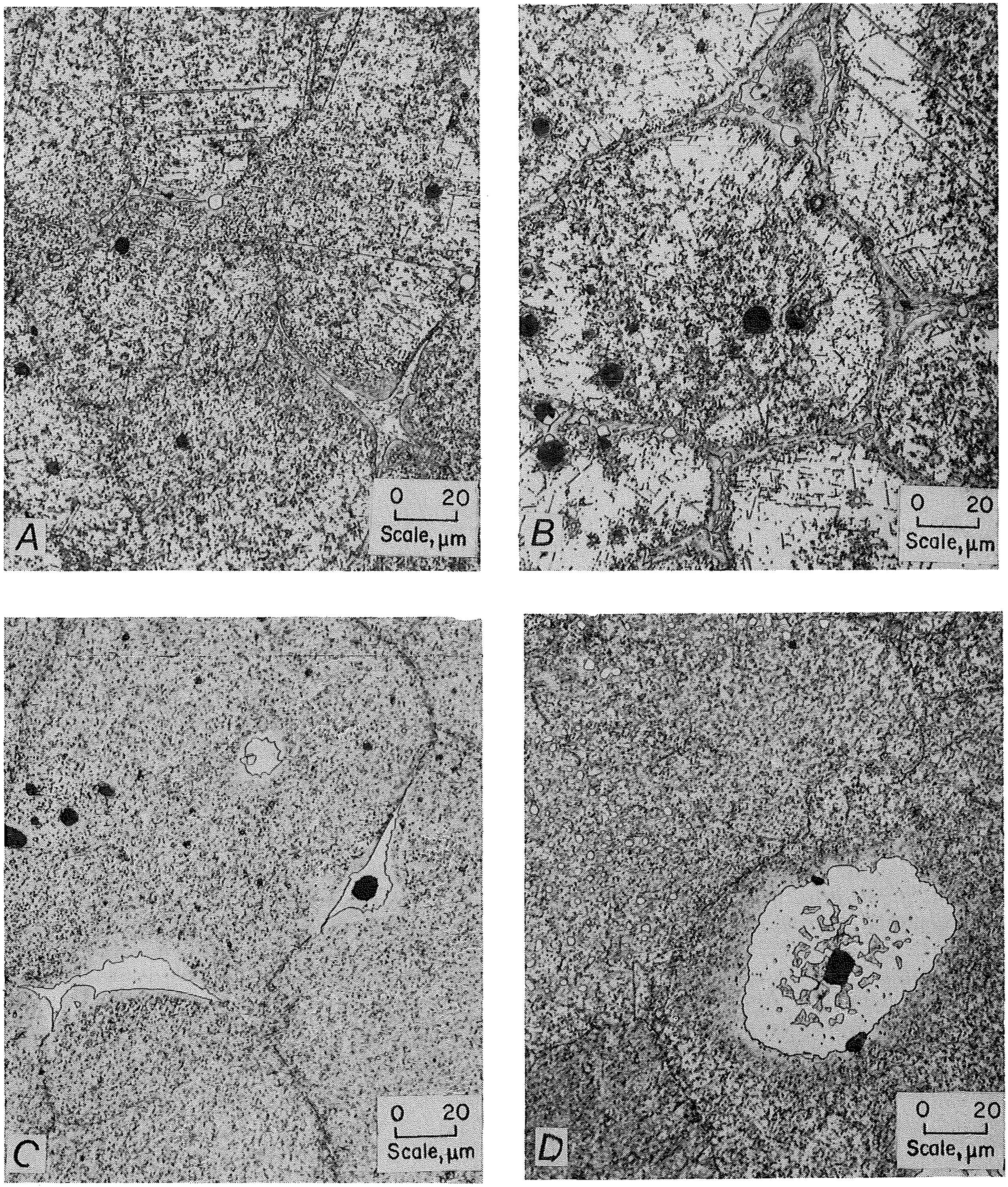

FIGURE 14. - Microstructures of consolidated and heat-treated intermediate-carbon P/M alloys. *A*, Solution-treated, 0.63 pct C alloy after 27 hr at 815° C; *B*, solution-treated and aged, 0.63 pct C alloy after 64 hr at 815° C; *C*, solution-treated and aged, 0.60 pct C alloy after 62 hr at 814° C; and *D*, solution-treated and aged, 0.82 pct C alloy after 92 hr at 815° C. Glyceregia etch.

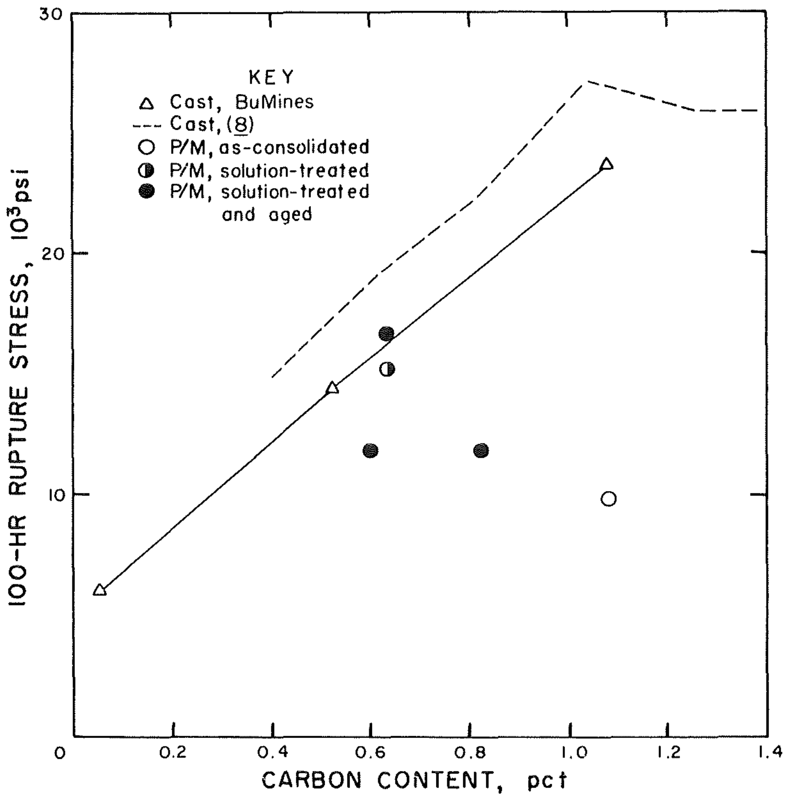

FIGURE 15. - One-hundred-hour rupture strengths at 815° C of CRM alloys as a function of carbon content. The solid-line data were determined at the Rolla Research Center; the dashed-line data were taken from reference 8.

The 100-hr rupture strengths, determined from the stress-rupture data, are plotted against carbon content in figure 15. Included for comparison are strengths of laboratory cast alloys, one CRM and two reduced-carbon CRM, as well as the literature values for cast and aged CRM. The difference in the two cast curves is attributed to the aging and probable differences in macrostructure and microstructure due to different casting techniques. It is seen that the P/M alloy containing 0.63 pct C, which was heat treated at the higher temperature of 1,287° C (at or just above the solidus), approaches the strength of the cast alloys. The two intermediate-carbon alloys containing 0.60 and 0.82 pct C, which were heat treated in the solid state, exhibited rupture strengths that fell below the interpolated strength curve of cast CRM by 3,000 and 7,000 psi, respectively.

DISCUSSION

The poor cold-pressing characteristics of the CRM powder were not unexpected, considering the spherical particle shape and high carbon content. Micrographs of the sintered powder showed virtually no particle deformation as a result of compacting. The green strengths of the compacts were due primarily to the binding effect of the lubricants, as was implied by the increase in green strength with rising lubricant level. If 1,000 psi is specified as a minimum desirable strength, then by extrapolation, 3.8 wt-pct Polymist or roughly 25 vol-pct is required. A more practical approach would be to increase the compacting pressure to about 140,000 psi, at which a lower level of lubricant could be used.

Even with adequate green strength, the resulting sintered densities would be insufficient to generate tensile strengths comparable with those of the cast material. A maximum pressed density of 81 pct at 140,000-psi compacting pressure and 1 pct Polymist might be expected from data extrapolation. However, if the experiences with lower density compacts hold for this density, then a short-term, solid-state sinter (less than 2 hr below 1,265° C) would not generate densities much greater than 88 pct.

Where strength is not paramount, as for forging preforms, the high porosity would expose the compact to internal oxidation at the forging temperature. Below 90 pct density, the closed porosity (that isolated from the compact surface) is about only 1 pct. The remaining porosity would expose particle surfaces to atmospheric oxidation, thereby impeding further consolidation. The use of liquid-phase sintering would solve the porosity problem, but at the expense of ductility. Hot-forging trials of liquid-phase-sintered alloy specimens, as part of other work in progress, produced cracking in the brittle, intergranular continuous phase, while not significantly deforming the remaining microstructure.

Both extrusion and hot-forging plus rolling of the canned powder produced excellent consolidation as evidenced by the densities and room-temperature mechanical properties. The relatively low rupture stresses of the extruded material is attributed to the smaller grain size, which promotes grain boundary sliding at higher temperatures. The greater elongation and relative absence of necking, both symptoms of superplasticity in which grain boundary sliding predominates, support this mechanism.

Two factors contribute toward the large difference in rupture strengths between the cast CRM-6D and the forged-plus-rolled P/M alloy: the microstructure and the carbide distribution. The microstructures of the two are contrasted in figures 8 and 16. Note that the cast CRM displays a dendritic structure, giving it an effectively large-grain size with high aspect ratio (length-to-diameter). This morphology is conducive to high rupture strength (6). The P/M structure is equiaxed and relatively fine grained (although larger than that of the extruded CRM). Heat treatments up to the solidus temperature in the forged-plus-rolled alloy increased the grain size from 17 µm to only about 30 µm, while retaining the equiaxed structure in directions both parallel and transverse to the rolling direction. The presence of the carbides blocked grain boundary movement, thus preventing the development of the large-grained microstructure conducive to high-temperature creep strength.

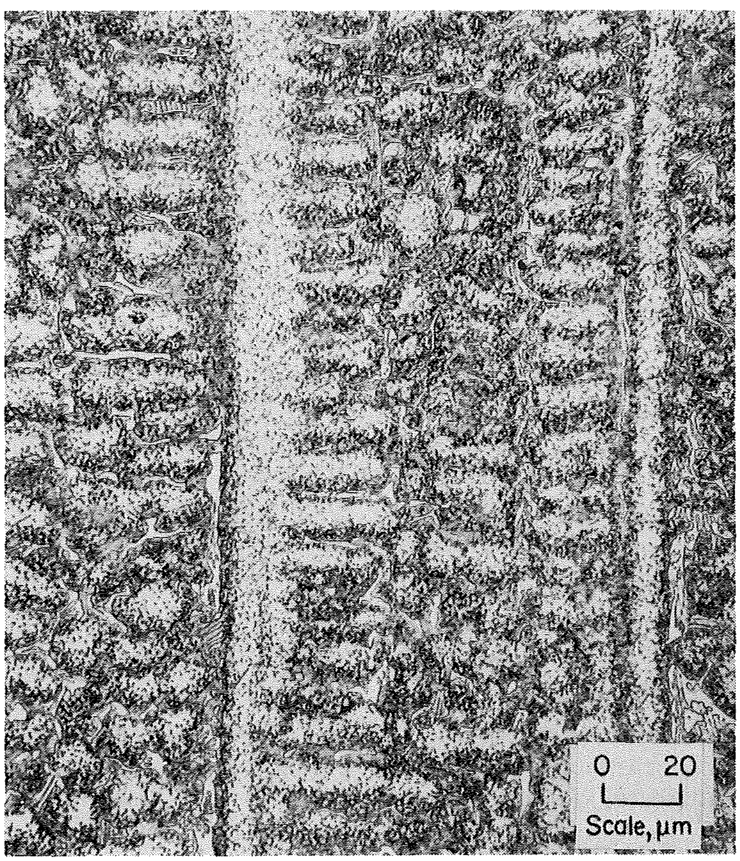

FIGURE 16. - Microstructure of cast CRM-6D after 300 hr at 815° C. Glyceregia etch.

The carbides also weakened the grain boundaries, leading to intercrystalline fracture. Although high-temperature oxidation of fracture surfaces prevented direct observation of grain-boundary carbides by SEM, such carbides do appear in optical micrographs of unfractured specimens (fig. 5). Furthermore, despite the surface oxidation, the fractures did appear to be entirely intergranular in the P/M specimens.

A third difference between the rupture strengths of P/M and cast alloys is due to the presence of a very fine carbide distribution that develops in the latter during high-temperature service. This precipitate phase appears in figure 16 as a darkening in the dendrites. No corresponding precitate appears in the high-carbon P/M specimen. Indeed, if the estimates of the $M_{23}C_6$ and CbC volume fractions are accurate, then one can account for about 0.97 wt-pct C in these precipitates, which are present at consolidation. With a carbon solubility of 0.03 wt-pct, a supersaturation of just 0.08 wt-pct C remains for aging precipitation.

The lower carbon content of the intermediate-carbon CRM alloys did allow for solid-state solution-treating, with two primary results: Grain growth occurred during solution-treatment, and a fine precipitation was generated during aging. Although both of these phenomena should have led to rupture strength increases, grain boundary precipitates, which appear in both the 0.60 and 0.82 pct C alloys, produced lower than expected strengths.

The nominally liquid-phase solution-treatment of the 0.63 pct C alloy failed to generate as fine a precipitate structure as was seen in the others. This may result from segregation of the carbon in an intergranular phase. If so, this phase is probably stronger than the solid-state grain boundaries at elevated temperatures, but more brittle at room temperature. The higher rupture strengths may also be due to the larger grain sizes.

SUMMARY AND CONCLUSIONS

Means of consolidating an atomized, prealloyed iron-base superalloy, CRM-6D, were investigated. Conventional pressing to 100,000 psi (50 tsi) and sintering for 1 or 2 hr at 1,150° to 1,300° C proved ineffective in developing dense, strong, and ductile material. Below 1,265° C, the powder remained solid but densified to less than 90 pct of theoretical. Above this temperature, liquid-phase sintering occurred, producing room-temperature ductilities of $\leq$0.1 pct. In neither case did tensile strength exceed 70,000 psi.

Canning, then forging and rolling at 1,200° C, consolidated the powder to a dense, strong alloy with tensile strength in excess of 130,000 psi and ductility $\geq$6 pct. Resultant rupture strengths were approximately equal to or higher than those of wrought stainless steels at 815° C (1), but fell below those of cast CRM-6D. Strengthening through grain-coarsening was not achieved due to the presence of coarse, insoluble carbides.

Reduced carbon levels together with heat treatments led to substantially higher rupture strengths. A solution-treated and aged P/M alloy with carbon reduced to 0.63 pct was as strong as the cast CRM alloy of comparable carbon content.

Extrusion at 1,200° C produced a fine-grained, nearly equiaxed structure with high-temperature properties approaching a condition of superplasticity. Rupture stresses were not improved over those of forged and rolled material.

The oxidation resistance of P/M CRM alloy was found to be equivalent to that of cast CRM-6D.

REFERENCES

1. American Society for Testing and Materials. An Evaluation of the Yield, Tensile, Creep, and Rupture Strengths of Wrought 304, 316, 321, and 347 Stainless Steels at Elevated Temperatures. ASTM Data Series DS 5S2, 1969, p. 62.

2. _____. Standard Methods of Tension Testing of Metallic Materials. E8-69 in 1976 Annual Book of Standards: Part 10, Metals--Mechanical, Fracture, and Corrosion Testing; Fatigue; Erosion, Effect of Temperature. Philadelphia, Pa., 1976, pp. 120-140.

3. _____. Standard Method of Test for Density and Inner Connected Porosity of Sintered Powder Structural Parts and Oil-Impregnated Bearings. E328-73 in 1975 Annual Book of Standards: Part 9, Electrodeposited Metallic Coatings; Metals Powders, Sintered P/M Structural Parts. Philadelphia, Pa., 1975, pp. 144-145.

4. _____. Standard Method of Test for Green Strength of Compacted Metal Powder Specimens. B312-64 in 1975 Annual Book of Standards: Part 9, Electrodeposited Metallic Coatings; Metal Powders Sintered P/M Structural Parts. Philadelphia, Pa., 1975, pp. 119-122.

5. Benjamin, J. S. Dispersion Strengthened Superalloys by Mechanical Alloying. Met. Trans., v. 1, October 1970, pp. 2943-2951.

6. Cairns, R. L., L. R. Curwick, and J. S. Benjamin. Grain Growth in Dispersion Strengthened Superalloys by Moving Zone Heat Treatments. Met. Trans., v. 6A, January 1975, pp. 179-188.

7. Roy, A., F. A. Hagen, and C. Belleau. Chrysler's Gas Turbine Car, Materials Requirements. Society of Automotive Engineers, Automotive Engineering Congress and Exposition, Jan. 13-17, 1964, Detroit, Mich., Paper 777C, pp. 6-12.

8. Roy, A., F. A. Hagen, and J. M. Corwin. Iron-Base Superalloys for Turbine Engines. J. Metals, v. 17, No. 9, September 1965, pp. 934-939.

☆U.S. GOVERNMENT PRINTING OFFICE: 1981-703-002/66 INT.-BU.OF MINES,PGH.,PA 25424