NASA Technical Memorandum 83632

Effects of Long-Time Elevated Temperature Exposures on Hot-Isostatically-Pressed Powder-Metallurgy Udimet 700 Alloys with Reduced Cobalt Contents

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Prepared for the
One hundred thirteenth Annual Meeting of the American Institute of Mining, Metallurgical and Petroleum Engineers
Los Angeles, California, February 26-March 2, 1984

NASA

E-2071

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SUMMARY

Because almost the entire U.S. consumption of cobalt depends on imports, this metal has been designated "strategic." To achieve conservation, the role and effectiveness of cobalt is being evaluated in commercial nickel-base superalloys. Udimet 700 type alloys in which the cobalt content had been reduced from the normal 17 percent to 12.7, 8.5, 4.3, and 0 percent were prepared by standard powder metallurgy techniques and hot isostatically pressed into billets. Mechanical testing and microstructural investigations were performed. The mechanical properties of the alloys with reduced cobalt contents which were heat-treated identically were equal to or better than those of the standard alloy, except that creep rates tended to increase as cobalt was reduced. The effects of long time exposures at 760° C on mechanical properties and at 760° and 845° C on microstructures were determined. Decreased tensile properties and shorter rupture lives with increased creep rates were observed in all alloy modifications. The exposures caused gamma prime particle coarsening and formation of sigma phase in the alloys with higher cobalt contents. Exposure at 845° C also reduced the amount of MC carbides.

INTRODUCTION

The extensive land surface of the United States contains a great wealth of minerals. And yet not every element is present in quantities sufficient to supply the requirements of its inhabitants for their well being and security. Materials which are predominantly or wholly imported and are essential to the economic health or defense of the United States have been designated strategic. Many strategic materials are used in the aerospace industry and most prominent among these are the elements chromium, cobalt, columbium, and tantalum, which are critical to the performance of turbine engines. For example, a F-100 engine contains 675 kg chromium, 400 kg cobalt, 65 kg columbium, and 1.5 kg tantalum in its heat resistant and structural parts (ref. 1). Let us take a closer look at cobalt. In 1982 the United States consumed about 4 700 000 kg and of that all but 9 percent was imported (ref. 2). Cobalt is perhaps unique in that its greatest use is in superalloys, which in turn are principally manufactured into aircraft engine parts. One such superalloy is Udimet 700.[1] This nickel base alloy contains nominally in weight percent, 17 cobalt, 15 chromium, 5 molybdenum, 4 aluminum, and 3.5

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[1]Trade name of Special Metals Company.

titanium. It is a heat treatable alloy and has a microstructure of about 45 percent gamma prime in a gamma matrix.

Udimet 700 finds application as cast and wrought products in turbine blades and turbine disks, and as hot isostatically pressed powder metallurgy (HIP-PM) products in turbine disks. This alloy is one of several under study in the program named COSAM directed toward Conservation Of Strategic Aerospace Materials. The purpose of the COSAM programs is to determine the roles and effectiveness of cobalt in superalloys. Specifically, in Udimet 700 various amount of cobalt have been replaced by increased nickel contents and the effect on mechanical properties and microstructures determined (ref. 3). The effects of long term exposures at elevated temperatures on such modified Udimet 700 alloys are reported in this paper and the resulting mechanical properties are explained on the basis of microstructures.

MATERIALS AND PROCEDURES

Alloys

Five compositions based on the nickel-base alloy Udimet 700 were used in this program. One of the alloys was of the standard composition containing 17 percent cobalt, 15 percent chromium, 5 percent molybdenum, 3.5 percent titanium, and 4 percent aluminum as major additions. In the other four alloys, cobalt was reduced to 12.7, 8.5, 4.3, and 0 percent and replaced by nickel. Master melts of each composition were argon atomized to powder by Special Metals Corporation. Stainless steel cans of approximately 3 mm wall thickness were filled with the powders, vacuum degassed, sealed, and hot isostatically pressed for 2 hr at 760° C with 3.5 MPa pressure followed by 3 hr at 1210° C with 100 MPa pressure. Final dimensions of the canned billets were about 55 mm in diameter by at least 300 mm length. The chemical analyses for the five compositions investigated in this program are given in table I. Thermally induced porosity (TIP) tests showed density reductions of less than 0.2 percent after 4 hr at 1210° C.

Heat Treatment

Prior to heat-treating, the billets were cut into blanks suitable for machining test specimens. The blanks were given a heat-treatment commonly used for Udimet 700 turbine disks, consisting of partial solutioning of the gamma prime followed by aging. The partial solutioning temperatures chosen were approximately 42° C below the gamma prime solvus of the alloys. The temperatures of the gamma prime solvus, as determined by DTA, were 1150°, 1160°, 1170°, 1180°, and 1188° C for the 17, 12.7, 8.5, 4.3, and 0 percent content alloys, respectively. The blanks were held at the partial solutioning temperature for 4 hr and quenched in oil. Blanks of all compositions were then given the same aging treatments of 870° C for 8 hr, 980° C for 4 hr, 650° C for 24 hr, and 760° C for 8 hr with air cooling after each of the four steps. Fully heat-treated mechanical test specimen blanks of each composition were further exposed (overaged) at 760° C for 1500 hr. Separately, coupons from all alloys in the fully heat-treated condition were given extended exposures of 72, 216, 500, 1500, and 5000 hr at 760° C and 845° C to determine their microstructural stability.

MATERIAL CHARACTERIZATION

Specimens for metallographic examination were first ground through 600 grit silicon carbide papers and then polished on cloth covered wheels with diamond paste or alumina slurries of 0.5 μm fineness. For optical examination, the gamma prime was preferentially dissolved by a solution of 33 percent hydrochloric acid, 33 percent acetic acid, 33 percent water and 1 percent hydrofluoric acid. Specimens for transmission electron microscopy were thinned in a methanol solution with 7 percent perchloric acid and 20 percent butanol.

The amount of large gamma prime particles, remaining after partial solution was measured as a volume fraction by point counting of scanning-electron-micrographs at 10 000 x magnification, in conformance with ASTM standard recommended practice E562. For fully aged materials, weight fractions of the total gamma prime content were determined from extractions. Gamma prime particle sizes were determined from edge lengths of gamma prime cubes.

The gamma prime phase was extracted by dissolving the gamma phase electrolytically in a solution of 1 percent citric acid and 1 percent ammonium sulfate in water at a current density of 0.075 A/cm^2. Weights were determined for the solid specimens before and after the extraction, and for the gamma prime residue collected by filtration. The residue was used for X-ray diffraction and electron microscopy of the gamma prime phase.

Extraction of the carbide, boride and sigma phases was by electrolysis in a solution of 90 ml methanol, 10 ml hydrochloric acid and 1 g tartaric acid at a current density of 0.075 A/cm^2.

Mechanical Tests

All mechanical tests were performed in air. Test procedures conformed to applicable ASTM recommended practices. Specimens were tensile tested at 650° C at a strain rate of 0.02 per min. The creep rupture tests were run at 760° C. Creep strain measurements were determined from the movement of extensometers attached to the shoulders of the specimens and monitored electronically by means of linear variable differential transformers (LVDT).

RESULTS

Microstructure

After the partial solutioning and 4-step aging treatment the microstructure consists of a gamma matrix with an approximate ASTM No. 7 grain size, containing gamma prime particles of three different size ranges. The largest, measuring at least 0.5 μm are residual from the partial solutioning. They occupy about 13 volume percent of the structure in all alloys. As shown in figure 1, these particles form rows in the higher cobalt level alloys and ogdoadic clusters at lower levels. Also faintly visible in figure 1 are particles measuring about 0.2 μm which were grown at the 980° C aging temperature. Finally, the 760° aging temperature formed ultrafine gamma prime particles measuring 0.02 μm. These are shown together with the larger particles in the transmission electron photomicrograph of figure 2. The ultrafine particles are most numerous in the 17 percent cobalt alloy and their number is significantly less when cobalt is completely replaced by nickel (fig. 3). Together the three sizes of gamma prime particles constitute about 46 percent of the total weight of each alloy.

The five alloys were overaged at 760 and 845° C for up to 5000 hr. The lower of these temperatures equals the last step of the aging heat treatment, but the higher exceeds it. Aging at either temperature caused growth of the fine and larger gamma prime particles at the expense and eventual disappearance of the ultrafine particles. This is shown in figures 4 and 5 for the alloys with 17 and 0 percent cobalt respectively. Growth of the fine gamma prime particles is represented graphically in figure 6. It was observed that at the 0 percent cobalt level the fine particles were somewhat larger and that the scatter of sizes also was slightly greater.

X-ray diffraction of carbide extractions made at selected time intervals proved that $M_{23}C_6$ carbides were present in all alloys in every overaged condition. MC carbides survived aging at 760° C. However as shown in table II, MC carbides were no longer present in the five alloys, when aging at 845° C reached 1500 hr.

Extended aging tended to produce sigma phase in alloys containing 8.5 percent or more cobalt (table II). The sigma phase was found by X-ray diffraction in specimens exposed 72 or more hr at 845° C or 500 more hr at 760° C, but was difficult to detect by metallography. An example of platelets in the 17 percent cobalt-content alloy aged 5000 hr at 845° C is presented in figure 7. The quantity of sigma platelets did not exceed 0.1 percent after any of the extended aging treatments.

Mechanical Properties

The tensile yield strengths, ultimate strengths and elongations of the as heat-treated alloys are nearly identical for the five compositions. Overaging causes a slight decrease in strength, but has no effect on ductility. The results for tensile tests at 650° C are graphically represented in figure 8. (The low elongation reported in the overaged 12.7 percent cobalt alloy is based on a single test result.)

The creep rupture lives were determined by single tests at 760° C and a 475 MPa stress. The results are shown in figure 9 and table III. In as heat-treated alloys the maximum life, 98 hr, was registered for the 4.3 percent cobalt content composition. For comparison, the life for the standard 17 percent cobalt alloy was 60 hr and for the cobalt-free composition, it was 51 hr. This trend it preserved in general after the 1500 hr overaging but the average of the rupture lives is reduced by about one half. Table III shows that the minimum creep rates of the as heat treated alloys increase as cobalt is replaced by nickel, from $4x8^{-10}s^{-1}$ for the 17 percent cobalt content alloy to $9x10^{-8}s^{-1}$ for the cobalt-free alloy. Overaging increases the creep rates in all alloys to about $1.2x10^{-7}s^{-1}$ except for the cobalt-free alloy where the rate is $3.7x10^{-7}s^{-1}$. No clear trend is evident for the ductility at failure between as heat treated and overaged alloys, nor between the composition and the various cobalt contents.

DISCUSSION

The volume fraction of ultrafine (0.02 μm) gamma prime particles in the modified Udimet 700 alloys is believed to play a major role in controlling the mechanical behavior of these alloys. When the quantity of the ultrafine particles is compared between the alloys of different cobalt contents in the heat treated condition it is observed to decrease as the cobalt content decreases (fig. 3). This means that more of the total gamma prime is represented by fine (0.2 μm) gamma prime, since the coarse particles occupy about 13 volume percent in every alloy and the total gamma prime present is essentially the same for all compositions. This observation is in accord with Van der Molen et al (ref. 4) who show that the volume fraction of gamma prime formed during aging is controlled by the temperature difference between the gamma prime solvus and the aging temperature. This temperature difference increases along with the gamma prime solvus (ref. 5) as cobalt decreases because the maximum aging temperature for all alloys was held constant at 980° C. The effect on tensile properties is not very great (fig. 8). However at 760° C the reduction in amount of ultrafine gamma prime particles as the result of cobalt removal, produced a consistent, small increase in minimum creep rates (table III). In contrast, when one compares rupture lives, a significant maximum is achieved at the intermediate cobalt content of 4.3 percent (fig. 9 and table III). At present it is unclear why there is a difference in behavior between rupture life and creep rates. This subject remains under investigation.

Prolonged aging at elevated temperatures causes the ultrafine gamma prime particles to disappear, either through being absorbed by diffusion to the fine and coarse particles already present in the microstructure, or by selective growth of some ultrafine particles. This change in the microstructures is associated in all alloys with slightly lowered tensile strengths at 650° C (fig. 8), significantly shorter times to reach one percent creep strain and failure, and higher minimum creep rates at 760° C (fig. 9 and table III). Evidently, under constant loading the absence of ultrafine particles leads to increased mobility of dislocations and accelerated failure.

The role of carbides on mechanical properties was considered also. No agglomerations or networks of carbides were observed. X-ray analysis indicated a near constant quantity of complex ($M_{23}C_6$) carbides during all overaging exposures (table II) and the influence of these carbides on properties should have remained constant. MC carbides also seemed to be little affected by overaging at 760° C and their effect should be discounted. Overaging at 845° C tended to remove the mono-carbides, but specimens so exposed were not subjected to mechanical tests, although here one may assume also, that the effect of their disappearance should be minor compared to that of the ultrafine gamma prime particles, which originally were so much more numerous.

The appearance of sigma phase platelets after longer exposures in the higher cobalt content alloys denotes some microstructural instability and can deteriorate the mechanical properties. But due to the relative scarcity and small size of the platelets, their presence in these alloys can be considered of negligible importance.

The results emphasize that a Udimet 700 type alloy in which one half to three quarters of the normal 17 percent cobalt content has been replaced by nickel could well be a substitute superior to the original alloy in HIP-PM applications. It should also be pointed out that the heat treatment given these test specimens was specifically designed for the alloy with 17 percent cobalt. Such a heat-treatment will not necessarily generate optimum microstructures and properties in the alloys with the reduced cobalt contents. Indeed, if operation at 760° C is contemplated, some extra insurance against rapid microstructural degeneration should be obtainable by raising the final aging from 760 to 780° C. While the resulting ultrafine gamma prime particles should then be coarser and less numerous, they would undergo a more gradual absorption by the fine gamma prime particles.

SUMMARY OF RESULTS

In the continuing effort to reduce U.S. dependence on imported strategic materials the need for cobalt and its effectiveness in Udimet 700 was investigated.

It was found that some or all of the cobalt can be replaced in HIP-PM Udimet 700 without affecting its usefulness as a superalloy. Indeed a reduction of the cobalt level from 17 to 8.5 or 4.3 percent may create an alloy of superior properties. Exposure of the alloys in the original (17 percent Co) and reduced cobalt compositions at 760 and 845° C for prolonged times result in:

1. Reduced tensile strength at 650° C.

2. Reduced rupture lives at 760° C.

3. Increased creep rates in rupture tests.

4. No significant effect on ductility.

5. Disappearance of ultrafine (0.02 μm) gamma prime particles from the microstructure.

6. Growth of the fine (0.02 μm) gamma prime particles in the microstructure.

7. Appearance of slight amounts of sigma phase in the alloys containing 8.5 or more percent cobalt after about 500 hr exposure at 760° C and 72 hr exposure at 845° C.

8. Disappearance of MC carbides in all compositions after 1500 hr exposure at 845° C.

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TABLE I. - ANALYZED COMPOSITIONS OF HOT ISOSTATICALLY PRESSED ALLOYS

Co	Cr	Mo	Ti	Al	C	B	Ni
Weight, percent							
17.0	14.8	5.10	3.58	4.04	0.06	0.026	Bal.
12.7	14.8	5.10	3.57	4.04	.06	.023	Bal.
8.5	14.8	5.00	3.54	4.08	.06	.022	Bal.
4.3	14.9	4.85	3.53	4.04	.07	.020	Bal.
0	15.0	5.00	3.51	4.00	.065	.019	Bal.

TABLE II. - EFFECT OF LONG TIME AGING ON UDIMET 700 TYPE ALLOYS

[X-ray diffraction of carbide extractions. Relative intensities: S = strong, M = medium, W = weak, 0 = absent, $M_{23}C_6$ = S after all exposures; MC = M after all exposures unless otherwise indicated.]

Time, hr	Percent Co in alloy				
	17	12.7	8.5	4.3	0
Aged at 760° C					
0	MC = S	MC = W	------	------	------
72	MC = S	MC = S	------	------	------
216	------	------	------	σ = W	------
500	σ = W	------	------	------	------
1500	σ = M	------	σ = W	------	------
5000	σ = S	σ = S	σ = W	------	------
	MC = W	MC = S	------	------	------
Aged at 845° C					
72	σ = W	σ = W	σ = W	------	------
	------	MC = S	------	------	------
216	σ = S	------	------	------	------
	MC = W	MC = W	MC = W	------	MC = W
500	σ = S	σ = W	------	------	------
	MC = W	MC = W	MC = W	MC = W	MC = W
1500	σ = M	σ = W	σ = W	------	-------
	MC = 0	MC = 0	MC = 0	MC = 0	MC = 0
5000	σ = S	σ = S	σ = W	------	------
	MC = 0	MC = 0	MC = 0	MC = 0	MC = 0

TABLE III. - SUMMARY OF CREEP RUPTURE RESULTS AT 475 MPa and 760° C

Cobalt, percent	Condition	Life, hr	Minimum creep rate, sec^{-1}	Time to reach strain of 1 percent, hr	After rupture	
					Elongation, percent	R of A, percent
17.0	As heat treated	60	$4.2x10^{-8}$	27	4.1	5.4
12.7		69	$5.4x10^{-8}$	44	3.7	4.0
8.5		75	$5.5x10^{-8}$	35	2.6	3.0
4.3		98	$6.1x10^{-8}$	21	2.4	4.0
0		51	$9.2x10^{-8}$	19	1.1	3.7
17.0	After 1500 hr exposure at 760° C	41	$1.3x10^{-7}$	20	2.6	2.5
12.7		46	$1.3x10^{-7}$	9.0	2.9	3.0
8.5		31	$1.3x10^{-7}$	6.8	.6	2.5
4.3		62	$1.2x10^{-7}$	9.2	1.8	4.4
0		19	$3.7x10^{-7}$	1.2	2.3	4.4

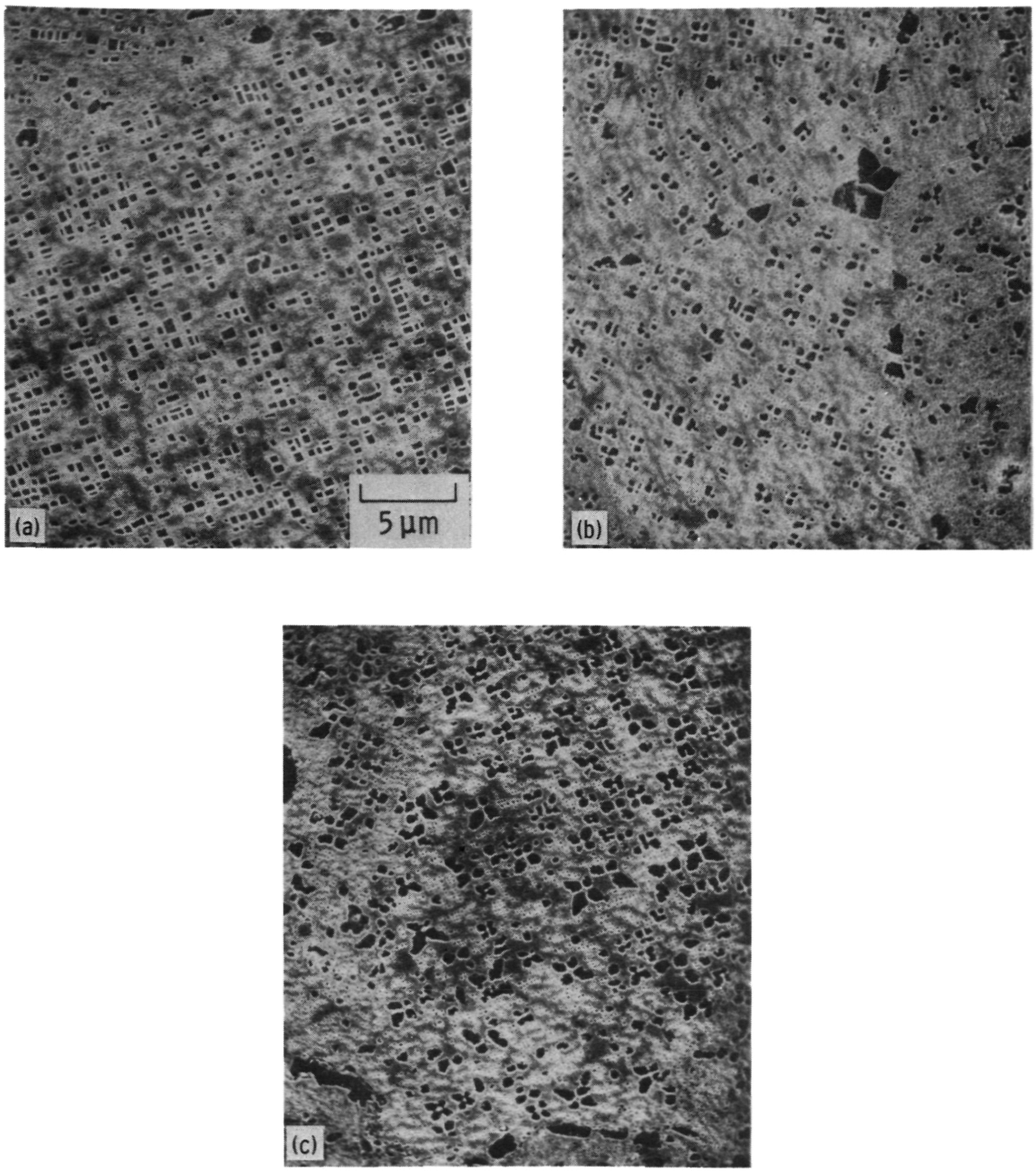

Figure 1. - Microstructures of heat-treated HIP-PM Udimet 700 alloys with, (a) standard 17 percent cobalt content, (b) 8.5 cobalt, and (c) 0 percent cobalt.

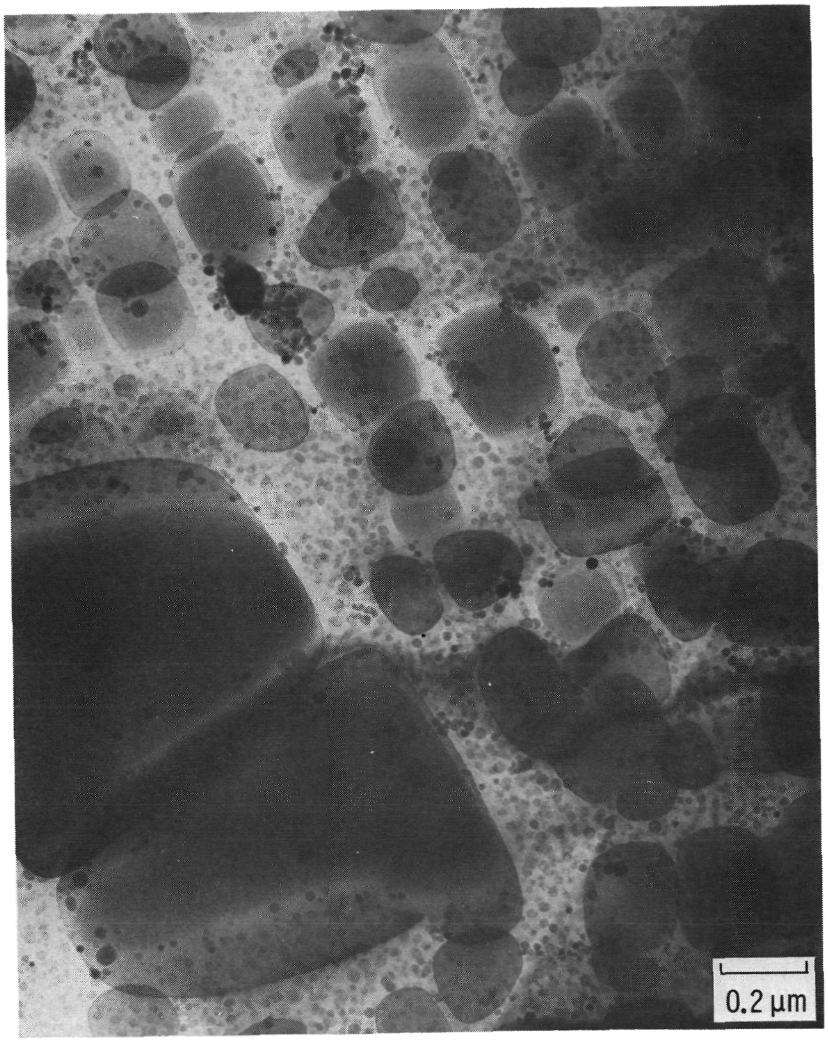

Figure 2. - Transmission electron micrograph of heat-treated HIP-PM Udimet 700 alloy showing 3 sizes of gamma prime particles.

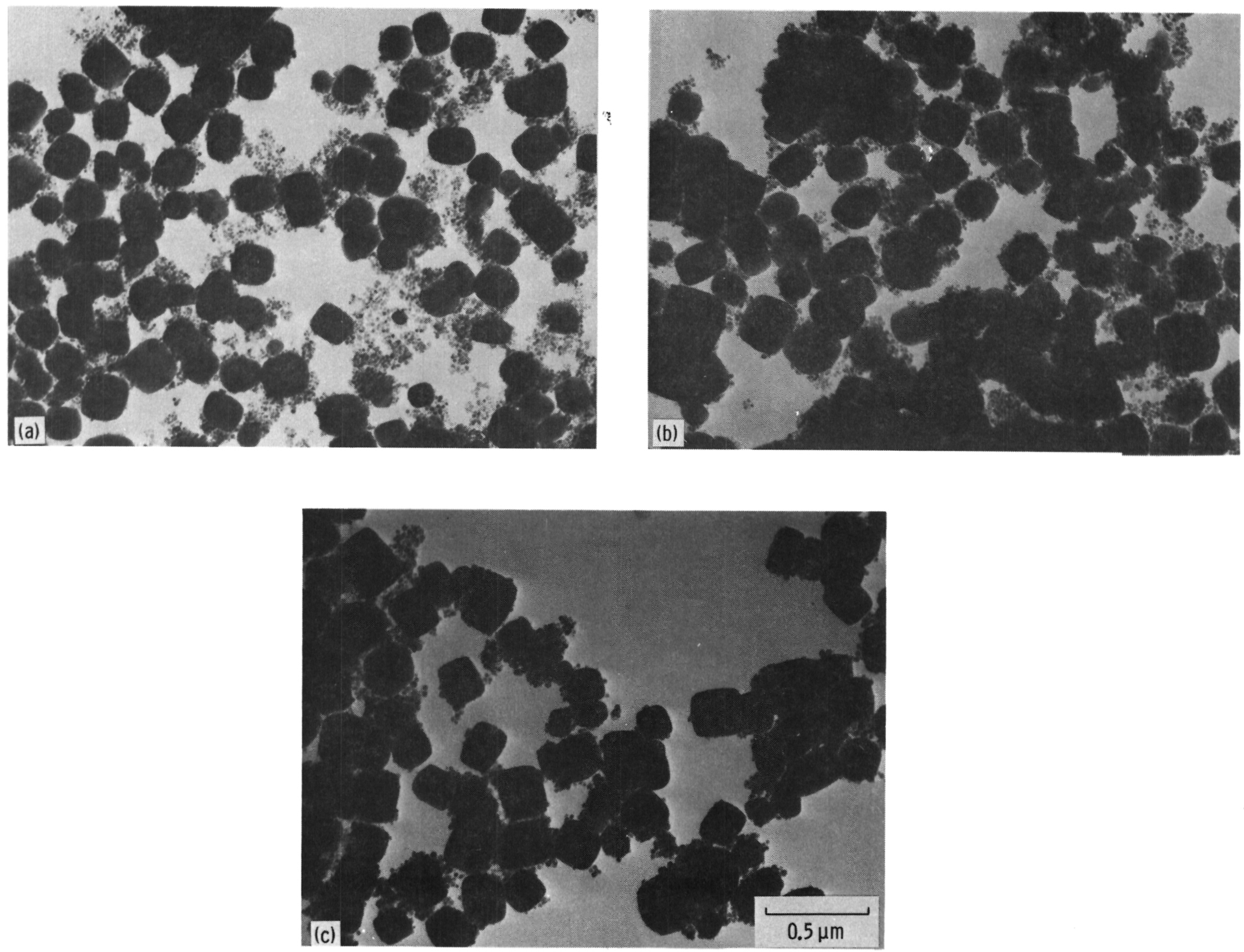

Figure 3. - Gamma prime particles extracted from heat-treated HIP-PM Udimet 700 type alloys. Note decreasing amounts of ultrafine gamma prime particles as cobalt decreases from, (a) 17 percent, through (b) 8.5 percent, to (c) 0 percent.

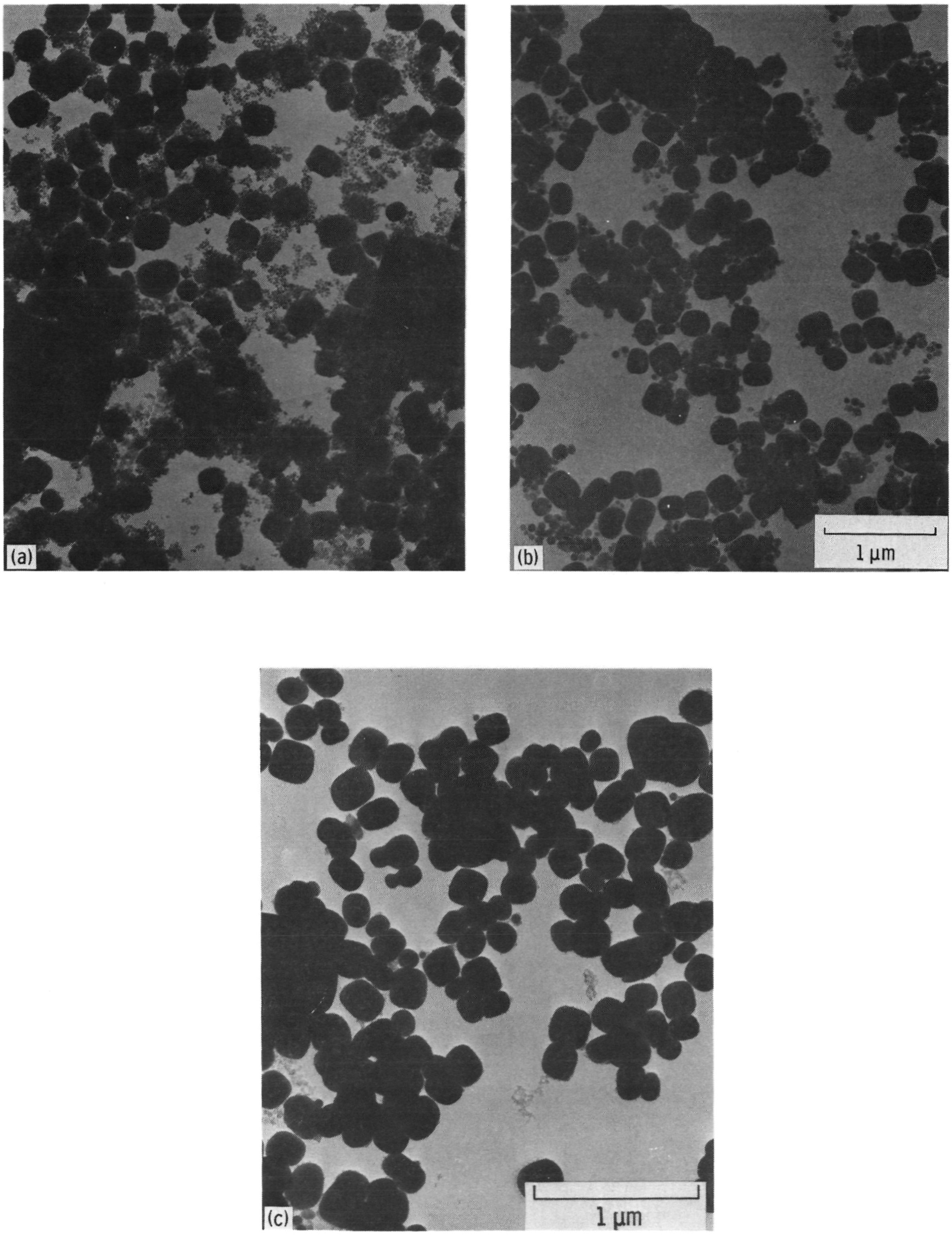

Figure 4. - Effect of exposure to 760° C on microstructure of HIP-PM Udimet 700. Shown are extracted gamma prime particles, (a) as heat-treated, (b) after 500 hours, (c) after 1500 hours. Note disappearance of ultrafine particles.

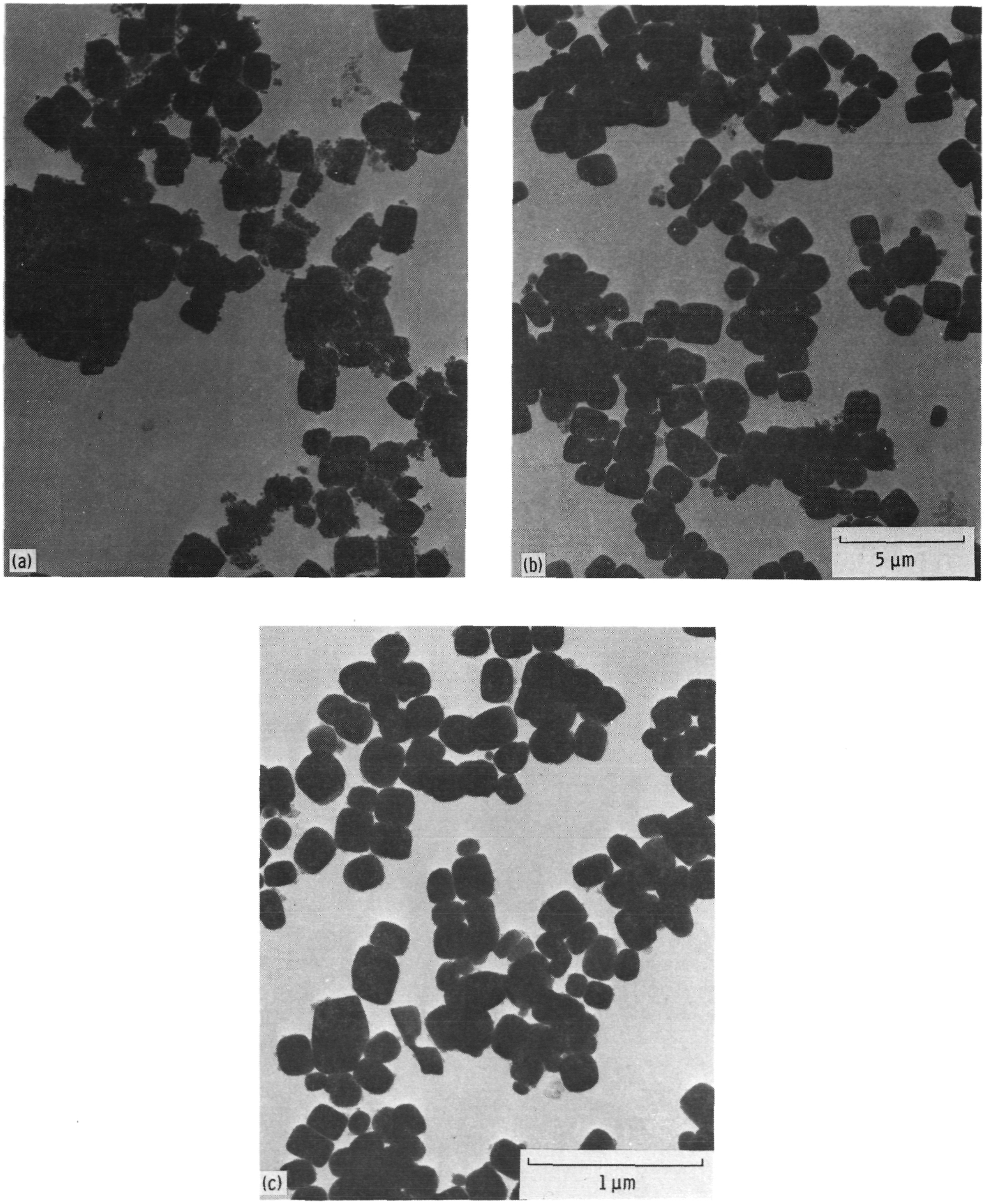

Figure 5. - Effect of exposure to 760° C on microstructure of HIP-PM Udimet 700. Shown are extracted gamma prime particles, (a) as heat-treated, (b) after 500 hours, (c) after 1500 hours. Note disappearance of ultrafine particles.

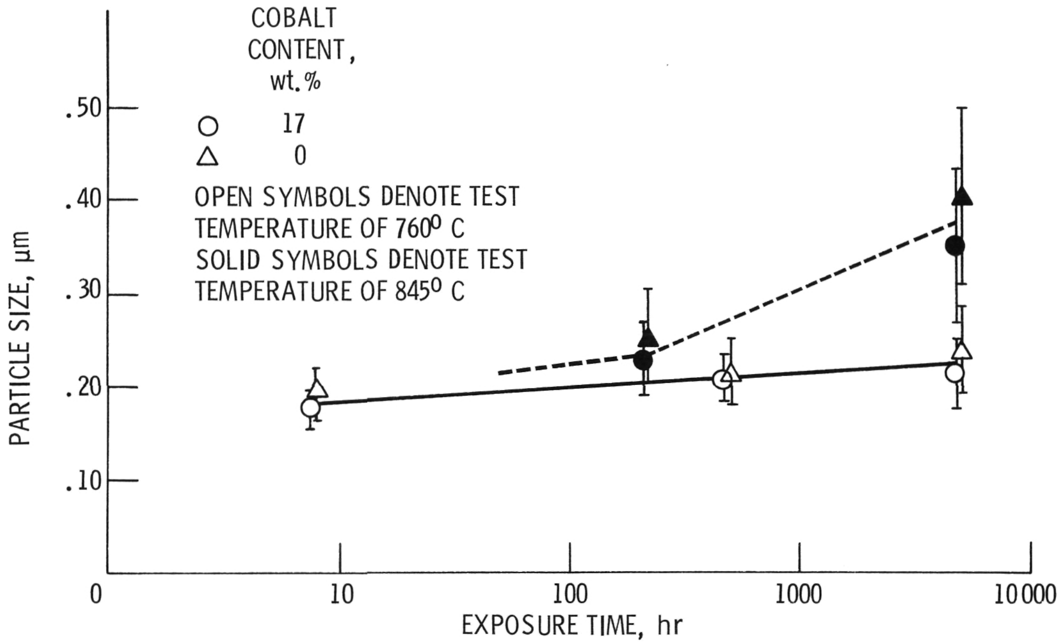

Figure 6. - Effect of exposure on microstructures of HIP-PM Udimet 700 with 17 and 0% cobalt contents. Graphs show growth of fine gamma prime particles during exposures to 760 and 845° C. Bars equal one standard deviation.

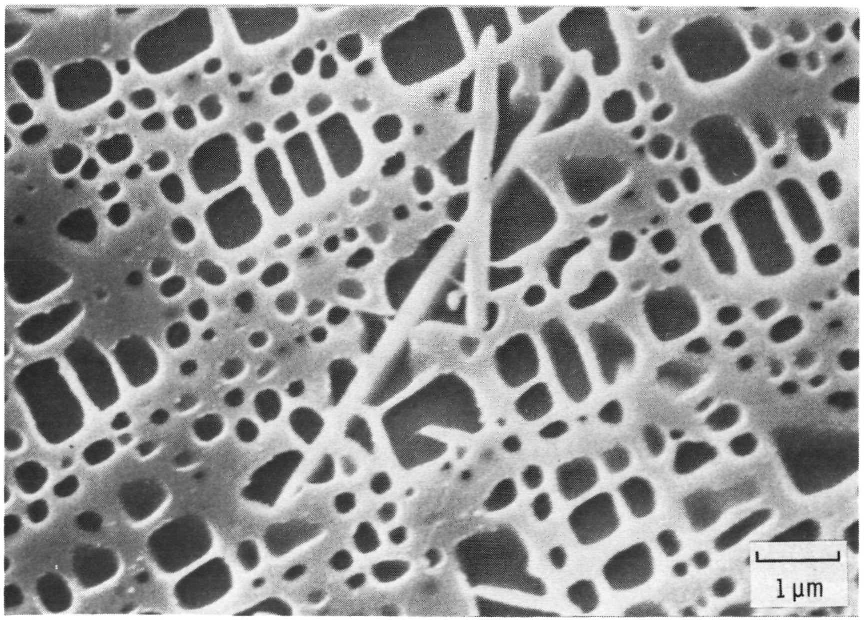

Figure 7. - Microstructure of HIP-PM Udimet 700 (17 percent cobalt) after 5000 hours exposure to 845° C showing platelets of sigma phase in a matrix of gamma with gamma prime particles.

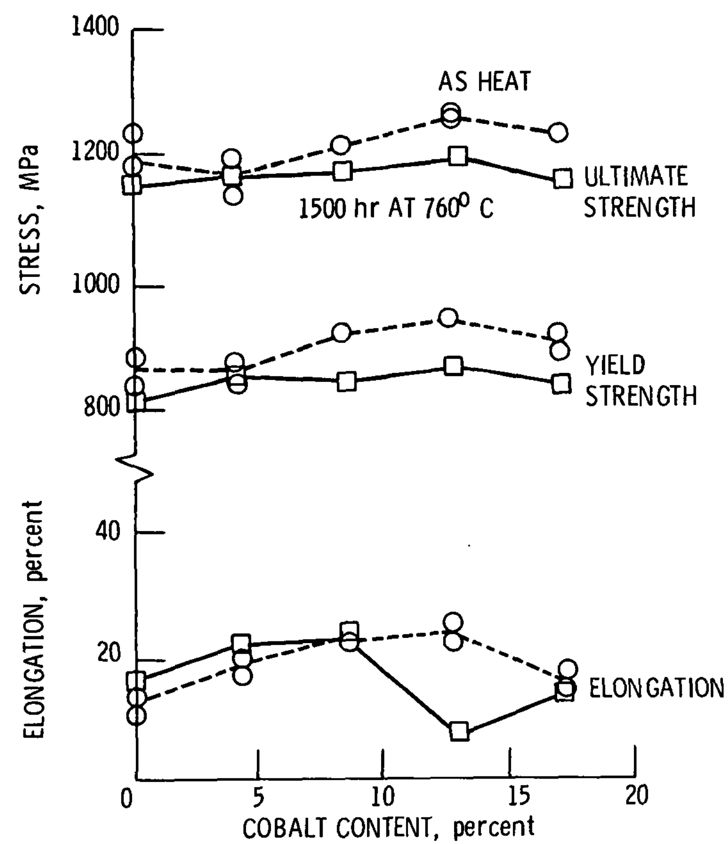

Figure 8. - 650° C tensile strength and ductilities of HIP-PM Udimet 700 alloys with various cobalt contents in the as heat-treated condition and after an exposure of 1500 hr to 760° C.

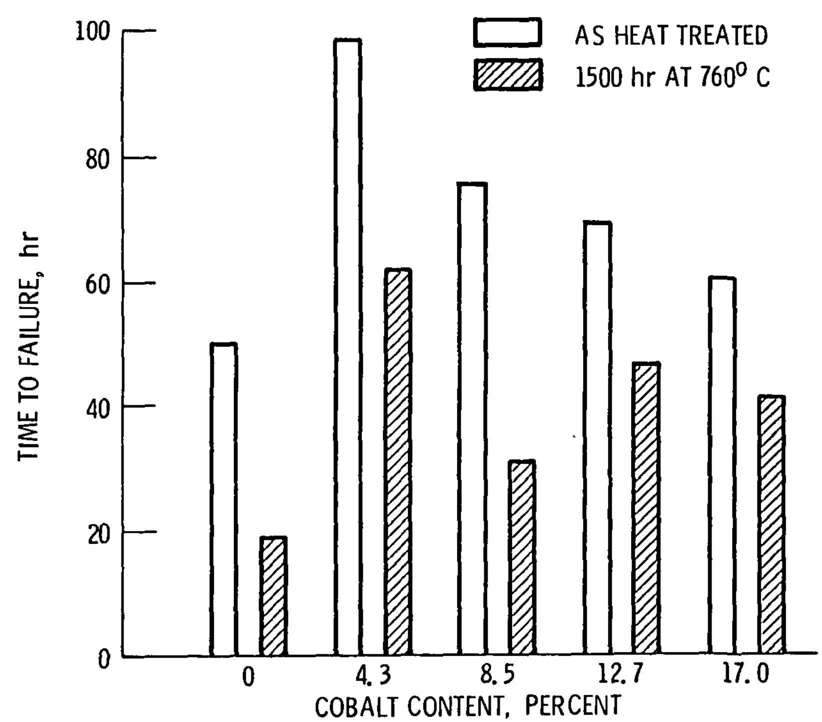

Figure 9. - Creep rupture lives of HIP-PM Udimet 700 alloys with various cobalt contents in the as heat-treated condition and after an exposure of 1500 hours to 760° C, when tested at 760° C under 475 MPa stress.

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM-83632		

4. Title and Subtitle	5. Report Date
Effects of Long-Time Elevated Temperature Exposures on Hot-Isostatically-Pressed Powder-Metallurgy Udimet 700 Alloys with Reduced Cobalt Contents	
	6. Performing Organization Code 505-33-1A
7. Author(s) Fredric H. Harf	8. Performing Organization Report No. E-2071
	10. Work Unit No.
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135	11. Contract or Grant No.
	13. Type of Report and Period Covered Technical Memorandum
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546	14. Sponsoring Agency Code

15. Supplementary Notes

Prepared for the One hundred thirteenth Annual Meeting of the American Institute of Mining, Metallurgical and Petroleum Engineers, Los Angeles, California, February 26-March 2, 1984.

16. Abstract

Because almost the entire U.S. consumption of cobalt depends on imports, this metal has been designated "strategic." To achieve conservation, the role and effectiveness of cobalt is being evaluated in commercial nickel-base superalloys. Udimet 700 type alloys in which the cobalt content had been reduced from the normal 17 percent to 12.7, 8.5, 4.3, and 0 percent were prepared by standard powder metallurgy techniques and hot isostatically pressed into billets. Mechanical testing and microstructural investigations were performed. The mechanical properties of the alloys with reduced cobalt contents which were heat-treated identically were equal or better than those of the standard alloy, except that creep rates tended to increase as cobalt was reduced. The effects of long time exposures at 760° C on mechanical properties and at 760 and 845° C on microstructures were determined. Decreased tensile properties and shorter rupture lives with increased creep rates were observed in all alloy modifications. The exposures caused gamma prime particle coarsening and formation of sigma phase in the alloys with higher cobalt contents. Exposure at 845° C also reduced the amount of MC carbides.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Heat resistant alloys; Nickel alloys; Cobalt; Aging (metallurgy); Mechanical properties; Microstructure; Udimet alloys	Unclassified - unlimited STAR Category 24

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price*
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