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Analyses of Elemental Partitioning in Advanced Nickel-Base Superalloy Single Crystals

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ANALYSES OF ELEMENTAL PARTITIONING IN ADVANCED NICKEL-BASE SUPERALLOY SINGLE CRYSTALS

by Robert L. Dreshfield and Kimberly J. Thomas

ABSTRACT

Aircraft propulsion engines for the High Speed Civil Transport which may be developed early in the 21st century will require significantly different durability requirements than those which currently power civil aircraft. The durability will be more difficult to achieve because it is expected that the new aircraft engines will have to operate at near maximum power for more than half of each flight compared to 5 - 10% for typical current aircraft.

To meet this requirement, a team of NASA, Pratt & Whitney Aircraft and General Electric personnel have been formed to develop an appropriate alloy for the mission. This report summarizes the work performed by a part of that team up to the retirement of one of its members (R. L. Dreshfield). The prime purpose of the report is to assemble the data obtained in a single document so that it may be more accessible to those who may wish to pursue it at a later date.

INTRODUCTION: Propulsion engines for the High Speed Civil Transport which may be developed early in the next century have durability requirements significantly different from the engines which power commercial jet aircraft. The more difficult durability requirements are the result of the high power required to maintain supersonic speed during the cruise phase of the aircraft's mission. Thus the turbine exit temperature is near its maximum for more than half of each flight, compared to 5 - 10% for typical current subsonic commercial aircraft.

To meet the requirements for an economical, durable propulsion system for the High Speed Civil Transport NASA, General Electric and Pratt & Whitney Aircraft have teamed to develop an advanced single crystal superalloy. As a part of that development program, it was deemed desirable to evaluate the effects of altering chemical composition on the chemistries and relative amounts of the γ and γ' phases of some of the alloys evaluated in the program. It was thought that this information would give the development team additional insight and assist in developing a suitable alloy. This report is a summary of the work performed to analyze the amount and chemical compositions of alloys selected from those prepared for the overall blade alloy development program. As the work was still in progress when one author (RLD) retired, the primary intent of this report is to archive the work that had been done to date for the benefit of those who wish to refer to it or perhaps continue the study in the future.

EXPERIMENTAL TECHNIQUES: The alloys were received as pieces which had been cut from the original slab castings. They had been given a heat treatment consisting of a solution treatment which was typically 4 hours at 1315 or 1327°C followed by a simulated coating treatment of 4 hours at 1079°C followed by an intermediate temperature age which was typically 12 hours at 871°C. Small pieces (typically 1 X 2 X 0.5 cm) were cut using an abrasive cut-off wheel. Uncut surfaces were then ground using coarse (180 grit) paper to remove the as-heat treated surfaces. The specimens were then subjected to electrolytic extractions and analyses to determine the compositions of the gamma prime and gamma phases as described in Appendix A.

The carbide extractions were performed first using procedures similar to those recommended in ASTM E 963-95 (1). After the carbides extraction was performed, gamma prime was extracted using procedures similar to those of Kriege and Baris (2). X-ray diffraction and chemical analyses were performed on the extracted residues. Chemical analyses were also performed on the residual liquor from the gamma prime extractions. It was assumed that the composition of the liquor represented the composition of the gamma phase.

<u>RESULTS AND DISCUSSION</u>: The bulk chemical analyses of the alloys studied were performed by the contractor team and are listed in Table 1.

Table 1
Composition of Alloys (w/o)

Alloy	Al	Co	Cr	Mo	Nb	Re	Ru	Ta	Ti	W	Hf
5	5.7	12.6	1.5	2.0	0.0	6.3	0.0	7.7	0.0	7.7	0.0
6	5.7	12.6	1.5	2.0	0.0	6.2	0.0	7.9	0.0	6.7	0.0
7	5.6	12.6	3.0	2.0	0.0	6.1	0.0	8.1	0.0	7.8	0.0
6a	5.5	12.5	1.5	2.0	0.0	6.3	0.0	7.8	0.0	6.7	0.0
11	5.4	15.0	1.4	2.0	0.0	6.2	0.0	7.8	0.0	6.8	0.0
12	5.4	19.9	1.5	2.0	0.0	6.1	0.0	7.8	0.0	6.7	0.0
13	5.4	19.9	1.5	2.1	0.0	5.4	0.0	8.0	0.0	7.0	0.0
14	5.5	19.5	1.5	2.0	0.0	5.4	0.0	7.9	0.0	8.3	0.0
15	5.4	20.0	1.5	2.0	0.0	6.5	0.0	8.0	0.0	7.1	0.0
16	5.7	19.7	1.5	2.0	0.0	6.3	0.0	8.0	0.0	7.9	0.0
17	5.5	12.3	4.2	2.0	0.0	5.7	0.0	8.4	0.5	5.4	0.0
19	5.6	12.3	4.2	2.0	0.0	5.0	0.0	8.4	0.5	6.3	0.0
21	5.5	12.2	2.8	2.0	0.0	5.7	1.6	8.3	0.5	5.6	0.0
23	5.6	12.3	3.0	2.0	0.0	5.2	1.5	8.2	0.5	6.3	0.0
25	5.5	20.1	1.5	2.0	0.0	5.5	1.6	7.9	0.0	6.9	0.0
26	5.5	20.2	1.5	2.0	0.0	6.5	1.6	7.8	0.0	7.8	0.0
27	5.6	19.7	1.5	2.0	0.0	6.0	3.0	8.0	0.0	6.9	0.0
28	5.6	12.3	1.5	2.0	0.0	6.1	1.0	7.9	0.0	7.0	0.0
29	5.5	19.6	1.5	2.0	0.0	6.2	1.0	7.9	0.0	5.9	0.0
30	5.6	12.5	1.5	2.0	0.0	6.3	3.1	8.0	0.0	5.9	0.0
31	5.7	19.9	1.5	2.0	0.0	5.7	3.1	8.1	0.0	6.9	0.0
32	5.5	12.3	1.5	2.0	0.0	5.6	1.0	7.9	0.0	7.0	0.0
33	5.6	19.6	1.5	2.0	0.0	5.7	1.0	7.8	0.0	6.0	0.0
34	5.4	12.5	1.5	2.0	0.0	5.7	3.1	8.0	0.0	5.8	0.0
21a	5.4	12.2	2.3	2.3	0.0	5.5	1.4	8.0	0.4	5.5	0.0
35	5.4	5.0	2.4	2.1	0.0	5.8	4.4	8.0	0.4	5.8	0.0
36	5.5	20.0	2.3	2.1	0.0	5.9	3.1	8.4	0.4	5.5	0.0
37	5.5	5.0	2.2	2.0	0.0	5.7	1.5	8.0	0.4	5.3	0.0
38	5.3	12.5	2.3	2.0	0.0	5.7	3.0	8.3	0.4	5.4	0.0
39	5.4	19.7	2.3	2.1	0.0	5.7	4.6	8.3	0.4	5.6	0.0
40	5.4	5.1	2.2	2.0	0.0	5.8	2.9	8.1	0.4	5.4	0.0
41	5.5	19.8	2.3	2.0	0.0	5.8	1.5	8.2	0.4	5.4	0.0
42*	5.3	12.7	2.3	2.0	0.0	5.6	4.8	8.2	0.4	5.7	0.0
43*	5.4	12.5	2.3	2.0	0.0	5.6	3.2	8.0	0.0	6.1	0.0
44	5.4	12.4	2.2	2.0	0.0	5.7	3.0	8.1	0.0	6.1	0.0
45	5.3	12.2	2.2	2.0	0.0	5.6	3.0	8.1	0.4	6.1	0.0
46	5.5	12.5	2.3	2.0	0.0	5.7	4.7	8.2	0.0	5.6	0.0
47	5.4	12.5	2.3	2.0	0.0	5.8	4.7	8.2	0.0	6.3	0.0
48	5.4	12.5	2.3	2.0	0.0	5.9	4.6	8.1	0.4	6.3	0.0
53	5.1	13.9	1.8	2.0	0.0	6.7	2.9	7.5	0.0	5.6	0.0
54	4.8	14.6	1.8	2.1	0.0	7.4	2.9	7.1	0.0	5.6	0.0
55	5.6	12.8	1.6	0.0	0.0	5.8	4.4	8.4	0.0	6.0	0.0
56	6.1	12.8	1.6	0.0	0.0	5.8	4.5	8.4	0.0	4.1	0.0

57	5.5	12.7	4.2	0.0	0.0	5.8	4.5	8.4	0.0	4.1	0.0
58	6.0	12.8	4.2	0.0	0.0	5.8	4.5	8.4	0.0	6.0	0.0
59	5.6	12.8	1.6	2.1	0.0	5.8	4.5	8.4	0.0	4.0	0.0
60	6.1	12.8	1.6	2.1	0.0	5.8	4.4	8.5	0.0	6.0	0.0
61	5.6	12.7	4.1	2.1	0.0	6.0	4.5	8.4	0.0	6.0	0.0
62	6.0	12.8	4.2	2.1	0.0	5.8	4.5	8.4	0.0	4.1	0.0
63	6.0	5.2	1.6	2.1	0.0	5.6	4.5	8.4	0.0	6.0	0.0
64	5.5	5.2	4.1	2.1	0.0	5.8	4.5	8.4	0.0	6.0	0.0
31R	5.6	20.6	1.5	2.1	0.0	6.1	2.9	8.0	0.0	7.1	0.0
77*	5.78	21.0	1.4	2.1	0.0	5.8	6.0	8.3	0.0	6.0	0.2
78	5.18	20.4	1.5	2.0	0.0	5.8	6.0	8.1	0.0	3.0	0.2
79	5.4	21.0	1.5	2.1	0.0	6.1	3.0	7.9	0.0	3.0	0.2
80	5.69	19.8	1.5	2.1	0.0	6.1	3.3	7.9	0.0	3.3	0.2
Rene' N5	6.2	7.6	6.9	1.5	0.0	3.1	0.0	6.3	0.0	5.2	0.0
PWA 1484	5.5	10.0	5.0	1.9	0.0	3.2	0.0	8.3	0.0	6.2	0.0
"K"	5.1	1.0	6.4	4.3	0.0	0.0	0.0	7.3	0.0	7.3	0.0
"MC-2"	5.0	4.9	7.8	2.0	0.0	0.0	0.0	6.1	1.6	7.7	0.0

* Samples became passive during gamma prime extraction.

Most of the compositions were part of designed experiments which were intended to determine the effects of specific elemental changes, however not all of the alloys melted for those studies were evaluated in this work. The designed experiments are summarized in Table 2. Shown in Table 2 are the alloys in the design and the effects intended to be studied in that design.

Table 2
Summary of Designed Experiments

Alloy Numbers	Variables
6a, 11, 12	Co
13 - 16	W, Re
17 - 24	W, Re, Ru, Ti
13 - 16, 25 - 26,	Ru, W, Re
27 -34	Co, W, Re, Ru
35 - 42	Co, Ru
38, 42 - 48	W, Ru, Ti
55 - 62	W, Mo, Al, Cr
60 , 61, 63, 64	Co, Cr
31, 65 - 77	Co, Mo, Ru
34, 53 , 54	amount gamma prime
76, 78 - 82	Al, Ta

Gamma Prime Amount

The amount of gamma prime extracted with the ammonium sulfate electrolyte are shown in Table 3. Only alloys 19, 27 and 28 showed x-ray diffraction evidence of phases other than gamma and gamma prime present in the "as heat treated" condition present in the extract from the HCl base electrolyte. Also shown in Table 3 is the amount of gamma prime which was calculated by using a mass balance based on the chemical analyses of the alloy, the gamma and gamma prime (3). The details of this calculation are described in Appendix B. In general the two values are considered to be in good agreement, thus adding credibility to the values. It should be noted that there is no accepted standard technique for quantitative extraction of gamma prime from nickel-base superalloys. Efforts to confirm these data by use of quantitative metallography proved inconclusive because of segregation in the alloys. The interdendritic regions were found metallographically to contain significantly greater amounts of gamma prime than the dendrite cores. As of this writing, no satisfactory technique was found to integrate the two regions (dendrite core and interdendritic) to obtain a value comparable to the extraction technique which gives a bulk average amount of gamma prime.

It was also observed that some alloys became passive during the gamma prime extraction which made it impossible to obtain quantitative extractions. The alloys which became passive tended to be ones which were given long high temperature exposures intended to assess the stability of the alloys or to grow the gamma prime to sizes more compatible with microscopic analytical tools such as EDS analysis. These exposures typically involved holding the sample at temperatures greater than 980°C for times greater than 100 hours. Also some of the alloys containing Ru which had received only the normal heat treatment became passive during the gamma prime extraction procedure, making it impossible to obtain a quantitative extraction from those alloys.

Table 3
Amount of Gamma Prime (w/o)

ALLOY	MASS BALANCE	EXTRACTION
N 5	Not performed	62.7
1484	Not performed	63.4
5	Not performed	62.4
6	Not performed	67.9
7	Not performed	61.9
6a	66.8	71.2
11	68.4	71.6
12	68.5	70.5
13	72.4	71.3
14	73.3	71.6
15	73.0	71.3
16	70.5	73.0
17	69.1	71.6
19	69.3	70.1
21	68.9	73.3
23	68.5	73.4
25	69.7	73.9
26	64.0	66.5
27	71.9	66.6
28	72.8	63.0
29	73.7	70.6
30	72.6	67.5
31	70.7	69.4
32	71.9	66.0
33	74.4	71.4
34	73.9	73.0
'21a	73.9	72.4
35	77.1	57.1
36	71.9	63.5
37	71.1	63.3
38	73.4	60.4

39	67.3	69.2
40	74.0	64.5
41	74.2	72.0
44	73.5	73.2
45	67.9	69.0
46	68.5	74.2
47	69.1	62.6
48	67.1	71.4
53	60.6	63.4
54	56.1	59.8
55	66.2	67.3
56	66.8	68.6
57	62.2	63.2
58	70.6	73.8
59	65.4	66.3
60	70.9	70.8
61	66.3	70.0
62	69.2	73.2
63	69.2	71.7
64	65.0	66.4
31R	70.1	70.7
77	66.7	63.4
78	67.7	67.9
79	69	68.7
80	64	64

Several attempts were made to find a correlation (regression analyses) between the amount of gamma prime and the chemical composition of the bulk alloy. No statistically significant correlations were found.

Gamma Prime and Gamma Chemistry

The chemical analyses, in atomic percent , for the extracted gamma prime are shown in Table 4 and for the gamma in Table 5.

Table 4

Composition of gamma prime of Alloys (a/o)

Alloy	Al	Co	Cr	Mo	Ni	Re	Ru	Ta	Ti	W
N5	17.0	5.1	4.1	0.7	67.0	0.2	0.0	3.6	0.0	2.3
1484	15.9	6.5	1.8	0.8	66.2	0.4	0.0	5.6	0.0	2.9
5	16.6	9.5	1.0	0.9	64.9	0.4	0.0	4.0	0.0	2.7
6	16.8	9.2	1.0	0.4	65.6	0.5	0.0	4.0	0.0	2.4
7	17.1	99.0	0.8	1.4	63.4	0.4	0.0	4.4	0.0	2.5
6a	14.8	8.9	0.8	1.0	67.9	0.4	0.0	3.9	0.0	2.3
11	14.8	11.0	0.8	1.0	65.6	0.5	0.0	4.0	0.0	2.4
12	15.1	15.9	0.8	1.1	60.4	0.5	0.0	3.9	0.0	2.3
13	15.0	16.2	0.9	1.1	55.5	0.5	0.0	4.0	0.0	2.4
14	15.1	16.8	0.9	1.1	59.0	0.5	0.0	3.7	0.0	2.9
15	14.3	16.8	1.0	1.1	59.4	0.6	0.0	4.0	0.0	2.7

16	15.9	16.0	0.8	1.1	59.2	0.5	0.0	3.7	0.0	2.8
17	16.3	8.6	1.5	1.0	65.9	0.3	0.0	3.9	0.8	1.9
19	16.2	9.1	1.5	0.9	65.1	0.3	0.0	4.0	0.8	2.1
21	12.7	8.9	1.0	1.3	65.7	0.4	0.8	4.1	2.8	2.4
23	12.6	9.2	1.0	1.3	65.3	0.3	0.9	4.0	2.6	2.6
25	16.1	15.8	1.2	1.2	58.0	0.6	0.9	3.8	0.0	2.5
26	15.7	16.4	1.1	1.4	55.8	0.6	1.0	4.3	0.0	3.6
27	16.4	16.9	1.2	1.1	55.3	0.7	2.1	3.7	0.0	2.4
28	17.0	9.3	1.0	0.9	64.8	0.5	0.6	3.7	0.0	2.2
29	16.6	15.8	1.0	1.0	58.6	0.6	0.5	3.8	0.0	2.0
30	16.8	9.1	1.3	1.0	63.0	0.5	2.3	3.8	0.0	2.1
31	16.1	16.9	1.3	1.2	55.2	0.7	2.0	4.1	0.0	2.5
32	16.7	9.3	1.0	1.0	64.7	0.4	0.6	4.0	0.0	2.3
33	17.8	16.1	0.9	1.0	57.3	0.6	0.6	3.7	0.0	1.9
34	18.0	9.5	1.4	1.0	61.6	0.5	2.1	3.7	0.0	2.1
21a	16.1	8.8	1.5	1.1	64.6	0.4	0.9	3.8	0.6	2.1
35	16.7	3.4	1.8	1.1	66.5	0.5	2.9	4.0	0.5	2.4
36	16.2	16.5	1.7	1.1	55.5	0.6	1.7	4.0	0.5	2.2
37	17.0	3.4	1.2	0.9	70.0	0.4	0.6	4.0	0.5	2.0
38	16.4	9.4	1.8	1.1	62.1	0.5	1.9	4.2	0.5	2.2
39	16.0	17.3	1.7	1.1	53.7	0.7	2.6	4.1	0.7	2.1
40	16.3	3.4	1.6	1.0	68.6	0.5	1.6	4.1	0.7	2.3
41	15.9	16.2	1.7	1.2	56.7	0.5	0.9	4.1	0.7	2.2
44	16.2	9.2	1.8	0.9	63.8	0.5	1.9	3.7	0.0	2.0
45	15.7	9.4	1.7	0.9	63.6	0.5	1.8	3.6	0.6	2.0
46	16.1	9.3	2.2	1.0	62.2	0.5	3.0	3.7	0.0	2.0
47	16.6	9.4	1.3	0.9	63.1	0.5	2.1	4.0	0.0	2.1
48	15.5	9.7	2.1	1.1	61.3	0.5	3.0	3.7	0.8	2.3
53	15.9	8.7	1.6	1.0	64.4	0.4	1.9	3.9	0.0	2.3
54	15.9	8.5	1.6	1.1	64.3	0.4	2.0	3.9	0.0	2.3
55	15.3	8.2	1.2	0.0	65.3	0.6	2.5	4.5	0.0	2.4
56	15.8	8.1	1.1	0.0	66.2	0.6	2.4	4.3	0.0	1.6
57	15.3	7.5	2.2	0.0	65.9	0.4	2.4	4.7	0.0	1.6
58	15.1	9.1	3.2	0.0	62.9	0.4	2.8	4.2	0.0	2.4
59	15.6	8.1	1.0	0.8	66.0	0.5	2.4	4.3	0.0	1.4
60	17.1	10.2	0.8	1.0	62.5	0.6	2.3	3.4	0.0	2.1
61	15.1	8.8	2.7	0.9	63.2	0.3	2.2	4.4	0.0	2.3
62	15.4	8.9	2.9	1.0	62.8	0.4	3.0	4.1	0.0	1.6
63	17.0	3.8	1.0	1.0	69.3	0.5	2.0	3.3	0.0	2.2
64	15.1	3.2	2.3	0.8	69.4	0.3	2.6	4.3	0.0	2.0
31R	15.9	17.0	1.5	1.2	54.9	0.7	2.2	3.8	0.0	2.8
77	15.6	18.6	1.3	1.2	51.3	1.0	4.2	4.0	0.0	2.8
78	15.6	16.2	1.3	1.1	56.5	0.7	2.0	3.8	0.0	2.6
79	15.7	17.3	1.1	1.1	55.7	0.8	1.9	3.7	0.0	2.6
80	16.2	18.1	1.0	1.1	55.0	0.9	1.7	3.5	0.0	2.5
Alloy	Al	Co	Cr	Mo	Ni	Re	Ru	Ta	Ti	W

Table 5

Composition of Gamma of Alloys (a/o)

Alloy	Al	Co	Cr	Mo	Ni	Re	Ru	Ta	Ti	W
6a	4.0	25.9	2.9	2.1	55.4	7.1	0.0	0.4	0.0	2.1
11	4.3	30.0	2.0	2.1	51.2	7.0	0.0	0.4	0.0	2.0
12	4.4	36.9	3.9	2.1	43.2	6.8	0.0	0.5	0.0	2.2
13	5.0	36.6	3.8	1.9	44.3	5.9	0.0	0.5	0.0	1.9
14	4.8	36.7	3.8	2.2	43.2	6.3	0.0	0.6	0.0	2.4
15	5.2	37.0	3.9	2.3	41.7	7.2	0.0	0.6	0.0	2.1
16	5.0	36.7	2.9	2.1	43.0	7.3	0.0	0.5	0.0	2.4
17	4.1	26.2	10.1	2.3	48.2	7.2	0.0	0.4	0.0	1.6
19	4.4	23.6	13.5	2.1	47.9	6.1	0.0	0.3	0.0	1.9
21	6.0	24.0	8.2	1.6	51.3	6.7	1.0	0.3	0.0	0.9
23	6.9	23.3	8.1	1.7	51.4	6.1	0.8	0.4	0.0	1.2
25	6.9	34.9	2.8	1.6	45.5	5.7	0.6	0.5	0.0	1.6
26	9.3	30.8	2.4	1.1	48.8	5.6	0.6	0.4	0.0	1.1
27	7.0	34.7	3.0	1.3	44.3	6.0	1.5	0.6	0.0	1.5
28	5.0	25.8	4.0	1.9	52.4	7.4	0.7	0.6	0.0	2.2
29	5.2	37.9	4.2	2.0	40.5	6.8	0.8	0.6	0.0	2.0
30	5.7	26.5	3.5	1.4	52.5	7.1	1.6	0.4	0.0	1.3
31	7.8	35.8	3.3	1.1	44.0	4.9	1.5	0.4	0.0	1.2
32	5.3	26.6	4.5	1.8	53.0	5.6	0.7	0.4	0.0	2.1
33	5.6	37.8	4.2	2.1	41.2	5.2	0.8	0.7	0.0	2.5
34	5.6	26.9	3.3	1.8	53.0	5.6	1.4	0.5	0.0	1.9
21a	4.6	27.2	6.2	1.6	51.3	6.7	0.8	0.4	0.0	1.3
35	5.3	11.4	7.4	2.2	60.2	7.2	3.8	0.4	0.3	1.8
36	6.5	36.7	6.2	1.3	39.8	6.1	1.9	0.4	0.0	1.0
37	5.4	10.3	6.8	2.2	64.7	6.1	1.4	0.8	0.0	2.3
38	6.2	25.8	6.2	1.3	51.3	6.2	1.7	0.4	0.0	0.9
39	9.3	31.5	5.1	1.2	43.0	4.7	3.1	0.8	0.1	1.2
40	5.8	10.4	6.5	2.8	62.0	5.9	2.5	1.0	0.3	2.7
41	5.8	37.5	6.0	1.4	40.7	6.4	0.7	0.4	0.0	1.2
44	6.0	24.9	4.9	1.3	54.1	5.7	1.3	0.4	0.0	1.4
45	7.0	22.8	5.7	1.5	52.9	5.4	2.3	0.6	0.1	1.7
46	7.1	24.3	4.3	1.0	54.8	5.4	2.1	0.3	0.0	0.8
47	5.9	24.0	6.1	1.6	50.6	5.5	3.9	0.5	0.0	1.9
48	8.1	22.6	4.6	1.1	54.4	5.4	2.4	0.4	0.1	1.0
53	5.4	26.4	3.1	1.5	56.1	4.6	1.2	0.3	0.0	1.5
54	5.2	26.7	2.9	1.5	56.0	4.7	1.2	0.3	0.0	1.5
55	6.6	24.5	2.4	0.0	59.0	4.3	2.6	0.1	0.0	0.3
56	7.1	24.8	2.6	0.0	57.9	4.3	2.8	0.1	0.0	0.4
57	5.7	23.7	8.4	0.0	53.9	4.3	3.2	0.1	0.0	0.6
58	6.3	25.7	9.1	0.0	49.8	5.6	2.5	0.1	0.0	0.9
59	6.1	24.4	2.9	1.0	57.2	4.4	3.2	0.2	0.0	0.7
60	7.9	23.6	4.5	2.1	50.7	5.1	2.6	1.1	0.0	2.4
61	5.9	24.4	9.2	1.4	49.7	5.2	2.4	0.2	0.0	1.4
62	6.8	24.8	9.1	1.2	49.6	5.4	2.2	0.2	0.0	0.7
63	8.0	10.0	4.3	2.2	62.9	5.0	4.0	1.1	0.0	2.5

64	6.0	10.3	9.9	1.6	61.6	5.2	3.1	0.3	0.0	1.9
31R	8.6	34.0	3.0	0.7	46.0	5.0	1.5	0.2	0.0	0.8
78	6.8	35.3	2.7	1.5	44.5	5.0	1.4	0.8	0.0	1.9
79	8.8	33.0	2.8	1.2	46.0	4.6	1.4	0.7	0.0	1.6
80	10.3	29.4	3.3	1.7	44.6	4.3	2.3	1.6	0.0	2.5

The data are, in general, consistent with values expected from earlier work and reported in various works in the open literature. Partitioning ratios for each element were calculated by dividing the concentration (atomic per cent) of each element in the gamma by its concentration in the gamma prime. Partitioning ratios greater than 1 (such as Cr and Re) tend to be in greater concentrations in the gamma phase and visa versa. The partitioning ratios for each element in each alloy are shown in Table 6.

Table 6
Gamma/ Gamma Prime Partitioning

Alloy	PAl	PCo	PCr	PMo	PNi	PRe	PRu	PTa	PTi	PW
6a	0.3	2.9	3.6	2.1	0.8	17.1	0.0	0.1		0.9
11	0.3	2.7	2.4	2.0	0.8	15.5	0.0	0.1		0.9
12	0.3	2.3	4.8	2.0	0.7	12.4	0.0	0.1		1.0
13	0.3	2.3	4.5	1.7	0.8	11.4	0.0	0.1		0.8
14	0.3	2.2	4.3	2.1	0.7	12.1	0.0	0.2		0.8
15	0.4	2.2	4.1	2.0	0.7	11.5	0.0	0.2		0.8
16	0.3	2.3	3.6	2.0	0.7	13.7	0.0	0.1		0.8
17	0.2	3.0	6.9	2.4	0.7	27.6	0.0	0.1	0.0	0.9
19	0.3	2.6	8.9	2.5	0.7	22.2	0.0	0.1	0.0	0.9
21	0.5	2.7	8.3	1.2	0.8	17.9	1.2	0.1	0.0	0.4
23	0.5	2.5	7.9	1.3	0.8	18.2	1.0	0.1	0.0	0.5
25	0.4	2.2	2.4	1.3	0.8	10.2	0.6	0.1		0.6
26	0.6	1.9	2.1	0.8	0.9	8.8	0.6	0.1		0.3
27	0.4	2.0	2.5	1.1	0.8	8.5	0.7	0.2		0.6
28	0.3	2.8	4.2	2.1	0.8	15.9	1.1	0.2		1.0
291	0.3	2.4	4.4	1.9	0.7	11.3	1.7	0.2		1.0
30	0.3	2.9	2.6	1.4	0.8	13.1	0.7	0.1		0.6
31	0.5	2.1	2.4	0.9	0.8	7.5	0.7	0.1		0.5
32	0.3	2.9	4.6	1.9	0.8	12.9	1.1	0.1		0.9
33	0.3	2.3	4.5	2.1	0.7	8.8	1.3	0.2		1.3
34	0.3	2.8	2.3	1.7	0.9	10.6	0.7	0.1		0.9
'21a	0.3	3.1	4.0	1.5	0.8	16.7	0.9	0.1	0.1	0.6
35	0.3	3.3	4.0	2.1	0.9	14.2	1.3	0.1	0.5	0.8
36	0.4	2.2	3.7	1.1	0.7	10.1	1.1	0.1	0.0	0.5
37	0.3	3.1	5.7	2.4	0.9	15.4	2.4	0.2	0.0	1.1
38	0.4	2.7	3.4	1.2	0.8	12.3	0.9	0.1	0.0	0.4
39	0.6	1.8	3.0	1.1	0.8	6.8	1.2	0.2	0.2	0.6
40	0.4	3.1	4.2	2.9	0.9	12.5	1.6	0.2	0.4	1.2
41	0.4	2.3	3.5	1.2	0.7	11.9	0.7	0.0	0.0	0.5
44	0.4	2.7	2.7	1.5	0.8	12.1	0.7	0.1	0.0	0.7
45	0.4	2.4	3.4	1.7	0.8	11.5	1.3	0.2	0.2	0.8
46	0.4	2.6	2.0	0.9	0.9	10.7	0.7	0.1		0.4
47	0.4	2.6	4.6	1.8	0.8	10.2	1.8	0.1		0.9
48	0.5	2.3	2.2	1.0	0.9	10.6	0.8	0.1	0.1	0.4
53	0.4	2.9	1.5	1.3	0.9	11.0	1.4	0.1		0.7
54	0.3	3.2	1.3	1.8	0.9	14.1	2.2	0.1		0.9
55	0.4	3.0	2.0	0.0	0.9	7.8	1.0	0.0		0.1
56	0.4	3.1	2.4	0.0	0.9	7.1	1.2	0.0		0.2

57	0.4	3.2	3.8	0.0	0.8	9.8	1.3	0.0	0.4
58	0.4	2.8	2.9	0.0	0.8	13.6	0.9	0.0	0.4
59	0.4	3.0	3.0	1.2	0.9	8.7	1.3	0.1	0.5
60	0.5	2.3	4.1	2.0	0.8	9.4	1.3	0.2	1.0
61	0.4	2.8	3.4	1.5	0.8	15.2	1.1	0.1	0.6
62	0.4	2.8	3.1	1.3	0.8	14.6	0.7	0.0	0.5
63	0.5	2.5	4.0	1.9	0.9	10.5	1.4	0.4	1.1
64	0.4	3.2	4.3	2.0	0.9	19.0	1.2	0.1	0.9
31R	0.5	2.0	2.0	3.0	0.8	7.1	0.7	0.1	0.3
78	0.4	2.2	2.1	1.4	0.8	7.1	0.7	0.2	0.7
79	0.6	1.9	2.5	1.9	0.8	5.8	0.7	0.2	0.6
80	0.6	1.6	3.3	1.5	0.8	4.8	0.9	0.4	1.0

It can be seen that the partitioning ratio is not constant for a given element in all alloys. The elements Al, Ta, Ni and Ti tend to partition more to the gamma prime phase, while Cr, Co, Mo, and Re tend to partition more to the gamma . Ru and W appear to partition to either phase depending on the composition of the alloy. Re, Cr and Co usually have the greatest ratios, while Ta, Ti and Al have the smallest ratios.

The linear relation between the elemental partitioning ratios and the concentration (atom percent) were analyzed using multiple linear regression analyses. Both the "Best Subsets" and "stepwise linear regression" routines were used to analyze the design of experiment sets. Only designed variables were included in each of the analyses. The results of the linear regression analyses are summarized in Table 7.

Table 7

Alloy	Al	Co	Cr	Mo	Re	Ru	Ta	Ti	W
PAl	+	+/-	-			+	-	+	+
PCo	+	-				-		-	-
PCr		-	+			-			
PMo			+			-			
PRe	-	-	+	+	+	-		-	+
PRu	+	-	-						-
PTa									
PTi		-	+			+			
PW	+	-		+		-			+

Where increasing the concentration of an element increased the elemental partitioning ratio (Px), a plus sign (+) is shown in the table. Conversely a minus sign (-) indicated that increasing the elements' concentration in the alloy decreased the elemental partitioning ratio. In one case, the effect of Co on PAl, some analyses indicated a positive response and others a negative one. This depended on the details of the specific designed experiment, but for the purpose of this report, one might assume that the likely response of the aluminum partitioning as a result of changing cobalt is likely to be small. Where no entry is shown, no significant response was observed in any of the several experiments performed. This should not be interpreted as implying that there is no response because some elements were not varied over a large range in any of the experiments.

Of interest is the observation that only Co and Ru decrease the partitioning of the refractory metals. In other words the amount of the refractory metals in the gamma prime is increased relative to the amount in the gamma as the Co or Ru in the alloy are increased. The effect of Ru had been previously noted by O'Hara (4).

CONCLUDING REMARKS: A number of alloys in an experimental alloy development program were subjected to phase extraction experiments to assist in the alloy development program. The results of the extraction analyses are summarized in the above report.

The results from this work were used qualitatively to assist in the development of a new series of alloys for application in a high speed commercial transport. The results aided in selecting alloy additions which might affect the amount of gamma prime and the relative alloying of the phases. As of this writing, the alloy development is still in progress.

Because this is an incomplete study, the reader is cautioned concerning drawing conclusions from the data. It does appear however that Ru and Co cause the refractory elements to partition more to the gamma prime as the amount of Ru and Co are increased. As has been often noted in earlier reports, Re and Cr partition strongly to the gamma, while Ta, Ti and Al partition strongly to the gamma prime.

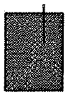
Unfortunately, in the alloys studied, it was observed that some of the Ru bearing alloys became passive in the extraction process. No satisfactory electroytes nor electrolysis conditions were found to overcome the problem This is an area of study which warrants further study. It was also noted that the quantitative nature of the extraction process could not be completely validated for the cast alloys studied. In the normally heat treated conditions, there was sufficient segregation to preclude simple quantitative metallographic analyses. When several alloys were given prolonged heat treatments at high temperatures to reduce segregation and enhance gamma prime growth, the alloys became passive in the extraction process, again preventing validation of the process. Thus validation of the quantitative nature of the extraction process in this class of alloys might also warrant further study.

APPENDIX A
Extraction and Chemical Analyses Procedure

APPARATUS: A glass beaker of about 400ml capacity, a platinum basket that surrounds the sample and serves as a cathode, and the sample hanging from a platinum wire that is the anode. A power supply that can be set at a constant current.

SAMPLE PREPARATION: Polish all sides of the alloy until a clean surface has been achieved. This can be done by hand with emery paper, using a 320 or 400 grit and finishing with 600 grit. The corners should also be rounded, to eliminate all activation sites.

Weld a piece of platinum wire to the smallest side of the sample (usually one of the ends). Set the spot welder at maximum power, and tack the wire in at least 3 places. This will give sufficient adherence to the metal. The platinum wire should be bent upwards so that the sample is hanging from wire.



REAGENTS:

CARBIDE ELECTROLYTE: Add and mix 1 part 12N Hydrochloric acid to 9 parts Absolute Methanol by volume to make a 10% HCl-methanol solution. Do this slowly with stirring since much heat is generated in this process. When the alloy contains W, Nb, Ta, Hf, add one part by weight tartaric acid to 100 parts by volume to the above 10% HCl-Methanol mixture, this will give a 1% Tartaric Acid in 10% HCl-Methanol solution.

GAMMA, GAMMA PRIME ELECTROLYTE: Add and mix 1 gm Ammonium Sulfate and 1 gm Citric Acid to 100 ml deionized water.

CARBIDE FILTER PAPER SOAKING ELECTROLYTE: Add and mix 1 part Absolute Methanol and 1 part Hydrochloric Acid by volume.

CONSTANT CURRENT ELECTROLYSIS: To achieve a constant current electrolysis a Hewlett-Packard System power supply was used. To set the power supply to a constant current the sample is setup and the leads are connected so that the basket is acting as a cathode (-) and the sample is the anode (+). The power is turned on ,and the CV (normal) light will be lit. Turn the large knob (clockwise) until the desired current is reached, then hit the button to go from voltage to current, turn the large knob counterclockwise until the light goes from CV(normal) to CC(limit). Then set the desired current and the current will be held constant.

CARBIDE DETERMINATION: The carbide phase is determined first, before the gamma, gamma prime phase. Set up the apparatus as shown in the diagram as described. Do not submerse the end with the platinum wire into the electrolyte solution. The current for the carbide phase is determined by finding the surface area and multiplying by 100 ma/cm^2. Using the carbide electrolyte, a cleaning extraction should be done first. Using only enough electrolyte to cover the sample and platinum basket, this extraction is run for about 15 minutes. The sample is then cleaned in methanol in the ultrasonic bath and dried in the oven for 30 minutes, cooled to room temperature and sandblasted. After sandblasting the sample is rinsed in water with a final rinsing in deionized water, dried in the drying oven, cooled to room temperature and an initial weight is taken.

The carbide phase electrolysis should take about 4 hours to dissolve 2-4g of sample. The extraction is setup like the cleaning extraction. After completion of the extraction, the electrolyte is filtered through a tared 0.1μm FH filter, rinsing the carbide phase now on the filter paper only with methanol. Multiple cleanings with methanol in the ultrasonic bath are done until no more residue is visibly coming off. All of these rinsings are also filtered onto the same filter paper since the residue is considered the carbide phase.

Dry sample and filter paper in the drying oven . Let both the carbide phase filter paper and sample cool to room temperature and weigh. After this weight is taken, the sample is sandblasted to remove any remaining carbide residue. After sandblasting the sample is thoroughly rinsed with tap water and a final rinsing with deionized water. The sample is then redried in the drying oven for 30 minutes and let cool to room temperature. This is the final weight of the sample.

In some instances the carbide phase now located on the filter paper will be contaminated with gamma/gamma prime. This will be observed in the x-ray diffraction pattern. To reduce the gamma/gamma prime, the filter paper can be soaked in the carbide filter paper soaking solution overnight, or during the day on low heat on the hot plate. When done soaking, the solution is filtered back onto the filter paper. This should help give a cleaner carbide x-ray diffraction pattern.

Inductively coupled plasma (ICP) elemental analysis can be done on this carbide phase, but was not done routinely in this study.

γ, γ' DETERMINATION: The gamma, gamma prime extraction is usually done after the carbide extraction. If a carbide extraction has been done then the gamma, gamma prime extraction can be done without further preparation. Measure the surface area of the sample using all side minus the end with the platinum wire. Multiplying the surface area by 75mA/cm2 gives the current at which to run the extraction. If a previous carbide extraction has not been done then a 15 minute cleaning extraction is done first. To do a cleaning extraction setup the apparatus as described and run an extraction for about 15 minutes in the gamma, gamma prime electrolyte. Then clean the sample in the ultrasonic bath with water. Dry completely in the drying oven and sandblast. When done sandblasting rinse well with water with a final rinsing with deionized water, dry in the drying oven, let cool to room temperature and weigh. This is the initial weight.

After the initial weight, setup the apparatus as described on the following page. The gamma, gamma prime extraction is run for 4 hours at the current determined above, this time can be reduced if large amounts of gamma prime are seen coming off of the sample. Using the gamma, gamma electrolyte take about 300 ml being careful not to submerse the welded end into the solution .

When done extracting the solution is vacuum filtered through a tared filter paper. Usually a tared FH Teflon filter (.45μm pore size).

The sample is put into a beaker (25ml) and cleaned in the ultrasonic bath with deionized water. It can take multiple cleanings in the ultrasonic bath to get the gamma prime off of the sample, it is very adherent. All of the wash is also filtered onto the sample filter paper. When the cleanings are done, the sample and filter paper are dried in the drying oven.

The solution is assumed to be gamma, and the precipitates on the filter paper gamma prime. After the sample has dried for 30 minutes it is cooled to room temperature. It is then weighed. After weighing it is sandblasted to remove any more gamma prime. Then rinse the sample well with tap water and a final rinsing with deionized water. Dry the sample in the drying oven for 30 minutes and both cool to room temperature and weigh.

The gamma prime phase is located on the filter paper, after weighing may be sent for x-ray diffraction, and the gamma phase in solution should be analyzed on the ICP within 24 hours.

The gamma prime is returned after the x-ray diffraction pattern is determined and the sample dissolved off of the filter paper and analyzed for elemental composition with the ICP. The gamma prime can be dissolved off of the filter paper with 3-5ml of 12:5 HF:HNO_3, after the solution has come to a boil, the filter paper is removed after being rinsed with 1-2ml of HCl.

Ruthenium in the sample is analyzed with atomic absorption due to the poor sensitivity of the ICP for Ru. An aliquot of the sample (gamma or gamma prime) is taken and made to bring the final solution to a concentration of 2M HCl, 0.09 H_2SO_4, and 0.02M KCN. This matrix helps to stabilize the ruthenium in solution and enhance the molar absorptivity.

APPENDIX B
Mass Balance Determination of Weight Fraction of Gamma Prime from Chemical Analyses

Consider a two element phase diagram such as that presented in Figure -1. From the phase diagram the concentration of A and B in α (C_α^A and C_α^B) and β (C_β^A and C_β^B) can be determined at any temperature T. The concentration of A and B in the alloy (C_{Alloy}^A and C_{Alloy}^B) are also known. The reverse lever rule can be used to determine the weight fraction of β, f_β, present. This yields two equations,

$$f_\beta = \frac{C_{Alloy}^A - C_\alpha^A}{C_\alpha^A - C_\beta^A} \qquad [-1]$$

$$f_\beta = \frac{C_{Alloy}^B - C_\alpha^B}{C_\alpha^B - C_\beta^B} \qquad [-2]$$

The equations can be rearranged to the following

$$f_\beta(C_\alpha^A - C_\beta^A) = (C_{Alloy}^A - C_\alpha^A) \qquad [-3]$$

$$f_\beta(C_\alpha^B - C_\beta^B) = (C_{Alloy}^B - C_\alpha^B) \qquad [-4]$$

In this form it is obvious that the equations describe a straight line with X axis being $C_\alpha^x - C_\beta^x$ and the Y axis being $C_{Alloy}^x - C_\alpha^x$. Here x is either element A or B depending on which equation is used. It is desirable to have the X and Y axes the same for both equations. For a two element alloy,

$$C_x^B = 1 - C_x^A \qquad [-5]$$

Substituting Equation [-5] into Equation [-4] yields Equation [-3]. Therefore a plot such as the one in Figure -2 can be constructed. For the ideal case the slope of the curve is equal to the weight fraction of the phase of interest (β) and the intercept is equal to 0. Deviations from the ideal case are an indication of how inaccurate the measurements of the various concentrations are.

For a more complex, *n*-element alloy, the same concepts apply. In the narrowly defined case of the alloy having only two phases, Equations [-3] and [-4] can be extended to the generalized form of

$$f_\beta(C_\alpha^x - C_\beta^x) = C_{Alloy}^x - C_\alpha^x \qquad [-6]$$

and a plot with the same axes again generates a line with a slope equal to the weight fraction of the β phase and an intercept of 0.

In the case of an *n*-element alloy, the plot can also yield additional information. The line for the plot may be generated using least squares regression analysis. Deviations between the predicted and observed values should be small if the analysis of the alloy is performed with little experimental error. However, if one element is analyzed or reported incorrectly, the difference between the predicted and observed values will be larger. The regression analysis will also yield indications that the goodness of fit for the line is poor. Therefore the plot can be used to also assess the quality of the chemical analyses as well as the extraction.

Based on this analysis the weight fractions of γ' for each alloy were determined by plotting the chemical analyses as in Figure -2.

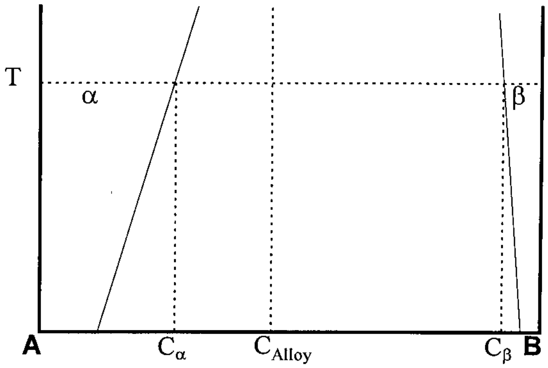


Figure -1 -
Typical Two Element Phase Diagram

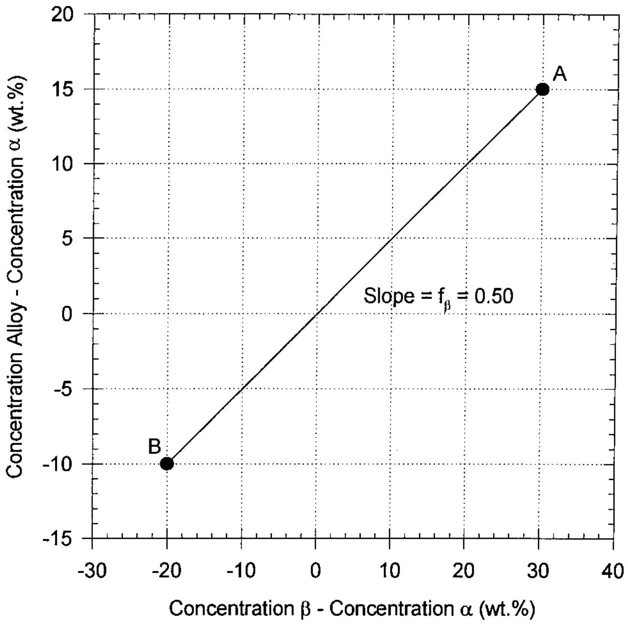


Figure -2 -
Plot Of $C^x_{Alloy} - C^x_\alpha$ Versus $C^x_\beta - C^x_\alpha$ For Two Elements

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14. ABSTRACT
Aircraft propulsion engines for the High Speed Civil Transport which may be developed early in the 21st century will require significantly different durability requirements than those which currently power civil aircraft. The durability will be more difficult to achieve because it is expected that the new aircraft engines will have to operate at near maximum power for more than half of each flight compared to 5 to 10 percent for typical current aircraft. To meet this requirement, a team of NASA, Pratt & Whitney Aircraft, and General Electric personnel have been formed to develop an appropriate alloy for the mission. This report summarizes the work performed by a part of that team up to the retirement of one of its members, R.L. Dreshfield. The prime purpose of the report is to assemble the data obtained in a single document so that it may be more accessible to those who may wish to pursue it at a later date.

15. SUBJECT TERMS
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