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DIRECTIONALLY SOLIDIFIED IN SITU
METAL MATRIX COMPOSITES

FINAL REPORT, PART II

Prepared for

Naval Air Systems Command
Contract No. N00019-74-C-0409

by

G. Haour, F. Mollard, B. Lux, and I. G. Wright

November, 1976

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This work constitutes Part II of an alloy screening program to identify new eutectics based on Ni, Co and Fe suitable for the manufacture of directionally solidified turbine blades operating at up to 1150°C. During this Part II, thirteen alloy systems, selected for screening during Part I, have been experimentally investigated, leading to the identification of three eutectics of potential interest: Co-Al-Cr, Co-Si-Nb and Co-Cr-Zr.

As a result of the overall program, Part I and Part II, a total of 9 eutectics have thus been found to meet the objectives of a melting point(Cont'd)

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above 1200°C, a specific gravity below 9 g/cm³ and a reasonably low parabolic oxidation rate (less than 10^{-7} g^2/cm^4.sec at 1150°C). They are listed below in order of decreasing oxidation resistance:

Alloy	Oxidation rate at 1150°C in flowing oxygen (g^2/cm^4.sec)	Eutectic temperature (°C)	Specific gravity (g/cm3)
Co-Al-Cr	$< 10^{-11}$	1310	7.6
Co-Si-Nb	2.6×10^{-10}	1230	8.1
Ni-Cr-Ti	10^{-8}	1220	6.6
Co-Cr-Mo	1.3×10^{-8}	1340	8.9
Co-Al-Nb	1.5×10^{-8}	1240	8.6
Co-Cr-Zr	2.4×10^{-8}	1235	7.8
Co-Cr-Nb	6.5×10^{-8}	1280	8.5
Co-Al-Ta	7.3×10^{-8}	1290	8.9
Co-Si-Zr	10^{-7}	1240	8.1

These eutectics thus have, before any development, an oxidation resistance comparable or superior to the chromium free *γ/γ'-δ eutectic superalloy Ni-2.55 Al-21.75 Nb (wt %). It is therefore expected that an alloy optimization program would improve their oxidation behavior, as compared to the γ/γ'-δ alloy with 6% chromium, presently the most advanced eutectic of this class of materials. The overall alloy development should be carried out in the order of priority given in the above list; it should include:

o Improvement of the oxidation resistance of these eutectics by adding or adjusting chromium and/or aluminum.

o Evaluation of the tensile and creep strength of these eutectics on the basis of directionally solidified samples.

o Alloy development to maximize tensile and creep strength.

Further work is also recommended to identify eutectics in more complex systems, for example alloys containing reinforcing phases of carbides, nitrides and silicides, as well as multi-constituent alloys.

* gamma/gamma' - delta

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ABSTRACT

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As a result of the overall program, Part I and Part II, a total of 9 eutectics have thus been found to meet the objectives of a melting point above 1200°C, a specific gravity below 9 g/cm^3 and a reasonably low parabolic oxidation rate (less than 10^{-7} g^2/cm^4.sec at 1150°C). They are listed below in order of decreasing oxidation resistance:

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- Improvement of the oxidation resistance of these eutectics by adding or adjusting chromium and/or aluminum.

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DIRECTIONALLY SOLIDIFIED IN SITU
METAL MATRIX COMPOSITES

by

G. Haour, F. Mollard, B. Lux and I. G. Wright

I. INTRODUCTION

The program reported here was the continuation of work carried
out for Naval Air Systems Command under Contract N00019-74-C-0409 between
November 1, 1974, and September 30, 1975[1]. This work represents an
attempt to identify as yet unknown eutectics suitable as material for
directionally solidified gas turbine blades.

To date, only a small number of eutectic systems show promise
for use as gas turbine blade materials. Among these sytems are:
Ni-Al-Nb[2,3], Co-TaC[4-6], Ni-TaC[4]. These alloy systems have been
the subjects of extensive development programs, and the possibility
exists that other eutectic systems might have been overlooked. For this
reason, the present program was designed to rapidly screen as many alloy
systems as possible in an attempt to identify those having potential
for use as gas turbine blade materials.

Part I resulted in the identification of five ternary eutectics,
Ni-Cr-Ti, Co-Al-Nb, Co-Si-Zr, Co-Cr-Nb and Co-Cr-Mo, having potential for
use in gas turbine blades. Part II reported here included screening of 24
additional alloy systems, which had been selected during Part I but not
experimentally investigated because of time and funding constraints.

II. SCOPE OF THE PROGRAM

II-1. OVERALL OBJECTIVE

The overall objective of the present program was to identify
eutectics by screening alloy systems limited to those based on Ni, Co
or Fe and containing two reinforcing phases. In these systems:

- The matrix is composed of Ni, Co or Fe and contains in solid solution significant amounts of transition metals, such as Cr, and amounts of non-transitional elements such as Al. The function of Cr or Al would be principally to improve the oxidation characteristics and mechanical properties (by γ'-Ni_3Al strengthening for example) of the matrix;

- The second alloying element would form an intermetallic compound with the matrix as a result of the eutectic reaction. These elements include Ti, Zr, Hf, V, Nb, Ta, Cr...;

- The third constituent is a non-transitional element to form strong covalent compounds with the matrix. Such elements include B, Al, C, Si, N and P. These elements have a relatively low solubility in the matrix phase and would be expected to provide a strongly-bonded compound phase with high strength and stiffness. Some of them (Al, and Si) would be expected to have potential for modifying the scaling characteristics in a beneficial way.

The specific objective is to identify eutectics not previously investigated, having a melting point higher than 1200 C, which should in addition, exhibit good oxidation resistance consistent with their intended use as directionally solidified blades in gas turbines under operating conditions of 1000 hours at 2100 F (1150 C). They should also exhibit a density not in excess of 9 g/cm^3 and a microstructure making them readily amenable to directional solidification.

II.2. RESULTS OF PART I

These objectives were found to be met by five eutectics Ni-Cr-Ti, Co-Al-Nb, Co-Si-Cr, Co-Cr-Nb and Co-Cr-Mo. These were identified and characterized by their melting point, composition and oxidation behavior during Part I. These results were obtained by screening systems based on Ni, Co and Fe. Out of these, 52 were selected for experimental screening, of which 28 were effectively investigated during Part I, leading to the five promising eutectics mentioned.

II.3.   OBJECTIVE OF PART II

The purpose of Part II of this program was to identify eutectics, if any, in the 24 alloy systems which were selected but not experimentally investigated in the previous phase. Among these were alloys such as: Ni-Cr-Ta, Co-Cr-Hf, Fe-Cr-Mo, etc. with expected eutectic compositions so rich in Hf and Ta that their calculated density reached values of 9.5 to 10 g/cm^3, well in excess of the 9 g/cm^3 objective. Also, the Ni-C-V alloy was expected to exhibit very poor oxidation resistance, due to the presence of vanadium. These alloys were therefore not retained for experimental screening. This left the following 13 alloy systems to be investigated: Ni-C-Zr, Ni-Si-Zr, Ni-Si-Nb, Ni-Cr-Zr, Ni-Cr-Mo, Co-Al-Ti, Co-Al-Cr, Co-Si-Nb, Co-Cr-Ti, Co-Cr-Zr, Co-Cr-Ta, Fe-Si-Nb, and Fe-Cr-Si. In addition, attempts were made to prepare Al-rich off-eutectic compositions of two eutectics, Co-Al-Nb and Co-Al-Ta, identified in the previous phase of this program. This was to be done with a view to enhancing their oxidation resistance.

III.   SELECTION OF ALLOY COMPOSITIONS TO BE PREPARED
FOR THE EXPERIMENTAL SCREENING

The initial compositions to be prepared for these 13 alloy systems were selected during the first phase of the program[1]. Briefly, this was done on the basis of a semi-empirical treatment, according to which the region in the ternary phase diagram most likely to contain a ternary eutectic is the triangle delineated by the three straight lines joining the corners of the concentration diagram to the binary eutectic composition on the opposite side[7]. In the present experimental screening, the first alloy composition to be prepared was that represented by the center of gravity of such a triangle in the ternary phase diagram.

IV.   EXPERIMENTAL SCREENING

The process of identifying and characterizing a eutectic included the following steps: (1) preparation of alloy compositions, (2) measurement of the eutectic temperature, (3) measurement of the eutectic composition, (4) determination of the density of the eutectic alloy, and (5) assessment of the oxidation behavior.

For each alloy, the initial composition, derived from the semi-empirical treatment described, was prepared using metals of 99.97% purity or better. Appropriate amounts of charge materials were melted in a water-cooled silver boat arrangement. This technique is best suited to the production of small ingots from high purity materials without contamination from the crucible. The charge materials were melted and kept molten for a few minutes, using induction heating (50 kW at 375 kHz) under a protective argon atmosphere.

Any residual oxygen contained in the argon was trapped by a heated titanium sponge to further protect the sample from oxidation. The resulting ingot (50 to 80 g, about 6 cm long) was cut into pieces that were replaced in the silver boat and remelted to improve alloying. An average of four successive melting operations was found necessary to achieve sufficient alloying of the charge materials.

Samples prepared in this way were metallographically examined for the presence of a eutectic. In the cases where none was observed, additional ingots were prepared to cover a range of composition within a few percent of the initial composition.

Where the initial sample was observed to contain some eutectic between islands of a primary phase, a second alloy composition was prepared in an attempt to increase the proportion of eutectic. The procedure was then repeated until the eutectic represented 20 to 40% of the metallographic section, a proportion sufficient to allow convenient measurement of the eutectic temperature by Differential Thermal Analysis (DTA) and of the composition, by X-ray microprobe analysis.

V. OXIDATION STUDIES

Screening of the oxidation behavior of these alloys was based upon the measurement of their oxidation kinetics, and upon observations of the morphology of the oxide scale formed, as assessed by optical and metallographic methods. Since developed alloys will in practice receive a protective coating, the objective is to attain reasonable, inherent oxidation resistance to prevent catastrophic failure in the event of coating failures. The standards used for comparison are the oxidation rates of alloys of Co-35Cr and Ni-30Cr, both Cr_2O_3-formers, in the same environment.

Specimens of dimensions approximately 1x1x0.1 cm were cut from the end pieces of ingots from the silver boat, using carborundum or diamond-tipped cut-off wheels, and notches were cut in opposite sides to accommodate the platinum suspension wire. All specimens were polished through 200, 400 and 600 grit silicon carbide papers, then on 6 μm and 1 μm diamond wheels and finally on a wheel coated with 0.03 μ alumina. After measuring, degreasing and weighing, the specimens were oxidized in a recording thermobalance at 1150°C in slowly-flowing, dry oxygen at 100 mm Hg pressure. After oxidation for 50 hrs, or until approximately half the metal thickness had been consumed if a rapid oxidation rate was observed, the specimens were cooled to room temperature (tendencies for scale spallation being noted), reweighed, examined in a low-power stereo microscope, then mounted in epoxy for metallographic examination.

VI. RESULTS AND DISCUSSION

In the 13 alloy systems experimentally screened in this study, ten were found to present a eutectic. Out of these eutectics, three (Ni-Si-Zr, Ni-Si-Nb and Co-Al-Ti) did not meet the 1200°C eutectic temperature objective. The remaining seven eutectics had measured melting points above 1200°C. Their compositions were therefore determined by means of an X-ray micro-analyzer. These results are summarized in Table 1.

VI-1. NICKEL BASE ALLOYS

(a) Ni-C-Zr

In this system, a eutectic was observed in the three alloy compositions prepared: 75 Ni-5C-20Zr, 70 Ni-10C-20Zr and 65 Ni-10C-25Zr (wt %). Its structure, shown in Fig. 1a, consists of a Ni-ZrC eutectic with dendrites of Ni and primary phases of C and ZrC. The flakes of primary carbon apparently extend into the ZrC lamellae in the eutectic. Its eutectic temperature was measured to be 1350°C and its composition was analyzed as being: 72 Ni-4C-24Zr (wt %). The specific gravity of the eutectic was determined to be 7.7 g/cm^3, well below the maximum level of 9 g/cm^3.

TABLE 1. SUMMARY OF THE RESULTS OF THE PRESENT WORK

Alloy System	Eutectic Found	Measured Eutectic Temperature (°C)	Measured Eutectic Composition (wt %)			Oxidation Rate (g^2/cm^4.sec) (2)
(1) Ni-base						
Ni-C-Zr	yes	1350	72	4	24	$\sim 10^{-7}$
Ni-Si-Zr	yes	1080	(1)	-	-	1.32×10^{-9}
Ni-Si-Nb	yes	1100	(1)	-	-	1.34×10^{-6} (3)
Ni-Cr-Zr	yes	1255	64.9	19.1	16	1.21×10^{-8}
Ni-Cr-Mo	no	-	-	-	-	-
Ni-30Cr (4)	-	-	-	-	-	2.68×10^{-11}
(2) Co-base						
Co-Al-Ti	yes	1135	(1)	-	-	4.48×10^{-8}
Co-Al-Cr	yes	1310	72.5	6.7	20.8	$<10^{-10}$
Co-Si-Nb	yes	1230	68.3	7.2	24.5	$\sim 2.6 \times 10^{-10}$
Co-Cr-Ti	no	-	-	-	-	-
Co-Cr-Zr	yes	1235	62.8	19.8	17.4	$\sim 2.4 \times 10^{-8}$
Co-Cr-Ta	yes	1320	45.3	11.4	43.3	6.08×10^{-8}
Co-Al-Ta (5)	yes	1290	67.0	0.5	32.5	7.3×10^{-8}
Co-Al-Nb (5)	yes	1240	73.7	3.2	23.1	not representative, see text
Co-35Cr (4)	-	-	-	-	-	9.75×10^{-11}
(3) Fe-base						
Fe-Si-Nb	yes	1360	83.5	12	4.5	-
Fe-Si-Cr	no	-	-	-	-	-

(1) Eutectic temperature not meeting the 1200°C objective, therefore composition not analyzed.

(2) An oxidation rate less than 10^{-7} g^2/cm^4.sec is considered acceptable if no preferential attack of one phase is noted in the sample.

(3) Linear oxidation behavior, units g/cm^2.sec.

(4) Alloys used to set oxidation rate standards.

(5) Eutectics identified in Part I; Al-rich off-eutectics tested in Part II.

Fig. 1a. Ni-5C-20Zr   X500
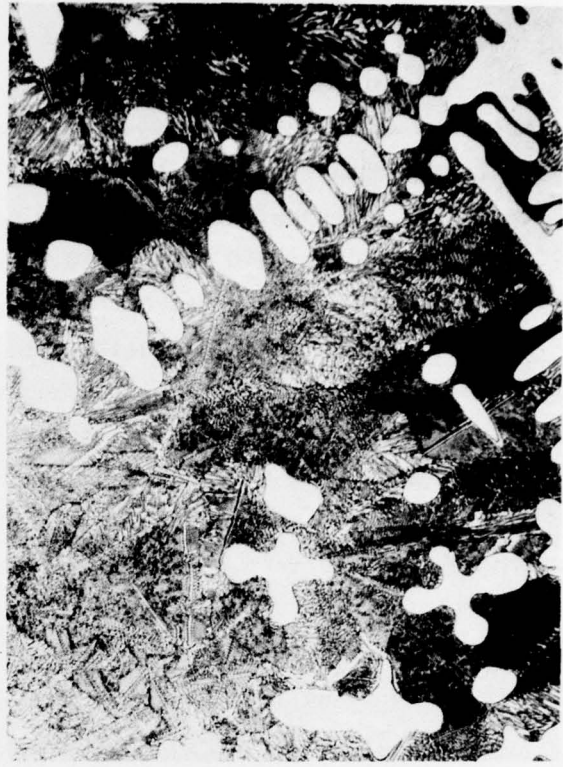Etched in $65H_2O$, $22H_2O_2$,
$13HCl$(vol%)

Fig. 1b. Ni-7Si-8Zr   X500
Etched in $65H_2O$, $22H_2O_2$,
$13HCl$

Fig. 1c. Ni-9Si-17Nb   X500
Etched in $65H_2O$, $22H_2O_2$,
$13HCl$

Fig. 1d. Ni-19.1Cr-16Zr   X500
Etched in $96H_2O_2$, $2HNO_3$,
$2HF$

A sample of the eutectic-containing composition 75 Ni-5C-20Zr oxidized at a rapid, parabolic rate of approximately 10^{-7} g^2/cm^4 sec, which is faster than the desirable oxidation rate of a Ni-30Cr alloy, as shown in Fig. 2. The oxidation behavior of the alloy is interesting in that an adherent scale of NiO was formed, the adherence apparently resulting from the numerous penetrations of the scale into the alloy. Beneath the main scale, a subscale of internally oxidized eutectic phase (the grey-appearing phase in Fig. 1a) was formed. Whenever this subscale approached a ZrC particle, the ZrC was rapidly oxidized with what appeared to be a very small increase in volume (no matrix cracking), and encroachment of the main scale into the alloy then occurred by oxide growth along the oxidized ZrC-matrix interface, and sometimes along adjacent carbon flakes. These general features are shown in Fig. 3a, the depth of internal oxidation being marked by short arrows, and an oxidized ZrC particle entering the main scale being indicated by arrow A.

Since the depth of oxide penetration of this alloy is considerable, 300 μm in one hour, some modification of the oxidation behavior is obviously necessary before the alloy can be given serious consideration. Directions for such modifications are not obvious. It appears necessary to reduce the ingress of oxygen into the matrix, which could be achieved by alloying to form a scale with a low dissociation pressure. However, thermodynamics indicate that ZrO_2 has a dissociation pressure almost the same as Al_2O_3 at 1150°C, so that even alloying to produce an alumina scale might not completely suppress internal oxidation.

(b) Ni-Si-Zr

A eutectic has been identified in this alloy system. Its lamellar structure between Ni-rich dendrites is shown in Fig. 1b. The eutectic composition has been closely approximated by preparing further compositions: 82 Ni-5Si-13Zr and, 85 Ni-7Si-8Zr and 74 Ni-8.5Si-17.5Zr (wt %). The eutectic temperature could thus be measured and was found to be 1080°C, well below the 1200°C objective.

The oxidation kinetics of a specimen of the Ni-7Si-8Zr alloy were within the range of being considered acceptable, as shown in Fig. 2, with a parabolic rate constant of 1.323 x 10^{-9} g^2/cm^4.sec, and the green scale formed was extremely adherent. However, the morphology of the oxidation
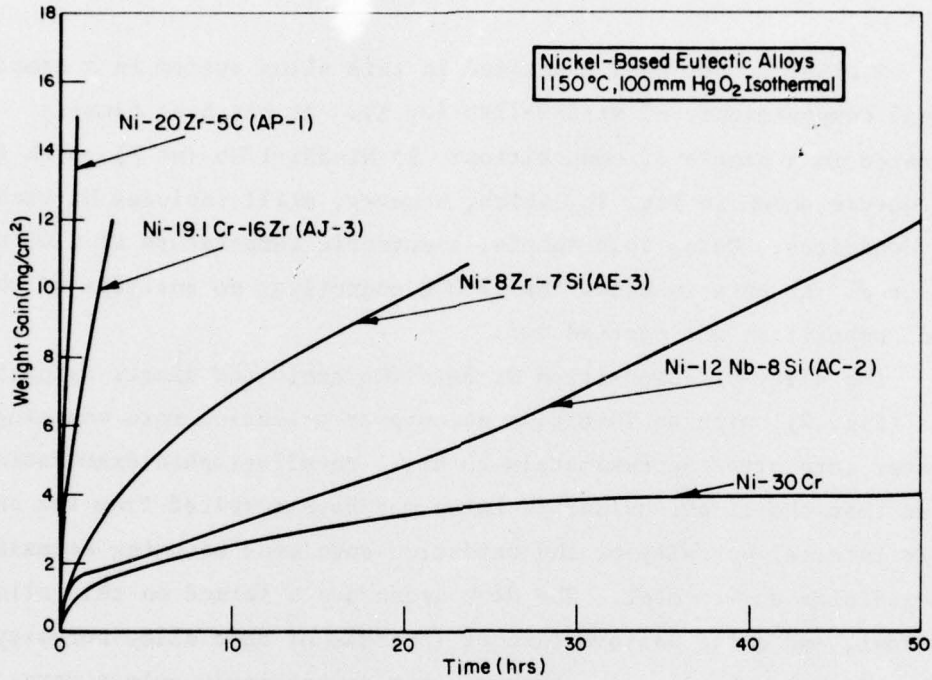
Fig. 2   Isothermal Oxidation Kinetics of Nickel-Based Eutectic Alloys
at 1150°C, 100mm Hg O_2

attack was such that oxide penetrated preferentially along the inter-dendritic eutectic (Fig. 3b) so that the complete thickness of the specimen had been penetrated in 24 hrs. The microstructure of the oxidized specimen appeared considerably different from that shown in Fig. 1b, with a much larger proportion of the alloy present as Ni-rich dendrites. This form of oxidation behavior is clearly unacceptable.

(c) Ni-Si-Nb

A eutectic has been identifed in this alloy system in a sample of nominal composition: 80 Ni-8Si-12Nb (wt %). It has been closely approximated in a sample of composition: 75 Ni-8Si-17Nb (wt %), with the microstructure shown in Fig. 1c, which, however, still includes Ni-rich primary dendrites. Using this sample, a eutectic temperature of 1100°C was measured. As this is below the 1200°C objective, no analysis of the eutectic composition was carried out.

The alloy of composition Ni-8Si-12Nb exhibited linear oxidation kinetics (Fig. 2), with an initially acceptable oxidation rate changing to a faster rate after approximately 20 hrs. Metallographic examination suggested that the linear oxidation rates may have resulted from the con-siderable internal porosity of the oxidation specimens becoming accessible to the oxidizing environment. The dark green scale formed on this alloy was adherent, and quite uniform (except in areas of open alloy porosity Fig. 3c). A thin subscale was observed, but surprisingly only a very limited amount of localized penetration of the eutectic phase occurred.

(d) Ni-Cr-Zr

A eutectic, identified in this system, was found to have a melting point of 1255°C, above the 1200°C objective. This eutectic was therefore further characterized by its composition, found to be: 64.9 Ni-19.1Cr-16Zr (wt %). This result was confirmed by preparing a sample of that composition, in which the resulting structure was indeed fully eutectic, as shown in Fig. 1d. The specific gravity of that eutectic sample was measured to be 8.2 g/cm^3, thus meeting the objective of a density below 9.
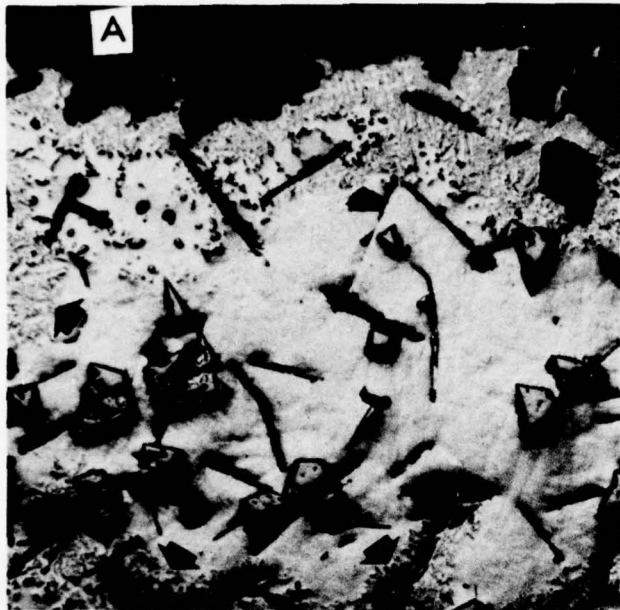
X100

Fig. 3a. Ni-5C-20Zr, Oxidized 1 hr
at 1150°C

X500

Fig. 3b. Ni-7Si-8Zr, Oxidized 24 hrs
at 1150°C

X100

Fig. 3c. Ni-8Si-12Nb, Oxidized 51 hrs
at 1150°C

X500

Fig. 3d. Ni-19.1Cr-16Zr, Oxidized 3 1/4
hrs at 1150°C

The oxidation rate of this eutectic composition was rapid, as indicated in Fig. 2, with a parabolic rate constant of 1.214 x $10^{-8}g^2/cm^4$.sec. Although the scale formed did not spall and was in general of a uniform thickness, extensive internal oxidation of the Zr-rich eutectic phase occurred with, in particular, rapid penetration of long, accicular (apparently Zr-rich) precipitates, to depths of 200 μm after 3 hrs. Fig. 3d shows of such an oxidized, accicular particle at the alloy/oxide interface. The remarks made on the possibilities for modifying the oxidation behavior of the Ni-5C-20Zr alloy also apply to this alloy.

(e) Ni-Cr-Mo

No eutectic was observed in this system in the alloy compositions prepared. These were, in wt %: 65 Ni-20Cr-15Mo, 50 Ni-40Cr-10Mo and 60 Ni-30Cr-10Mo. The two-phase dendritic structure encountered in this range of composition is shown in Fig. 4a.

VI-2. COBALT-BASE ALLOYS

(a) Co-Al-Ti

A eutectic phase was identified in this system in the initial composition prepared: 80 Co-7Al-13Ti (wt %). Its structure is shown in Fig. 4b, between islands of two different primary phases. Two additional compositions, 67 Co-8Al-25Ti and 77.3 Co-1.4Al-21.3Ti (wt %) were prepared to approximate the eutectic. The eutectic temperature was measured, in a sample of composition 80 Co-7Al-13Ti, to be 1135°C, below the 1200°C objective. No further characterization of the eutectic was therefore carried out. Further, the measured melting temperature indicates that, in fact, the eutectic identified in the present study is the Co-TiCo$_2$ binary eutectic, reported as having a melting temperature of 1135°C[8].

A specimen of the Co-1.4Al-21.3Ti alloy oxidized at a rapid, parabolic rate (Fig. 5) with a rate constant of 2.3 to 6.6 x 10^{-8} g^2/cm^4.sec. Although the metal/oxide interface was undulating, no obvious preferential
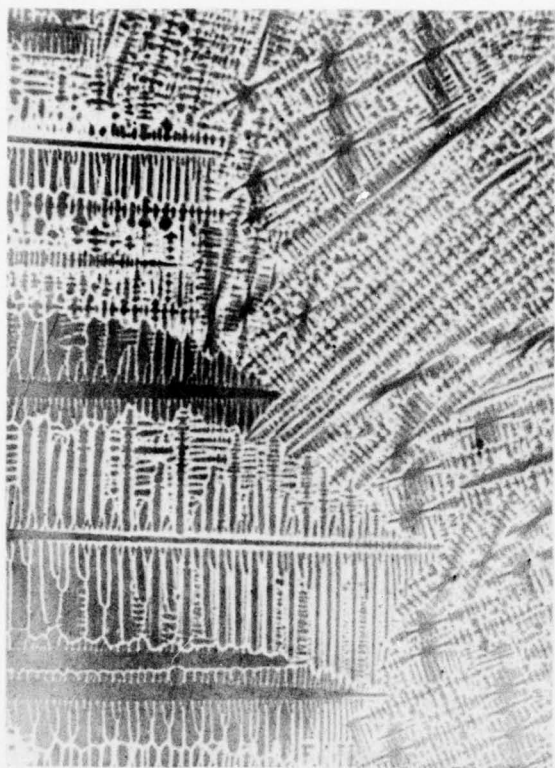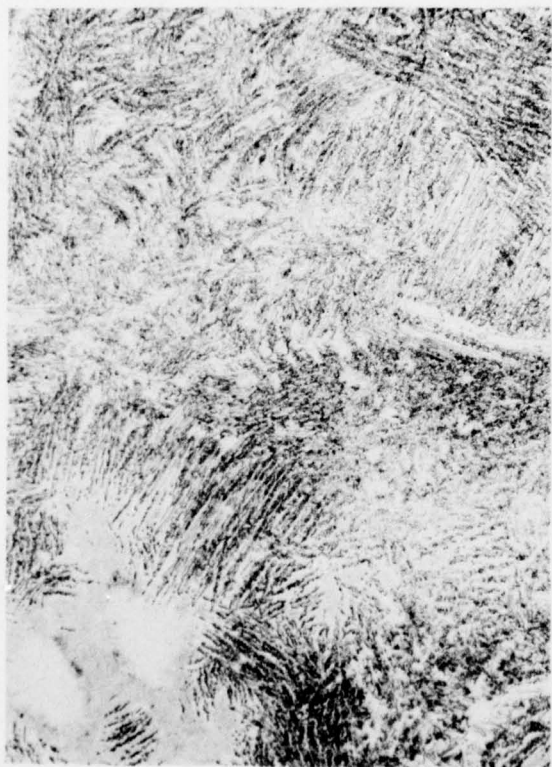
X50

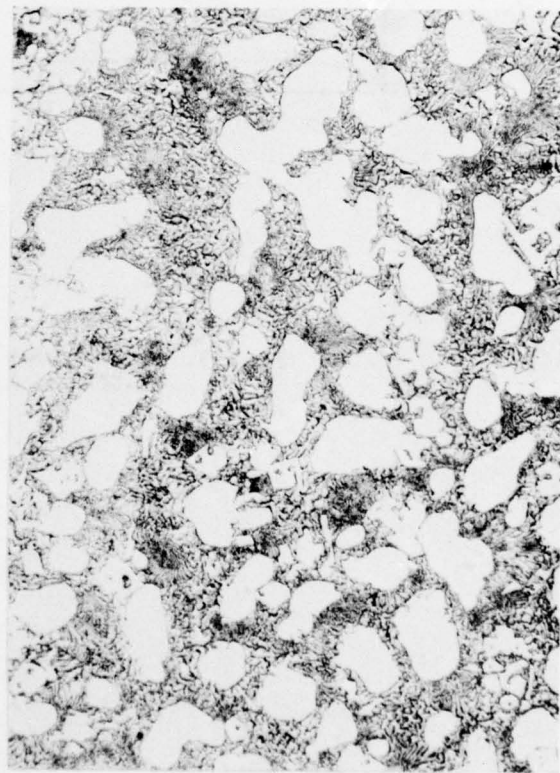Fig. 4a. Ni-30Cr-10Mo, Electro-
lytically Etched in 3%
Oxalic Acid
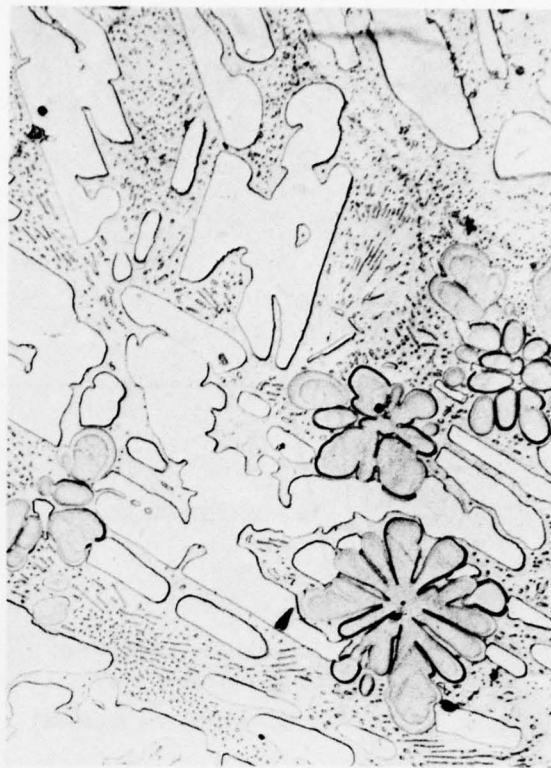
X500

Fig. 4b. Co-1.4Al-21.3Ti, Etched in
$96H_2O_2$, $2HNO_3$, $2HF$

X500

Fig. 4c. Co-10Al-20Cr, Electro-
lytically Etched in 3%
Oxalic Acid

X500

Fig. 4d. Co-7.2Si-24.5Nb, Etched
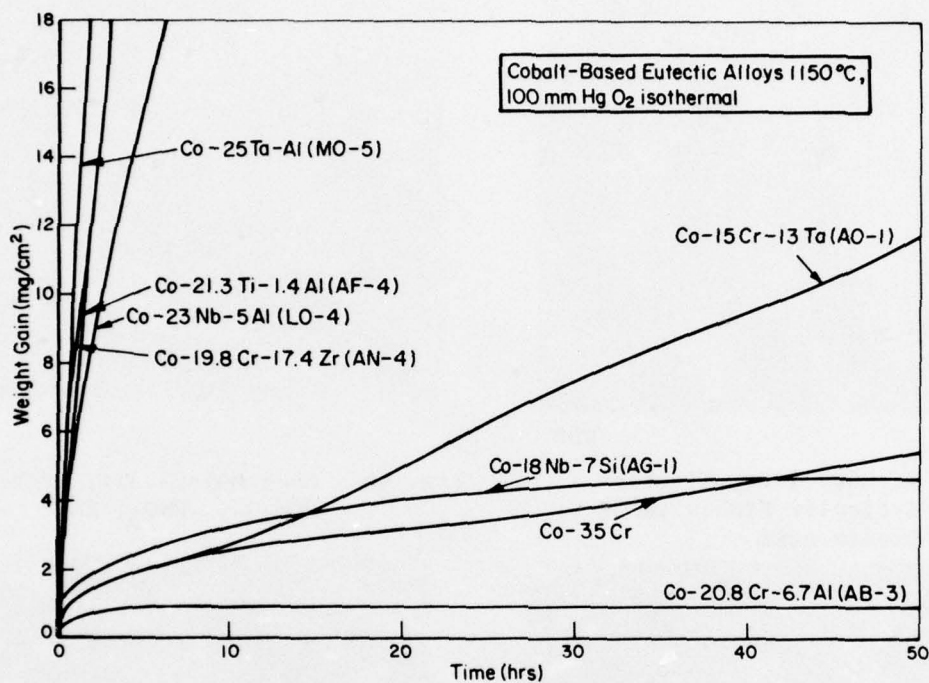in $65H_2O$, $22H_2O_2$, $13HC1$

Fig. 5. Isothermal Oxidation Kinetics of Cobalt-Based
Eutectic Alloys at 1150°C, 100mm Hg O_2

attack was observed, except where areas of porosity were open to the surface (6a). Instead, a uniform, thin layer (~7 μm after 4h) of discrete internal oxide particles (probably Al_2O_3) was formed. The main scale was of heterogeneous composition, reflecting the composition of the matrix, and apparently porous and nonadherent. This alloy is obviously too dilute in aluminum to form any sort of protective scale.

(b) Co-Al-Cr

The eutectic phase identified in this alloy system has been characterized by its melting temperature, 1310°C, and its composition: 72.5 Co-6.7Al-20.8Cr (wt %). The eutectic structure is shown in Fig. 4c in a sample of nominal composition: 70 Co-10Al-20Cr (wt %), therefore very close to the analyzed eutectic composition. This structure is composed of an array of very fine needles, with, however, some small islands of a primary Co-rich phase. The specific gravity of this composition was measured to be 7.6 g/cm^3, thus meeting the objective of a value below 9 g/cm^3.

The oxidation rate of the Co-6.7Al-20.8Cr alloy was, as expected, extremely low (Fig. 5) with an oxidation rate slower than parabolic. The oxidation behavior of alloys in this compositional range has been well characterized[9], and they are known to form protective, Al_2O_3-rich scales. In the cross section shown in Fig. 6b, two layers of scale are evident, as a result of movement of the loosened scale during metallographic mounting. In fact the scale is single-layered, and spalls in sheets on cooling. The means of improving the adhesion of the scale is straightforward, but incompletely understood, by the alloying addition of 0.1 to 0.2% of an oxygen-active element such as yttrium, lanthanum or hafnium.
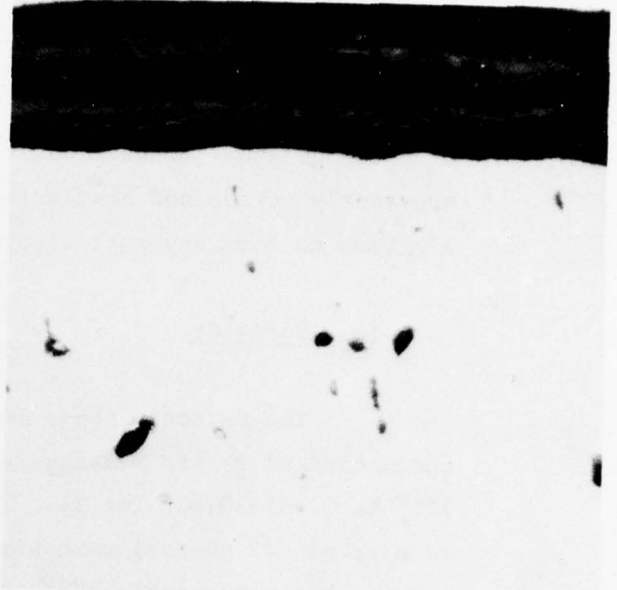
(c) Co-Si-Nb

In this alloy system, a eutectic was identified in the initial composition prepared. The preparation of two further compositions allowed the eutectic to be approximated closely enough so that its melting
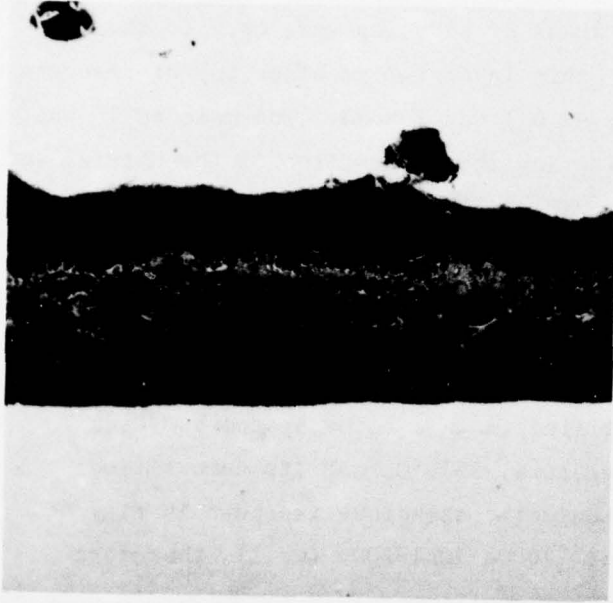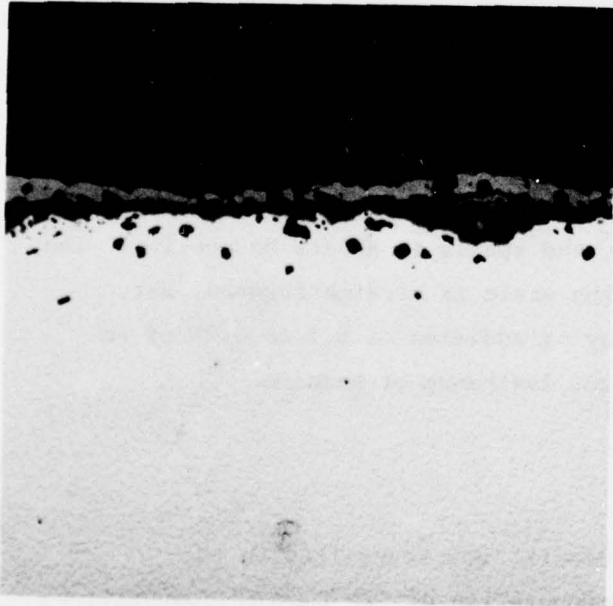
X100

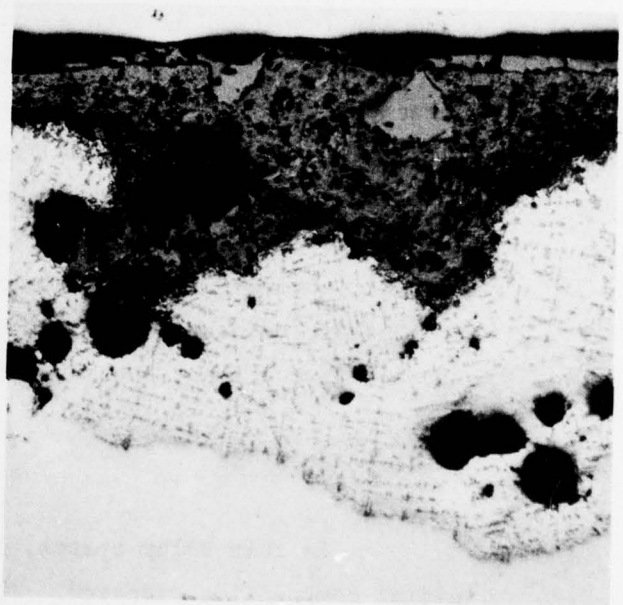Fig. 6a. Co-1.4Al-21.3Ti, Oxidized
4h at 1150°C

X500

Fig. 6b. Co-6.7Al-20.8Cr, Oxidized
68 hrs at 1150°C

X100

Fig. 6c. Co-7Si-18Nb, Oxidized
52 hrs at 1150°C

X100

Fig. 6d. Co-19.8Cr-17.4Zr, Oxidized
2 1/2 hrs at 1150°C

temperature, 1230°C, could be measured. This temperature is much higher than those obtained for the Ni-Si-Nb eutectic (1100°C, see above). Its composition was then determined to be: 68.3 Co-7.2Si-24.5Nb (wt %). In order to confirm this result, a sample of that composition was prepared. As shown in Fig. 4d, this sample was, however, found to present a significant amount of primary phase. This is likely due to a problem in the analysis. The eutectic phase is illustrated in Fig. 7a at the higher magnification made possible by the scanning electron microscope; this shows the very fine lamellae of the intermetallic phase in a Co-rich matrix. The specific gravity of the 68.3 Co-7.2Si-24.5Nb (wt %) composition was measured to be 8.1 g/cm^3, thus meeting the objective of a value below 9 g/cm^3.

A specimen of composition Co-7Si-18Nb exhibited protective oxidation kinetics (Fig. 5), giving a parabolic rate constant of 2.6×10^{-10} g^2/cm^4.sec up to 8 hrs., then oxidizing slower than parabolic. The scale formed was two layered (Fig. 6c), with an outer layer rich in CoO and an inner heterogenous layer apparently containing Nb and Si. Directly beneath the inner oxide layer was a layer of matrix (some 100-150 μm thick after 52 hrs.) almost completely denuded in Si and Nb, then a layer beneath this and of comparable thickness which consisted almost completely of eutectic phase, with few if any Co rich dendrites. Scattered throughout these matrix layers were what appeared to be Kirkendall voids, probably resulting from the diffusion of silicon or niobium into the subscale, and back diffusion of cobalt rejected at the metal/oxide interface. The formation of the eutectic-denuded zone at the metal/oxide interface appears beneficial since it effectively prevents selective oxide penetration along the eutectic phase. The reasons for the layer depleted in primary cobalt dendrites are not clear. However, the oxidation behavior of this alloy is sufficiently protective to warrant further investigation of the alloy system.

(d) Co-Cr-Ti

Three compositions had to be prepared in this system in order to investigate the Co-rich corner of the Co-Cr-Ti ternary phase diagram. No eutectic phase was, however, identified in any of the samples and therefore no further work was done on this alloy. The two-phase structure typical of the composition range explored is shown in Figure 7d.

(e)   <u>Co-Cr-Zr</u>

A eutectic was identified in the initial composition prepared
in this system, which was 81 Co-15Cr-4Zr (wt %). Three additional alloy
compositions were prepared, and a large enough amount of the identified
eutectic was present in a sample of composition: 67 Co-21Cr-12Zr (wt %)
to allow further characterization. Its melting point, 1235°C, similar to
that measured in this study for Ni-Cr-Zr, 1255°C (see above), and its
composition: 62.8 Co-19.8Cr-17.4Zr (wt %) were thus determined.

In order to experimentally confirm the result of the analysis,
a sample having the measured composition was prepared. Although its
microstructure, shown in Fig. 7b presents some primary phase, its compo-
sition is very close to that of the eutectic. A specific gravity of
7.8 g/cm^3 was measured in this sample, thus meeting the objective of a
specific gravity less than 9 g/cm^3.

The oxidation rate of this eutectic was found to be rapid, as
shown in Fig. 5, with a parabolic rate constant of 2.4 x 10^{-8} g^2/cm^4.sec.
The oxide scale formed was not very adherent, and consisted of a thin
outer layer of CoO, with a thick, non-uniform inner layer of CoO con-
taining oxides of chromium and zirconium, and a subscale of oxidized
Zr-rich phase of the eutectic (Fig. 6d). As discussed earlier, for the
other zirconium-containing alloys, the best chance of increasing the
oxidation resistance of this alloy is probably to add sufficient aluminum
to form an external alumina scale. Although the free energy of formation
of ZrO$_2$ is similar to that of Al$_2$O$_3$ at 1150°C, the effective activities
of Al and Zr in these alloys may be sufficiently different to suppress
ZrO$_2$ formation.

(f)   <u>Co-Cr-Ta</u>

For the eutectic identified in this alloy, a melting temper-
ature of 1320°C and a composition of 45.3 Co-11.4Cr-43.3Ta (wt %) have
been measured. Its lamellar eutectic structure is illustrated in
Fig. 7c for a sample of composition: 72Co-15Cr-13Ta(wt%).
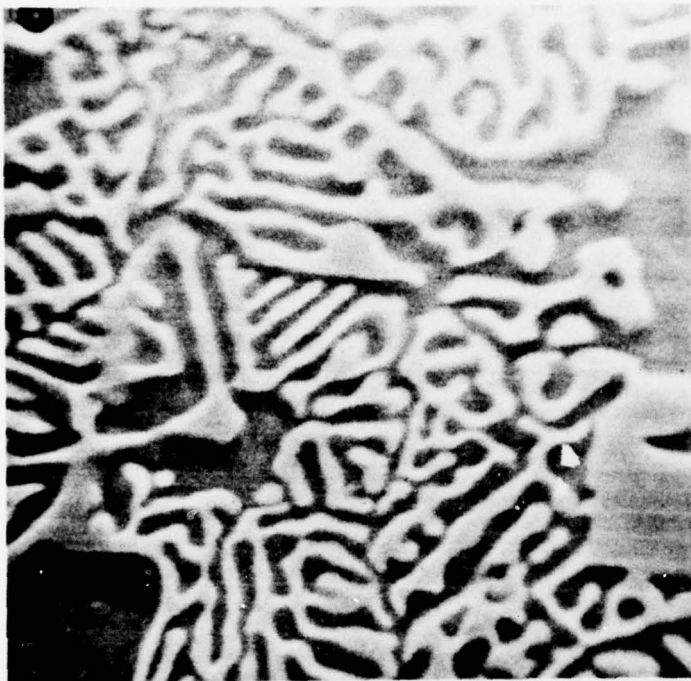
SEM X6000

Fig. 7a.   Co-7.2Si-24.5Nb   Eutectic Structure.
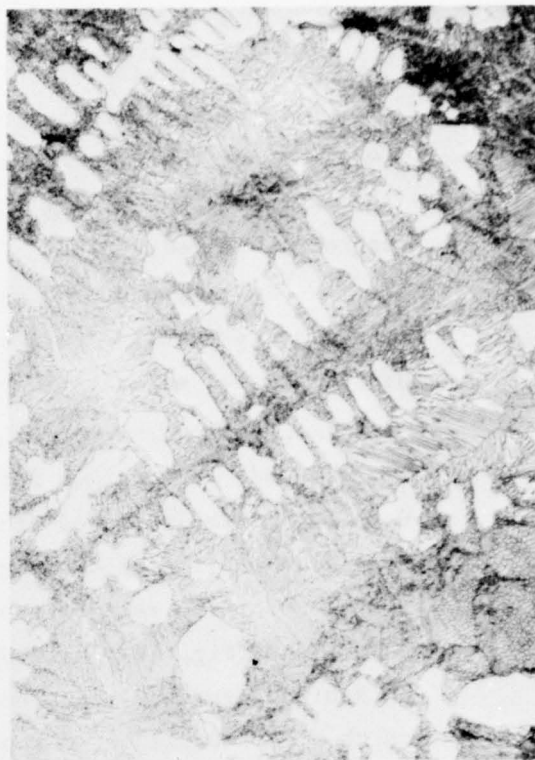
X500

Fig. 7b.   Co-19.8Cr-17.4Zr, Electro-
lytically Etched in 3%
Oxalic Acid
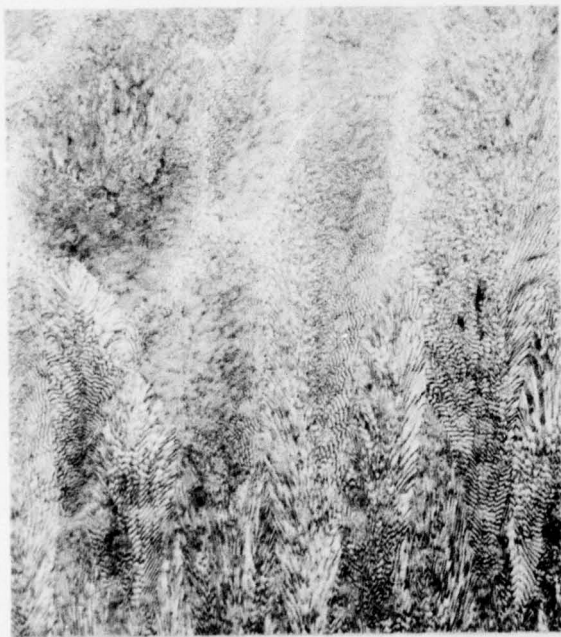
X500

Fig. 7c.   Co-15Cr-13Ta, Electro-
lytically Etched in 3%
Oxalic Acid
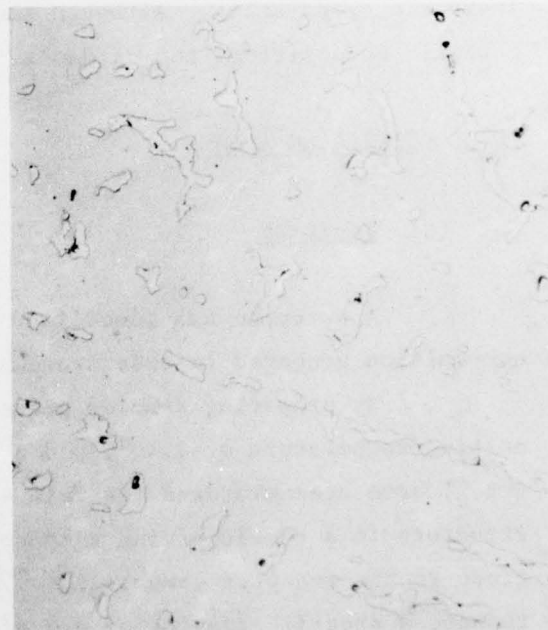
X500

Fig. 7d.   Co-28Cr-4Ti, Etched in $62H_2O-8HNO_3-15H_2SO_4$

The specific gravity of this alloy composition, containing 13wt%Ta, was measured to be 10.5 g/cm^3. Due to its measured higher Ta content (43.4wt%Ta), the eutectic would be expected to have an even higher specific gravity, making it inappropriate for the aeronautical application envisaged.

A specimen of composition Co-15Cr-13Ta was found to exhibit two-stage, protective parabolic oxidation kinetics (Fig. 5). Up to 12 hrs. exposure, a parabolic rate constant of 2.512 x 10^{-10} g^2/cm^4.sec was observed, after which a faster rate of 9.639 x 10^{-10} g^2/cm^4.sec. was established. The reason for this change appears to result from the breakdown of initially-formed protective scale at the corners and edges of the specimen, and the production of more voluminous, Co-rich scales in these areas. Over the rest of the specimen, a thin, protective scale was maintained (Fig. 8a), although this did not appear particularly adherent on cooling from the test temperature. Internal penetration was observed, which followed the network of second phase (Cr-, Ta-rich) particles in this alloy, reaching 80 μm after 52 hrs. The form of this penetration was not easy to discern. In Fig. 8a there appears a network of voids in the location of the second phase particles, but these might equally have been the sites of internal oxide particles mechanically removed during metallographic preparation. Although the sample tested was not the fully eutectic composition, the oxidation behavior appears very promising.

VI-3. IRON-BASE ALLOYS

(a) Fe-Si-Nb

A eutectic was identified in this alloy, in the initial composition prepared in this system: 73 Fe-11.5Si-15.5Nb (wt %).

By preparing samples presenting larger amounts of eutectic, a melting temperature of 1360°C and a composition of 83.5 Fe-12Si-4.5Nb (wt %) have been measured for this eutectic. Fig. 9a illustrates its structure in a sample having a composition 73 Fe-11.5Si-15.5Nb (wt %) close to the measured composition. This alloy composition was measured to have a specific gravity of 8.3 g/cm^3, below the 9 g/cm^3 acceptable maximum.
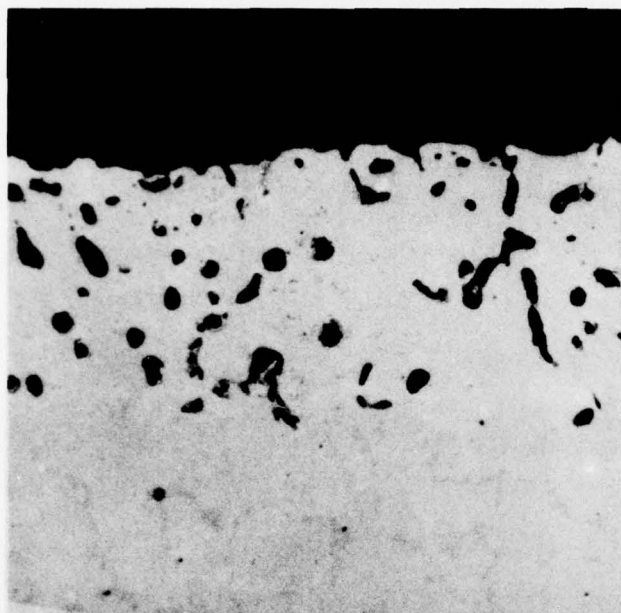
X500

Fig. 8a. Co-15Cr-13Ta, Oxidized
          52 hrs at 1150°C

X30

Fig. 8b. Fe-6.5Si-8.5Nb, Oxidized
          15 mins. at 1150°C

X100

Fig. 8c. Co-5Al-23Nb, Oxidized 3 hrs
          at 1150°C
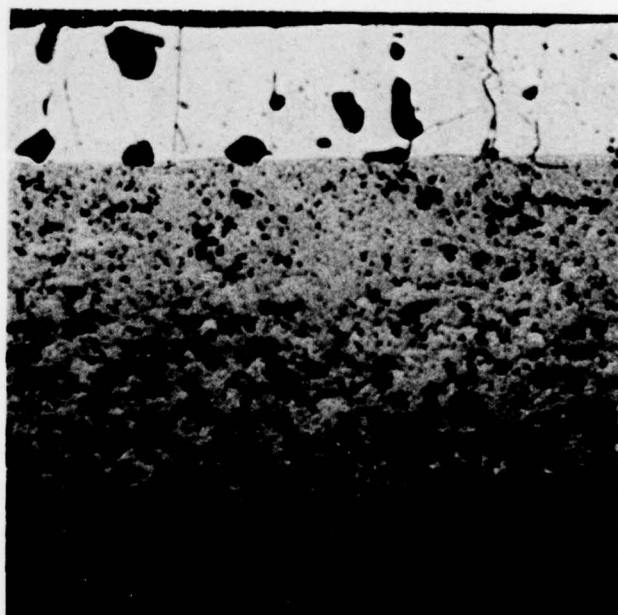
X100

Fig. 8d. Co-7Al-25Ta, Oxidized 3 1/2
          hrs at 1150°C
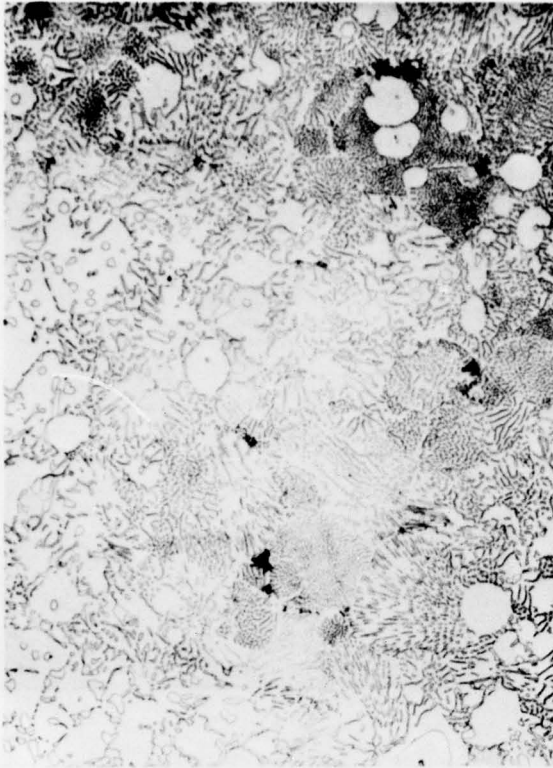
X500

Fig. 9a. Fe-11.5Si-15.5Nb, Etched
in 65H$_2$O, 22H$_2$O$_2$, 13HCl
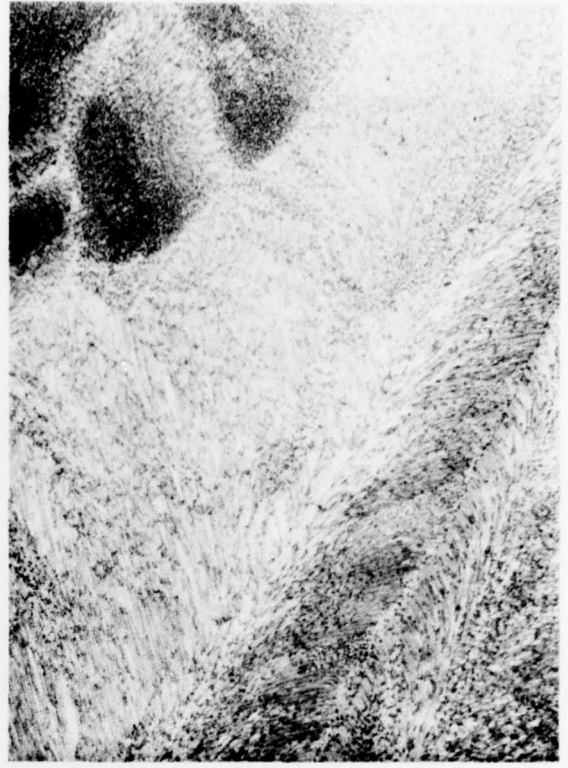
X500

Fig. 9b. Co-6Al-10Nb, Electrolytically
Etched in 3% Oxalic Acid
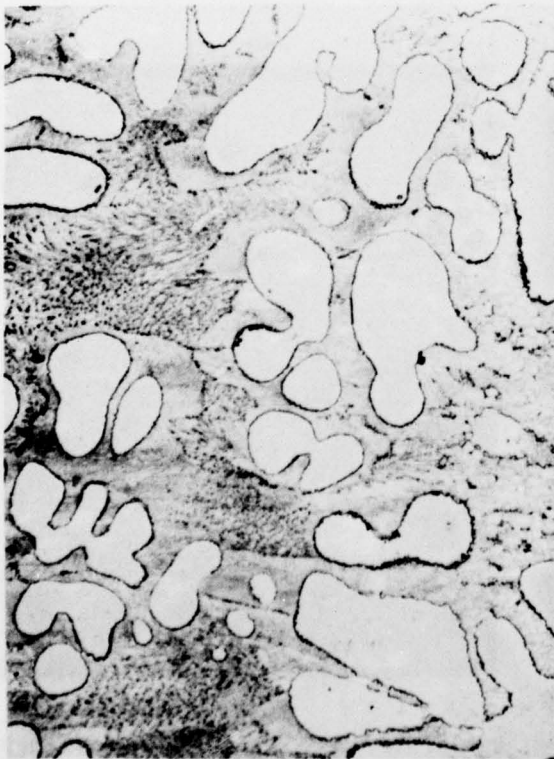
X500

Fig. 9c. Co-6Al-29Ta, Electrolytically
Etched in 3% Oxalic Acid
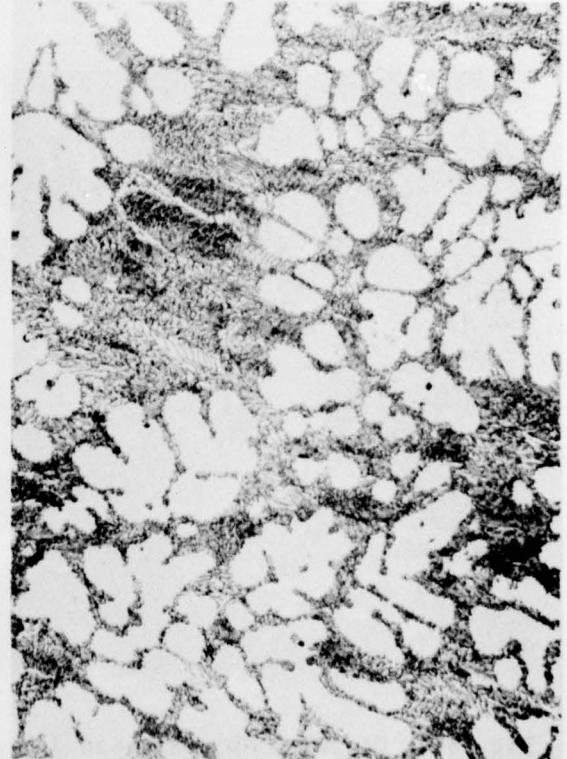
X500

Fig. 9d. Co-7Al-25Ta, Etched in 65H$_2$O,
22H$_2$O$_2$, 13HCl

Specimens of an Fe-6.5Si-8.5Nb alloy oxidized at a catastro-
phically fast rate, gaining 33.2 mg/cm^2 in 15 minutes. The scale formed
appeared to have been molten at temperature, indeed a dendritic structure
could be detected in parts of it (Fig. 8b). Surprisingly, the alloy/oxide
interface was perfectly uniform so that the rate of metal thinning should
be accurately calculable. This composition is obviously unsuitable for
the intended application. However, it is somewhat removed from the
eutectic composition, so that this system should not be excluded from
further evaluation until the oxidation behavior of the eutectic composition
has been determined.

(b) Fe-Si-Cr

No eutectic has been observed in any of the three alloy
compositions prepared to investigate a range of Fe-rich compositions in
this alloy system. The microstructure typical of this range of composition
is illustrated in Figure 10.

VI-4. OFF-EUTECTIC COMPOSITIONS

During Part I of this screening program, two systems, Co-Al-Nb
and Co-Al-Ta, have been found to present ternary eutectics with the low
levels of aluminum of 3.2 and 0.5 wt %, respectively. In order to improve
the oxidation behavior of these systems[1], attempts were made to prepare
them with Al-rich off-eutectic compositions.

(a) Co-Al-Nb

An alloy composition was prepared with a nominal composition
of 72Co-5Al-23Nb (chemical analysis gave 76 Co-6Al-18Nb (wt %)). This
represents a significant increase in the Al content over that of the
73.7 Co-3.2Al-23.1Nb eutectic identified in Part I of this program. This
Al-rich composition was found to exhibit a eutectic structure, as shown
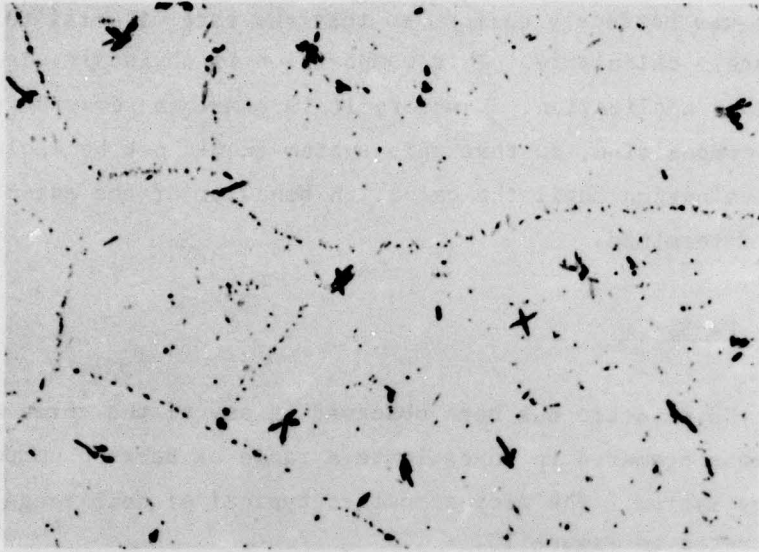in Fig. 9b.

Fig. 10.   Fe-7Si-15Cr(wt%), Electrolytically etched in
3% oxalic acid.   500X

The aluminum-modified composition oxidized to give apparently nonprotective kinetics in Fig. 5, but in fact formed a highly protective scale over most of the specimen surface. Rapid oxidation occurred over small areas of apparently single phase, indicated in Fig. 8c, to produce a very voluminous, yellow oxide, apparently Nb_2O_5. Because of the weight gain resulting from the formation of the Nb_2O_5, the overall oxidation kinetics are obviously distorted and do not reflect the overall good oxidation resistance of the two-phase regions of the alloy.

Neglecting the oxidation behavior of the single-phase areas, this eutectic alloy now meets the oxidation criteria of this program.

(b) Co-Al-Ta

An attempt was made to prepare a pseudo-binary eutectic with a higher Al content than analyzed in the eutectic identified in the present phase: 67 Co-0.5Al-32.5Ta (wt %). For this, a sample of composition 65 Co-6Al-29Ta (wt %) was prepared in the silver-boat. The microstructure of this sample is shown in Fig. 9c. As it includes an important fraction of Co-rich primary phase, another alloy composition was prepared in an attempt to approximate more closely the pseudo-binary eutectic. This second alloy composition, 68 Co-7Al-25Ta (wt %), had a structure shown in Fig. 9d, close to being fully eutectic. The difference in structure between these two samples illustrates the relatively large influence of a small change (a few wt %) in composition.

A sample of composition 68 Co-7Al-25Ta oxidized at a parabolic rate of $7.3 \times 10^{-8} g^2/cm^4$ sec, which is slightly slower than that of Co-10Cr, the upper boundary of acceptable oxidation rate. The scale morphology of a specimen which was completely oxidized in 3 1/2 hrs., shown in Fig. 8d, is essentially similar to that of Co-10Cr, with an outer layer of CoO and inner layer of CoO containing oxides of aluminum and tantalum. The oxidation behavior of this alloy could probably be improved by increasing the level of aluminum until an Al_2O_3-rich scale could be formed; however, the amount of increase is unknown, and could be sufficiently large to move the composition out of any eutectic field.

VII. SUMMARY AND CONCLUSIONS

In this continuation of an alloy screening program, 13 alloy systems, based on Ni, Co or Fe, have been experimentally investigated. These alloys had been selected for screening during Part I of the program but had not been studied for lack of time.

Out of the 13 systems, Ni-Cr-Mo, Co-Cr-Ti and Fe-Si-Cr were found not to present a eutectic in the range of compositions explored. Among the ten identified eutectics, three were found to have potential for use in gas turbine blades. They are:

Alloy System	Measured Eutectic Temperature (°C)	Measured Eutectic Composition (wt %)			Oxidation rate at 1150 C in 100 mm Hg O_2 (g^2/cm^4.s.)
Co-Al-Cr	1310	72.5	6.7	20.8	$< 10^{-11}$
Co-Si-Nb	1230	68.3	7.2	24.5	2.6×10^{-10}
Co-Cr-Zr	1235	62.8	19.8	17.4	2.4×10^{-8}

These properties, particularly the good oxidation behavior already obtained for Co-Al-Cr and Co-Si-Nb, justify further evaluation. It is recommended that this should be done by preparing directionally solidified samples of these eutectics in order to measure the resulting mechanical properties in tensile and creep tests, and the oxidation behavior of the aligned structures.

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(7) J. C. Hubert, M. S. Thesis (University of Montreal, 1968).

(8) Constitution of binary alloys, M. Hansen and K. Anderko (McGraw-Hill, New York, 1958).

(9) See, for instance, G. R. Wallwork and A. Z. Hed, Oxdtn. Metals, $\underline{3}$, 243 (1971).

APPENDIX A

LIST OF ALLOY COMPOSITIONS PREPARED (NOMINAL COMPOSITIONS)

Alloy	Alloy compositions prepared (wt%)			Ternary eutectic found
Nickel Base				
Ni-C-Zr	75	5	20 (1)	yes
	70	10	20 (2)	yes
	65	10	25 (2)	yes
Ni-Si-Zr	82	5	13 (1)	yes
	74	8.5	17.5 (2)	yes
	85	7	8 (2)	close to eutectic
Ni-Si-Nb	80	8	12 (1)	yes
	75	8	17 (2)	close to eutectic
Ni-Cr-Zr	70	20	10 (1)	yes
	85	12	3 (2)	yes
	64.9	19.1	16 (3)	eutectic composition
Ni-Cr-Mo	65	20	15 (1)	no
	50	40	10 (2)	no
	60	30	10 (2)	no
Cobalt-base				
Co-Al-Ti	80	7	13 (1)	yes
	67	8	25 (2)	yes
	77.3	1.4	21.3 (2)	yes
Co-Al-Cr	81	5	14 (1)	yes
	70	10	20 (2)	very close to eutectic
Co-Si-Nb	75	7	18 (1)	yes
	71	9	20 (2)	yes
	68.3	7.2	24.5 (3)	measured eutectic composition
Co-Cr-Ti	55	40	5 (1)	no
	68	28	4 (2)	no
	75	21	4 (2)	no
Co-Cr-Zr	81	15	4 (1)	yes
	60	29	11 (2)	yes
	67	21	12 (2)	yes
	62.8	19.8	17.4 (3)	very close to eutectic
Co-Cr-Ta	72	15	13 (1)	yes
Iron Base				
Fe-Si-Nb	73	11.5	15.5 (1)	yes
	80	10	10 (2)	yes
	85	6.5	8.5 (2)	close to eutectic
Fe-Si-Cr	79	7	14 (1)	no
	85	10	5 (2)	no
	80	15	5 (2)	no

(1) Initial alloy composition derived from the semi-empirical selection method.

(2) Alloy composition prepared to approximate more closely the eutectic composition or, in systems not found to present a eutectic, to check further on the absence of eutectic.

(3) Alloy composition prepared on the basis of the analysis by X-ray microprobe.

Compositions designated by (1) have been prepared during the previous period. Those marked (2) or (3) have been prepared during the present period.

APPENDIX B

SUMMARY OF OXIDATION BEHAVIOR

	Code	Oxidation Rate, $kp(g^2 cm^{-4} s^{-1})$	Remarks
Co-15Cr-13Ta	AO-1	2.512×10^{-10} (0-12 hrs) 9.639×10^{-10} (12-50 hrs)	Thin scale over most of specimen. CoO-rich scale at edges.
Co-20.8Cr-6.7Al	AB-3	slower than parabolic	Dk. blue scale spalled in sheets.
Co-19.8Cr-17.4Zr	AN-4	$\sim 2.4 \times 10^{-8}$ (0-2 1/2 hrs)	Patches of CoO, and of powdery black, nonadherent scale.
Co-18Nb-7Si	AG-1	$\sim 2.6 \times 10^{-10}$ (0-8 hrs), then slower than parabolic	Uniformly adherent, thin dk. grey scale.
Co-23Nb-5Al	LO-4	1.456×10^{-8} (0-6 hrs.)	Mostly protective, Al_2O_3-rich scale. Single, massive outgrowth of Ta_2O_5.
Co-25Ta-7Al	MO-5	7.273×10^{-8} (1-3 1/2 hrs)	CoO-rich, shiny scale.
Co-21.3Ti-1.4Al	AF-4	2.3×10^{-8} (0-2 hrs) 6.63×10^{-8} (2-4 hrs)	Non-adherent, dk. grey with green underside.
Ni-19.1Cr-16Zr	AJ-3	1.214×10^{-8} (0-3 1/2 hrs)	Uniform thick, grey scale, with accicular shapes visible.
Ni-12Nb-8Si	AC-2	1.272×10^{-6} (2-30 hrs) 1.417×10^{-6} (30-50 hrs) linear	Wrinkled green/black scale, no obvious spallation.
Ni-8Zr-7Si	AE-3	1.323×10^{-9} (0-23 hrs)	Adherent, faceted scale, some patches of NiO.
Ni-20Zr-5C	AP-1	9.78×10^{-8} (25-60 min)	Pale green, nodular but adherent scale.
Fe-8.5Nb-6.5Si	AI-3	--	Scale appears to have been molten.

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