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(NASA-TM-X-71765) DIFFCTIONALLY SOLIDIFIED PSEUDO-EINARY EUTECTICS OF Ni-Cr-(Hf, Zr) (NASA) 10 p HC \$3.25 CSCL 11F

N75-29243

Unclas G3/26 31430

DIRECTIONALLY SOLIDIFIED PSEUDO-BINARY EUTECTICS OF Ni-Cr-(Hf. Zr)

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TECHNICAL PAPER to be presented at Conference on In-Situ Composites II sponsored by American Institute of Mining, Metallurgical, and Petroleum Engineers, National Aeronautics and Space Administration, Army Research Office (Durham) and Office of Naval Research and Arranged by General Electric Corporate Research and Development,
Bolton Landing, Lake George, New York, September 2-5, 1975

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ABSTRACT

A pseudo-binary eutectic, in which the intermetallic Ni₇Hf₂ reinforces the Ni-Cr solid solution phase, was previously predicted in the Ni-Cr-Hf system by a computer analysis. This report is concerned with the experimental determination of pseudo binary eutectic compositions and the directional solidification of the Ni-Cr-Hf,Zr, and Ni-Cr-Zr eutectic alloys. To determine unknown eutectics, chemical analyses were made of material bled from near eutectic ingots during incipient melting. Nominal compositions in weight percent of Ni-18.6Cr-24.0Hf, Ni-19.6Cr-12.8Zr-2.8Hf, and Ni-19.2Cr-14.6Zr formed aligned pseudo-binary eutectic structures. The melting points were about 1270° C. The reinforcing intermetallic phases were identified as noncubic (Ni,Cr)₇Hf₂ and (Ni,Cr)₇(Hf,Zr)₂, and face centered cubic (Ni,Cr)₅Zr. The volume fraction of the reinforcing phases were about 0.5.

INTRODUCTION

The search for new materials for high operating temperatures in gas turbine engines has led to the development of directionally solidified eutectic (DSE) alloys, which are also known as In-Situ Composites. The DSE alloys being evaluated for eventual use as turbine materials range from ductile-ductile systems, where both matrix and reinforcement are ductile, to brittle-brittle systems, where both phases are brittle. For achieving high temperature strength exceeding that of conventional superalloys, the reinforcing phases in DSE alloys generally desired are either high temperature intermetallic compounds or refractory compounds. Lamellar intermetallic Ni3Cb reinforced Nibase alloys and monocarbide (TaC) reinforced Nibase alloys have both received intensive attention as advanced gas turbine blade materials.

The first-stage turbine buckets in gas turbine engines are subjected to the most severe combination of stress and temperature in a hostile environment. Binary systems cannot achieve the high strength combined with high temperature oxidation resistance needed for the gas turbine service. Fractical alloys will be multi-component systems.

In complex unknown systems, the general shape of the liquidus surface and the eutectic isotherms can be found by exploratory thermal analysis of selected compositions. 5,6 The eutectic composition can be found by extrapolation of

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thermal analysis data. Zone-melting has also been suggested as an efficient method of finding the eutectic composition once a near-eutectic composition is established. Modern analysis equipment such as electron microprobe or energy dispersive X-ray analysis are able to determine local eutectic compositions in alloys which may extend over a range of concentrations. Computer calculations of phase diagrams have also been developed to provide information on phase stability and transformation.

The feasibility of finding a high temperature eutectic in the Ni-Cr-Hf system has been predicted by a computer analysis. The calculated alloy system is a pseudo-binary eutectic in which the intermetallic Ni₇Hf₂ reinforces the Ni-Cr solid solution phase. The estimated melting point of the pseudo-binary eutectic was about 1315° C, and the predicted composition in weight percent of the eutectic alloy was Ni-30.3Cr-8.9Hf.

The objective of this work was to determine experimentally the composition of the eutectic in the Ni-Cr-Hf system predicted by computer calculation and to characterize its microstructure and solidification behavior. A further objective was to explore Zr analogues of the Ni-Cr-Hf alloy in which Zr was partially or totally substituted for Hf to reduce the alloy density.

During the course of this work, an experimental technique was developed for obtaining material for chemical analysis of the eutectic composition by collecting the low melting alloy bled from an off-eutectic ingot during incipient melting.

EXPERIMENTAL PROCEDURE

Ingot Preparation

The purity, shape, and supplier of the alloying elements used in this work are shown in Table I. All the elements were more than 99.8 percent pure except Hf which contained 2.35 percent Zr. The ingots were made as follows. The desired alloy composition was melted in a zirconia crucible in a 50 kW 10 kHz, vacuum induction furnace. An initial charge of Ni was melted under a partial argon atmosphere, followed by additions of Cr, Hf, and Zr. After holding about 5 minutes at 1650° C for homogenization, the melt was poured at about 1590° C into a copper mold to produce bar ingots for subsequent remelting.

Eutectic Composition Determination

A simple experimental technique was developed for obtaining eutectic material for chemical analysis from initial melts of off-eutectic alloys. A cast bar ingot (1 cm diam, 12 cm length) was put into a closed end tube crucible (i.d., 1.3 cm; o.d., 1.8 cm; length, 30 cm). The crucible with the bar positioned vertically was placed inside a Bridgman furnace, which was constructed for directional solidification (fig. 1). Under a flowing argon atmosphere, the sample was slowly heated for melting in the furnace during about a 2-hour period. As the temperature approached the melting point, alloy of eutectic composition bled from the surface. The bled-out eutectic accumulated in the space between the sample and the tube crucible. The eutectic which had bled out solidified because of a water spray which cooled at the bottom of the crucible. The bled-out eutectic was stripped off the bar, and cleaned in a solution of three parts HCl and one part H₂O₂ prior to chemical analysis.

Directional Solidification

Cast ingots in a tute crucible (i.d., 1.3 cm; o.d., 1.8 cm; length, 30 cm) were remelted in a modified Bridgman furnace as in figure 1, and directionally solidified to produce an aligned structure. In the directional solidification apparatus of figure 1, the heat was supplied to the melt by radiation from the graphite susceptor positioned inside an induction coil. The power was supplied by a 7.5 kW radio frequency generator operating at 400 kHz. Heat was removed

by a water spray or by radiation to a water-cooled ring. The degree of super-heat in the melt in this work was about 250° C. For a given power setting, a flat interface was obtained just below the induction coil by adjusting the thickness of the insulator between the susceptor and water spray ring (fig. 1). Alumina tubes were used as crucibles for the Ni-Cr-Hf alloys. They were cooled with the water spray. Zirconia tubes were used for the Zr-containing alloys. To avoid thermal shock, a water-cooled copper toroid was used instead of the water-spraying ring when using the thermal shock prone zirconia cruci-

Measurement of the temperature gradient at the solid-liquid interface was made by five shielded Pt-PtRd thermocouples with the hot junction located at 2 cm intervals vertically in the melt. The crucible with the melt containing the thermocouples was lowered through a water spray ring at a constant rate of 5 cm/hr. The temperature variations at each point during the freezing process were recorded as a function of time (or distance). Thermal gradients at the melting point of 1270° C were then found from slopes of the temperature-distance curves. The temperature gradients were in the range of 250° to 300° C/cm. With this thermal gradient, the directional solidification was conducted by lowering the crucible out of the induction coil at a constant rate in the range of 1 to 6 cm/hr. A flowing argon atmosphere was used in order to retard evaporation and prevent oxidation.

Metallography

The structure and alinement of the directionally solidified alloys were examined by optical microscopy. Transverse and longitudinal sections were mounted in bakelite, followed by grinding through 600 grade abrasive paper. Intermediate and final polishing were accomplished using 0.6 and 0.3 micron alumina. The polished sample was etched with 33HCl-33HN03-33H20-lHf solution. The volume fraction of the reinforcing phase was determined from a scanning electron micrograph of the lightly etched transverse section by using quantitative scanning television microscopy. 10

Identification of Intermetallic Phases

An extraction of the intermetallic phase in the aligned cutectic was conducted electrolytically by dissolution of the matrix. The aligned eutectic sample was connected to the anode, and platinum mesh was used as the cathode. An electrolyte of 1 percent ammonium sulphate and 1 percent citric acid in water was used in dissolving the Ni-Cr solid solution matrix phase at a current density of about 0.02 amp/cm2.11 After about 10 hours of electrolysis, the matrix had been etched away to a depth of about 3 mm. The specimen was then washed by allowing it to soak in distilled water to remove all electrolyte within the sample. No shape change in the specimen was noticed after the dissolution of the matrix because the intermetallic phase remained on the surface of the specimen. The residue of the intermetallic phases were removed by scalpel, and subjected to chemical analysis by an arc emission spectrographic analysis technique.12 The Debye-Sherrer powder method was used to study the crystal structure of the extracted intermetallic residue. Ni-filtered Cu-, radiation was used operating at 40 kV and 40 ma. The X-ray diffraction patterms were compared with those of ASTM E-413 to identify the crystal structure.

RESULTS AND DISCUSSION

Identification of the Eutectic Composition

The microstructure of an ingot of Ni-30.3Cr-8.9Hf, the composition calculated to be the Ni₇Hf₂-NiCr solid solution eutectic alloy, is shown in figure 2. The light phase is the Ni-Cr solid solution and the eutectic is

rresent interdendritically. It is evident that this composition is considerably off-eutectic.

A simple technique developed in this work was used to determine the eutectic composition experimentally. This technique obtains eutectic material from an off-eutectic alloy by the use of incipient melting which is characteristic of off-eutectic alloys. A sample was slowly heated for melting in the directional solidification apparatus shown in figure 1. As previously mentioned, when the melting point was reached, eutectic bled from the surface of the sample, and accumulated in the space between the sample and crucible. Figure 3(a) shows a longitudinal section of the bled-out sample. The "A" region is the bled-out eutectic material. The "B" region is the as-cast bar which was not remelted, and "C" region is the completely melted region. The middle region "M" is where the eutectic composition was remelted but the proeutectic phase of Ni-Cr solid solution was not remelted. The bled-out eutectic portions were stripped from the sample for chemical analysis. The microstructure of the bled-out material of the "A" region was fully entectic as shown in figure 3(b). The chemical composition of the bled-out eutectic material are in Table II. On the basis of this analysis, a nominal composition of Ni-18.6Cr-24.0Hf was selected to produce the pseudo-binary cutectic. The structure of this alloy when cast into a copper mold was found to be fully eutectic, confirming the eutectic nature of the composition detaimined by the bleed-out technique.

Characterization of the Ni-Cr-Hf Eutectic

Directional solidification of the Ni-18.6Cr-24.0Hf was conducted to produce aligned structures. Figure 4(a) shows the microstructure of transverse section of a directionally solidified sample. The dark phase is the intermetallic, and the light phase is the NiCr solid solution. The volume fraction of the reinforcing intermetallic phase is approximately 0.5. The alignment of the lamellar plates parallel to the growth direction can be seen in the longitudinal section of figure 4(b).

Finer lamellar structures were produced with the increasing growth rate. The interlamellar spacings decreased from about 1 μ at 1 cm/hr to about 0.5 μ at 6 cm/hr. The interlamellar spacing could be an experimental variable to optimize the strength of the aligned cutectic. Increasing solidification rate, which decreases the interlamellar spacing, was reported to improve tensile, 14 creep, 15 and fatigue 16 properties of lamellar cutectics.

Good alignment of the lamellar platelets in the Ni-18.6Cr-24.0Hf eutectic was possible below a growth rate of about 4 cm/hr with a thermal gradient of about 250° C/cm. For the growth rate of 6 cm/hr, a transition from lamellae to rods or perhaps the formation of some primary Ni-Cr solid solution occurred in the eutectic. The lamellar to fiber transition was reported to be caused in other eutectics 17 by either a high growth rate (R) or a low thermal gradient (G), which lower the ratio of G/R. The critical G/R is usually a constant for a given alloy system. 18 It is estimated to be 65° C hr cm-2 for the Ni-Cr-Hf eutectic by assuming G to be about 250° C/cm and R about 4 cm/hr.

The melting point of these eutectic alloys was in the vicinity of 1270° C. The density of this alloy was about 9.1 g/cm³.

Zr-Modification

Substitution of Zr for Hf in the Ni-Cr-Hf system was made in order to reduce density and to determine the effect of the substitution on structure. Partial or total substitution of Zr for Hf was possible while maintaining the pseudo-binary eutectic structure. Because of the much lower atomic weight of Zr, 91.22, compared to 178.50 for Hf, the density of the Zr containing alloys was much reduced even though the atomic percent of Hf plus Zr was held constant at 9 a/o. A nominal composition of Ni-19.6Cr-2.9Hf-11.9Zr resulted in a pseudo-binary eutectic alloy with a density of about 8.4 g/cm³. Figure 5 shows the microstructure of the transverse section of the directionally solidified Ni-19.6Cr-2.9Hf-19.8Zr eutectic alloy. The alloy for which Hf was totally replaced by Zr had a density of 8.2 g/cm³. The microstructure of the direc-

4___

tionally solidified Ni-Cr-Zr eutectic alloy is shown in figure 6.

The thermal gradient experienced in the directional solidification of Ni-Cr-Hf, Zr and Ni-Cr-Zr eutectics was about £0° C/cm. The lower thermal gradient than that used in the Ni-Cr-Hf eutectic, about 250° C/cm, allowed a maximum growth speed of about 1.5 cm/hr for the production of aligned structure. The critical G/R would be 55° C hr cm-2 by assuming G to be about 80° C/hr and R 1.5 cm/hr.

Identification of Reinforcing Phases

The identification of the reinforcing intermetallic phases in the Ni-Cr-Hf, Ni-Cr-Hf,Zr, Ni-Cr-Zr eutectics was conducted by dissolving the matrix Ni-Cr solid solution phase. The compositions of the intermetallic residues are summarized in Table III. The composition of the residue in Ni-Cr-Hf can be expressed by (Ni,Cr)7Hf2, and that of the residue in Ni-Cr-Hf,Zr as (Ni,Cr)7(Hf,Zr)2. In the Ni-Cr-Zr eutectic, however, the composition of the residue may be expressed as (Ni,Cr)5Zr.

The powder X-ray diffraction pattern of the residue in the Ni-Cr-Zr showed a FCC structure of AuBes type, 13 corresponding to that of Ni₅Zr. The X-ray diffraction patterns showed the residues in Ni-Cr-Hf and Ni-Cr-Hf, Zr eutectics to be complex noncubic types.

Comparison With Other Lamellar Eutectics

Table IV lists some of the physical properties and growth parameters of the three alloys studied in this investigation and of two well known lamellar eutectic alloys, $Ni_3Al-Ni_3Cb(\gamma'-\delta)^{19}$ and the 6 percent Cr version of $Ni/Ni_3Al-Ni_3Cb(\gamma/\gamma'-\delta)^3$. The melting points of all the alloys are similar as are their densities except for the Ni-Cr-Hf alloy which is considerably more dense. The Hf and Zr bearing alloys have larger volume fractions of reinforcing phase which might contribute to high temperature strength but probably at the expense of low temperature ductility. Their critical G/R and hence ease of processing lie between those of the $\gamma/\gamma'-\delta$ and $\gamma'-\delta$.

CONCLUDING REMARKS

The eutectic between (Ni,Cr)₇Hf₂ and Ni-Cr solid solution was determined experimentally using a computer-calculated phase diagram of the Ni-Cr-Hf system as a starting point. The microstructure and solidification behavior were determined for this system and for two analogous eutectic alloys in which Zr was partially or completely substituted for Hf. The eutectic alloys investigated all had melting points of about 1270° C which is close to those of γ' - δ and γ/γ' - δ . They were lamellar eutectics with about 0.5 volume fraction of reinforcing phase, which is somewhat greater than that of the Ni₃Cb(δ) reinforced alloys. While the large volume fraction of reinforcing phase should favor strength, it might also be expected to have a deleterious effect on ductility.

The alloys containing high concentrations of Zr had the advantage of lower densities than the alloy without Zr additions. Their densities overlapped the densities of $\gamma'-\delta$ and $\gamma/\gamma'-\delta$. The alloys containing no Hf were reinforced by a cubic intermetallic (Ni,Cr)₅Zr which may enhance high temperature ductility by offering more slip systems than the complex noncubic structure of the intermetallics in the Hf containing alloys.

A simple method was developed for experimentally determining the eutectic composition of new systems. Eutectic material bled from the surface of off-eutectic ingots during incipient melting was collected for chemical analysis. Even for ingots containing more than 60 percent proeutectic phase in Ni-Cr-Hf, it was possible to obtain, in a single step, a composition that could be grown as an in-situ composite. It is evident that the compositions obtained did not represent invariant eutectics because they required a finite G/R to produce plane front growth. Perhaps, the true invariant eutectic compositions could have been found by iterations of the method with bars successively close to the eutectic.

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TABLE I. - RAW MATERIALS

Material	Purity (w/o)	Form	Supplier	
Ni Cr Hf	99.9 99.8 99.8	Electrolytic chips Electrolytic chips Sponge	INCO Union Carbide AMAX	
 Zr	(2.35 percent Zr included) Reactor grade	Sponge	хама	

TABLE II. - RESULTS OF SPECTROGRAPHIC

AMALYSIS OF THE BLED-OUT MATERIALS

IN N1-30.3 Cr-8.9Hf ALLOYS

Sample	Composition, w/o				
number	Ni	Cr	ĦĖ	2r	
HE-2-1 HE-2-2 HE-2-3 HE-2	59.3 60.5 61.8 59.6	15.9 15.6 16.1 16.9	23.7 22.8 21.1 22.4	1.1 1.0 1.0	

TABLE III. - COMPOSITIONS OF THE INTERMETALLIC RESIDUES BY ARC EMISSION

SPECTROGRAPHIC ANALYSIS

Alloy	Composition, wt.% (at.%)				Formula	
	Hf	Zr	Cr	NI	at.Z(Ni+Cr) at.Z(Hf+Zr)	
Ni-Cr-Hf	44.9 (21.2)	0.3 (0.2)	0.6 (1.2)	54.2 (77.5)	(3.6)	(Ni,Or)7HT2
Ni-Cr-Hf,Zr	5.70 (2.16)	26.70 (19.80)	0.90 (1.17)	66.7 (76.87)	(3.5)	(N1,Cr)7(Hr,Zr)2
Ni-Cr-Zr		25.7 (10.9)	1.6 (1.1)	72.7 (49.2)	⁸ (4.6)	(Ni,Cr)5Zr

^aAssumed to be 5 in writing the formula of (Ni,Cr)₅Zr.

TABLE IV. - PHYSICAL PROPERTIES AND SOLIDIFICATION PARAMETERS OF LAMELLAR FUTECTIC ALLOYS

'Properties	Systems					
	NI-Cr-HC	Ni-Cr-Hf,Zr	Ni-Cr-Zr	γ/γ'-6 (ref. 3) Ni/Ni ₃ Al-Ni ₃ Cb-6Cr	γ'-6 (ref. 19) NigAl-NigCb	
Melting point,	1270	1270	1270	1270	1280	
Density, g/cm ³	9.1	8.4	8.2	8.5	8.1	
Vol.% of reinforcement	50	50	50	37	44	
Identity of reinforcement	(Ni,Cr)7Hf2	(Hi,Cr)7(Hf,Zr)2	(N1,Cr) ₅ Zr	Ni ₃ Cb	мі _З сь	
Crystal structure of reinforcement	Unknown-complex noncubic	Unknown-complex noncubic	FCC	Orthorhombid	Orthorhombie	
Interlamellar spacing	lµ at R = 1 (cm/hr)	1.2 µ at R = 1.5 (cm/hr)	2µ at R = 1 (cm/hr)	3u at R = 3 (m/hr)	3u at R = 2 (cm/hr)	
aG/R, oc hr cm ⁻²	65	55	55	150	<35	

⁴⁶G/R is a critical ratio of thermal gradient (G) over rate of growth (R) for the condition of coupled criented growth.

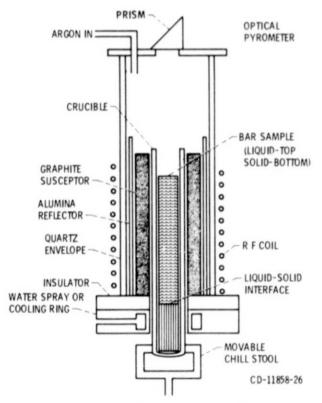


Figure 1. - Directional solidification apparatus.

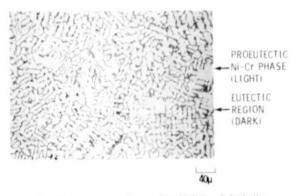


Figure 2. - Microstructure of as-cast Ni-30.3 Cr - 8.9 Hf alloy.



A: BLED-OUT
B: AS-CAST
C: REMELTED AND
SOLIDIFIED
M: EUTECTIC REGION
MELTED



Figure 3. - Metallographical study of Ni-30, 3 Cr-8, 9 Hf alloy.



(b) MICROSTRUCTURE OF THE BLED-OUT REGION ("A") SHOWING FULL EUTECTIC STRUCTURE.
LIGHT PHASE NI-Cr SOLID SOLUTION AND DARK PHASE INTERMETALLIC.

Figure 3, - Concluded.



(a) TRANSVERSE.





(b) LONGITUDINAL.

Figure 4. - Concluded.



TRANSVERSE.

Figure 5. - Microstructure of DS eutectic of Ni-19.6 Cr-2.9 Cr-2.9 Hf - 19.8 Zr, R = 1.3 cm/hr.



Figure 6. - Microstructure of directionally solidified Ni-19.2 Cr-14.8 Zr eutectic alloy, R • 1.0 cm/hr.