UNCLASSIFIED

~~SECRET~~

Report No. BMI-1277

C-84 Reactors - Special Features
of Aircraft Reactors
(M-3679, 21st Ed.)

This document consists of 32 pages,
No. 118 of 150 copies, Series A.

Contract No. W-7405-eng-92

PROPERTIES OF ARC-MELTED CHROMIUM-BASE ALLOYS

by

Daniel J. Maykuth
John W. Roberts, Jr.
Robert I. Jaffee

~~Classification cancelled (or changed to UNCLASSIFIED)~~
~~Letter from Declassification Council,~~
~~By authority of dated April 26, 1962~~
~~J. Zulli~~ TIE, date May 22, 1962

July 14, 1958

~~RESTRICTED DATA~~

This document contains restricted data as defined in the Atomic
Energy Act of 1954. Its transmittal or disclosure of its contents
in any manner to an unauthorized person is prohibited.

BATTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus 1, Ohio

~~SECRET~~

UNCLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

SECRET

Report No. BMI-1277

C-84 Reactors - Special Features
of Aircraft Reactors
(M-3679, 21st Ed.)

3 and 4

Copy No.

Distribution

1-3	Air Force Ballistic Missile Division
4-5	AFPR, Boeing, Seattle
6	AFPR, Boeing, Wichita
7	AFPR, Curtiss-Wright, Clifton
8	AFPR, Douglas, Long Beach
9-11	AFPR, Douglas, Santa Monica
12	AFPR, Lockheed, Burbank
13-14	AFPR, Lockheed, Marietta
15	AFPR, North American, Canoga Park
16	AFPR, North American, Downey
17-18	Air Force Special Weapons Center
19	Air Materiel Command
20	Air Research and Development Command (RDIAEF)
21	Air Research and Development Command (RDZPSP)
22	Air Technical Intelligence Center
23-25	ANP Project Office, Convair, Fort Worth
26	Albuquerque Operations Office
27	Argonne National Laboratory
28	Armed Forces Special Weapons Project, Sandia
29	Armed Forces Special Weapons Project, Washington
30	Army Ballistic Missile Agency
31	Assistant Secretary of the Air Force, R & D
32-37	Atomic Energy Commission, Washington
38	Atomics International
39-40	Bettis Plant (WAPD)
41	Bureau of Aeronautics
42	Bureau of Aeronautics General Representative
43	BAR, Aerojet-General, Azusa
44	BAR, Convair, San Diego
45	BAR, Grumman Aircraft, Bethpage
46	BAR, Martin, Baltimore
47	Bureau of Yards and Docks
48-49	Chicago Operations Office
50	Chicago Patent Group
51	Curtiss-Wright Corporation
52	Engineer Research and Development Laboratories
53-56	General Electric Company (ANPD)
57	General Nuclear Engineering Corporation
58	Hartford Area Office
59	Knolls Atomic Power Laboratory
60	Lockland Area Office
61	Los Alamos Scientific Laboratory
62	Marquardt Aircraft Company
63	Martin Company
64	National Advisory Committee for Aeronautics, Cleveland
65	National Advisory Committee for Aeronautics, Washington
66	National Bureau of Standards
67	Naval Air Development Center
68	Naval Air Material Center
69	Naval Air Turbine Test Station
70	Naval Research Laboratory
71	New York Operations Office
72	Nuclear Metals, Inc.
73	Office of Naval Research
74	Office of the Chief of Naval Operations (OP-361)
75	Patent Branch, Washington
76	Phillips Petroleum Company
77-80	Pratt and Whitney Aircraft Division
81	Redstone Arsenal
82	San Francisco Operations Office
83	Sandia Corporation
84	School of Aviation Medicine
85	Sylvania-Corning Nuclear Corporation
86	Technical Research Group
87-94	Union Carbide Nuclear Company (ORNL)
95	USAF Headquarters
96	USAF Project RAND
97	U. S. Naval Radiological Defense Laboratory
98-99	University of California Radiation Laboratory, Livermore
100-117	Wright Air Development Center
118-142	Technical Information Service Extension, Oak Ridge
143-150	Battelle Memorial Institute

SECRET

~~SECRET~~

5 and 6

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	7
INTRODUCTION	7
EXPERIMENTAL PROCEDURES	8
Materials	8
Alloy Preparation	10
Fabrication	11
Evaluation	11
EXPERIMENTAL RESULTS	13
Melting	13
Hardness, Structure, and Oxidation Resistance	15
Fabricability	22
Hot Strength	29
DISCUSSION AND CONCLUSIONS	29
REFERENCES	31

~~SECRET~~

~~SECRET~~

7

PROPERTIES OF ARC-MELTED CHROMIUM-BASE ALLOYS

Daniel J. Maykuth, John W. Roberts, Jr., and Robert I. Jaffee

Twelve binary chromium-base alloy systems were screened for hot workability, oxidation resistance, and hot strength using small arc-melted ingots. The alloying elements investigated included iron, manganese, molybdenum, niobium, rhenium, ruthenium, tantalum, titanium, tungsten, vanadium, zirconium, and yttrium in amounts up to 20 w/o.

Of the fabricable alloys tested, the best combinations of oxidation resistance and strength, in the range of 2000 to 2300 F, were found in binary alloys containing 5 w/o molybdenum, 10 w/o tungsten, and 2 w/o niobium. While yttrium additions do not contribute to the hot strength of chromium, the effectiveness of yttrium in improving the oxidation resistance of chromium is outstanding. About 1 w/o yttrium appears optimum for this purpose.

Experiments with the preparation of large chromium-base alloy ingots showed that cold-mold arc melting was not suitable for preparing ingots containing over about 1 w/o of alloy addition. However, the feasibility of preparing sound, higher alloy-content ingots by induction melting and casting was demonstrated.

INTRODUCTION

This program was concerned solely with the preparation and evaluation of arc-melted chromium-base alloys. It was undertaken to supplement similar work on chromium-base alloys, being made using powder-metallurgical techniques, by the General Electric Aircraft Nuclear Products Division and its other subcontractors. The ultimate objective of the combined work is the development of an oxidation-resistant chromium-base alloy capable of service at temperatures of 2000 F or greater.

The present work was initiated with the preparation of small alloy ingots in 12 binary chromium-base alloy systems. The alloy-addition elements investigated included iron, manganese, molybdenum, niobium, rhenium, ruthenium, tantalum, titanium, tungsten, vanadium, zirconium, and yttrium in amounts up to 20 w/o. Each of these ingots was screened for fabricability, oxidation resistance, and hot strength.

Subsequently, experiments in the melting, casting, and fabrication of larger alloy ingots were performed to explore possible methods for semicommercially manufacturing arc-melted chromium-base alloys.

Because elevated-temperature properties were of primary interest, none of the alloys were evaluated for mechanical properties at temperatures below 2000 F.

~~SECRET~~

~~SECRET~~

8

EXPERIMENTAL PROCEDURES

Materials

This program was initiated using powdered chromium, prepared by the Lunex Corporation, as the base material. However, during the evaluation of the first series of alloys it was found that the fabricability of an unalloyed sample of the Lunex chromium was definitely inferior to that of an unalloyed iodide chromium control sample. When subsequent analysis showed the Lunex chromium contained 0.13 w/o carbon, the base material for this investigation was shifted to iodide chromium to avoid the detrimental effects of carbon. Analyses and representative microphotographs of both of the types of chromium used are given in Table 1 and Figure 1, respectively. The lack of oxide particles in the structure of the Lunex chromium is apparently due to the reduction of the oxygen content by carbon during melting.

TABLE 1. ANALYSIS OF CHROMIUM-BASE METALS USED

Impurity Element	Impurity Content, ppm	
	Lunex Chromium Powder	Iodide Chromium Crystal Bar
Oxygen	395	10
Nitrogen	50	10
Carbon	1300	30
Sulfur	Nil	80
Aluminum	<5	<2
Calcium	<5	<2
Copper	50	2
Iron	20	<2
Magnesium	<5	<2
Manganese	10	<2
Molybdenum	100	1
Nickel	10	<1
Silicon	10	<20
Titanium	20	<2

The 12 alloying addition metals used were of the highest purity available. As indicated in Table 2, most of the addition metals were of at least 99.9 per cent total purity.

~~SECRET~~

SECRET

9

250X

N43477

- a. Unalloyed Lunex Chromium, Showing Included Carbides

500X

N44716

- b. Unalloyed Iodide Chromium, Showing Scattered Oxide Inclusions at Grain Boundaries

FIGURE 1. REPRESENTATIVE MICROSTRUCTURES IN THE TWO GRADES OF CHROMIUM USED IN THE PRESENT INVESTIGATION

Both samples water quenched after 24 hr at 2000 F.

Etchant: 10 per cent oxalic acid.

SECRET

~~SECRET~~

10

TABLE 2. SOURCE AND QUALITY OF ALLOY-ADDITION METALS

Addition Metal	Form ^(a)	Source or Method of Preparation	Total Purity, w/o
Iron	P	Plastic Metals Division; National Radiator Company	99.9
Manganese	P	Electrolytic process	99.9
Molybdenum	P	Fansteel Metallurgical Corporation	99.9
Niobium	P	Ditto	99.9
Rhenium	P	Chase Brass and Copper Company, Inc.	99.9+
Ruthenium	P	The American Platinum Works	99+
Tantalum	P	Fansteel Metallurgical Corporation	99.9
Titanium	S	Sponge process	99.8
Tungsten	P	Fansteel Metallurgical Corporation	99.9
Vanadium	C	A. D. Mackay, Inc.	99.7
Zirconium	X	Iodide process	99.9+
Yttrium	C	GE-ANPD	--

(a) P = powder; S = sponge; C = chips; X = crystal bar.

Alloy Preparation

All but two* of the alloys investigated in this program were made by arc melting under an inert atmosphere. The alloy charges consisted of pressed compacts made from the elemental metals. To facilitate compacting, the iodide chromium was crushed to a minus 16-mesh size.

Basically two melting techniques were used, the choice of these depending on ingot size. For screening purposes, a 40-g, 3/8-in.-round and 2-1/2 to 3-in.-long "finger" ingot was used. For follow-up purposes, 2 to 6-lb ingots ranging from 2 to 2-1/2 in. in diameter and 3 to 6 in. in length were prepared.

Charges for the 40-g ingots were pressed under 75×10^3 psi in a 1 by 1-1/4-in. die and melted directly, under helium at an absolute pressure of 8 in. of mercury, using a water-cooled tungsten electrode and a shaped copper crucible. The arc furnace was mounted on a hinged joint which permitted a gentle side-to-side rocking motion of the molten charge to facilitate alloying. Each ingot was given a minimum of four melts and was inverted between melts to insure chemical homogeneity.

Charges for the larger ingots were pressed as 1 by 1 by 8-in. compacts under 75×10^3 psi. These were joined end-to-end by fusion arc melting under an inert atmosphere to form a consumable electrode. Some difficulty was experienced with cracking of the welds in the alloy bar electrodes during cooling. These difficulties were minimized by using unalloyed chromium as filler metal at the joints.

*Two ingots were prepared by induction-melting techniques. This work is described in a later section of this report.

~~SECRET~~

~~SECRET~~

11

Melting of the large ingots was accomplished using an automatically controlled power input of 30 to 32 v and 1500 to 2000 amp under helium at an absolute pressure of 15 in. of mercury. A magnetic mixing coil was employed around the water-cooled copper crucible to facilitate alloying action. Simple cylindrical-shaped crucibles were used.

Fabrication

Because of anticipated difficulties in hot working, metallic sheaths were used to provide lateral constraint for the arc-melted chromium-base screening alloys. In the follow-up work with larger ingots, various methods of hot working the unsheathed ingots were explored. The techniques used and results obtained are described later.

To provide a good fit inside the sheaths, each of the as-cast screening-alloy ingots was ground to a 1/4-in.-round cylinder. For the first alloy series (including all of the Lunex chromium alloys and an unalloyed iodide chromium control), 3/8-in.-OD capsules of mild or stainless steel were used. The sheathed ingots were hot swaged to a finished diameter of 0.25 in. for an over-all reduction of 56 per cent in hot working. The quality of the swaged alloys was checked first by radiography of the sheathed samples and again after removal of the sheath material. The mild steel sheathing was removed by dissolution in nitric acid while the stainless steel was removed electrolytically.

This work showed that higher working temperatures and additional constraint of the alloys were desirable in hot working. Consequently, the sheaths for the iodide chromium-base alloys were made from 1/2-in.-diameter stock of nickel or molybdenum. Nickel was used for those ingots having cast hardnesses of 200 VHN or less while molybdenum was used for each of the harder ingots. The 1/2-in.-diameter sheathed alloys were also swaged or rod-rolled to a finished diameter of 0.25 in. for an over-all reduction of 75 per cent in hot working. After radiographic inspection, the nickel sheathing was removed by dissolution in nitric acid and the molybdenum sheathing was removed by air oxidation at 2200 F.

All of the sheathed ingots were preheated in a hydrogen atmosphere for hot working.

Evaluation

Each of the screening alloys was investigated for hardness, structure, fabricability (i.e., the ability to withstand hot plastic reduction without failure), oxidation resistance, and hot strength.

The hardness and structure of each alloy were determined on as-cast samples of each ingot and also on cast samples which had been annealed 24 hr at 2000 F in hydrogen and water quenched.

~~SECRET~~

Oxidation Resistance

Oxidation resistance was determined using cylindrical samples machined from the 1/4-in.-round as-cast screening-alloy ingots. Weight gains were determined after heating each sample for 100 hr in a static air atmosphere, in an electric furnace, at 2000 F. These data provided a rough measure of the rate of reaction of individual alloys with air at 2000 F.

Following this, weight losses were determined after wire brushing each sample. These data afforded a means of comparing the adherence of the surface oxidation products.

Hot Strength

Generally, the as-fabricated screening alloys were in the form of rods, 4 to 6 in. long, with slightly ovate diameters of about 0.15 in. Most of the hot-workable alloys were too brittle at room temperature to withstand machining into complex test pieces. Consequently, hot-bend tests were selected as the most expedient means of comparing the alloys' hot strengths.

The individual samples were carefully ground to smooth cylinders about 5/8 in. in length. These were placed across a 0.5-in.-wide V-groove on a vertical molybdenum slab which was supported by four horizontal molybdenum rods that extended to a steel base located outside of the furnace. Loads were applied to the mid-span of the samples by another molybdenum rod which was tapered, at the point of sample contact, to a 1/32-in. radius. Actual loading was effected by applying weights to a pan which was mechanically linked to the load bar by a wire and pulley arrangement. The bend deflection at the mid-point of the sample was measured directly by a spring-actuated dial gage in contact with the molybdenum loading rod.

In practice, a bend sample was positioned in the bend die at room temperature. Then, the assembly consisting of the die, support rods, steel base, and loading pan was moved forward until the die reached a predetermined position in the hot zone of a molybdenum-wound hydrogen-atmosphere furnace. Temperatures were measured optically by sighting directly on the bend-test sample. Load-versus-deflection data were obtained by applying individual 10 to 14-oz weights to the pan at 30-sec intervals with the corresponding deflections being observed at 15 sec after each increment of loading. In an individual run, loading was continued until the elastic range of the sample had obviously been exceeded.

In most cases, smooth load-deflection curves were obtained from which it was not possible to select a definite value for the individual alloy's elastic limit. Therefore, it was more or less arbitrarily decided to compare the alloy strengths using the calculated fiber stress required to produce an elongation of 0.5 per cent in the outer fiber of the bend sample. The fiber stress, S , was calculated using the relationship $S = \frac{Pl}{r^3}$, where P is the concentrated load, l is the distance between supports (0.5 in.), and r is the sample radius.

~~SECRET~~

13

It was recognized that the stress to produce a bend elongation of 0.5 per cent was slightly beyond the elastic limit of the alloys. Hence, the absolute values of the fiber stresses calculated using the above relationship are in some error and are not meaningful as design stresses. However, this approach did furnish an effective means of comparing the hot strengths of the alloys using the limited amount of material available. Also, it was later observed that the bend-stress values obtained were of the same order of magnitude as the values of ultimate strength obtained for chromium and chromium-base alloys in tensile tests performed under similar conditions by other investigators.

EXPERIMENTAL RESULTS

Melting

Screening Alloys

No difficulties or anything unusual occurred during the melting of the 40-g screening-alloy ingots. Spot-check analyses were conducted on four alloys. As shown by the results below, these data indicate that the actual alloy compositions are fairly close to their intended values:

<u>Ingot</u>	<u>Alloy Content (Balance Chromium), w/o</u>	
	<u>Intended</u>	<u>Actual</u>
C 120	2 Nb	1.62 Nb
C 121	5 Ta	4.91 Ta
C 122	5 Mo	4.38 Mo
C 123	5 W	5.21 W

Large Alloy Ingots

Fourteen ingots were prepared by consumable-electrode arc-melting techniques during the course of this investigation. Five of these were made using Lunex chromium as a base, the balance with iodide chromium. Two of the iodide chromium ingots were later remelted using induction-melting techniques.

The compositions, weights, hardnesses, and type of melting procedure used for each of the ingots are listed in Table 3 in the chronological order of their preparation. Pertinent observations on the behavior of the individual heats are summarized as follows:

Lunex Chromium-Base Alloys. The melting stock for four of the five ingots melted (i.e., all excepting Ingot C 96 in Table 3) was prepared at GE-ANPD. Prior to their assembly as consumable electrodes, the compacts for these four ingots were given a lithium-sintering treatment at GE-ANPD. The alloy compacts for Ingot C 96, on the

~~SECRET~~

other hand, were sintered in dry hydrogen for 4 hr at 2450 F. All five ingots were melted using the standard, consumable-electrode arc-melting techniques described earlier.

TABLE 3. MELTING DATA FOR LARGE CHROMIUM-BASE-ALLOY INGOTS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Ingot Size, lb	As-Cast Hardness, VHN	Melting Procedure(a)
<u>Lunex Chromium Base</u>				
C 124	100 Cr	6.4	137	S
C 125	100 Cr	6.2	--	S
C 126	1 Nb	6.0	413	S
C 96	2 Mo, 1 Y	2.0	200	S
C 127	2 Mo, 1 Y	6.2	345	S
<u>Iodide Chromium Base</u>				
C 196	100 Cr	2.6	135	S
C 194	1 Y	2.9	130	S
C 195	4 Ta	2.6	225	S
C 197	100 Cr	2.6	140(b)	M
C 198	1 Ti	2.6	203(b)	M
C 201	1 Y	2.8	142(b)	M
C 202	5 Ta	2.8	237(b)	M
C 203	2 Nb	2.8	209(b)	M
C 204	7.5 Mo	4.4	197(b)	M
C 202	5 Ta	1.0	275(b)	I
C 203	2 Nb	2.4	195(b)	I

(a) S designates standard consumable-electrode arc-melting procedure.

M designates modified consumable-electrode arc-melting procedure.

I designates induction-melting procedure.

(b) Converted from Rockwell A hardness values.

Each of the ingots showed surface porosity and a shrinkage cavity. These defects were more severe in the alloys than in the unalloyed chromium. In addition, each of the alloy ingots showed many cracks emanating from the shrinkage pipe.

Several heavy slag layers formed and were entrapped in the 2 w/o molybdenum-1 w/o yttrium ingot (C 96) made from the hydrogen-sintered compacts. Only superficial surface slagging occurred on the same alloy made from the lithium-treated chromium while no slag was observed on any of the other Lunex chromium ingots. This shows that yttrium exerts a powerful slagging action for the impurities contained in Lunex chromium and that the impurity content of the Lunex chromium was significantly reduced by

~~SECRET~~

15

the lithium-sintering treatment. However, as shown in Table 3, the lithium-treated alloys were much harder than alloys of the same nominal compositions which had not been subjected to this treatment. This indicates that a countercontamination or reaction occurred in the alloys as a result of the lithium treatment.

Iodide Chromium-Base Alloys. From the foregoing work, it was not clear as to whether the cracks observed in the Lunex chromium-alloy ingots were inherent to the melting procedure or whether these were associated with the relatively high impurity content of this chromium. To check this point, the first three iodide chromium-base ingots (C 194-196, inclusive) were melted using the same techniques.

Visual and radiographic examination indicated the unalloyed chromium and 1 w/o yttrium ingots (C 196 and C 194, respectively) were free of cracks while the 4 w/o tantalum ingot (C 195) showed a single, shallow shrinkage crack on top. However, after machining all surfaces of each ingot, many hairline cracks were apparent on all surfaces of the 4 w/o tantalum ingot as well as a few fine cracks on the ends only (i.e., original top and bottom) of the 1 w/o yttrium ingot. These cracks apparently formed as a result of the thermal stresses set up during the extremely rapid cooling of the ingots after melting.

In an attempt to eliminate this difficulty, the melting technique for the next alloy (1 w/o titanium, Ingot C 198) was modified by reducing the power input in the final melting stage and by cutting back the flow of cooling water around the crucible. Also, the ingot was removed from the crucible while it was still hot (surface temperature about 500 F) and immediately stress-relief annealed for 1 hr at 2000 F. Visual and radiographic inspection of this ingot, after machining all surfaces, showed it was free of defects. Consequently, this melting procedure was used to prepare Ingots C 201-204, inclusive. However, subsequent inspection of these four ingots showed that each contained radial cracks over its entire length. It was thus apparent that, because of their sensitivity to thermal cracking, cold-mold arc-melting techniques are not suitable for the preparation of large chromium-base-alloy ingots containing over about 1 w/o of alloy addition.

Subsequently, two ingots were used to explore the feasibility of induction melting. Initially, melting and slow cooling of the 5 w/o tantalum alloy (C 202) was attempted using a vacuum atmosphere and a beryllia crucible. While the resulting ingot was crack-free, weight losses (due to chromium volatilization) were excessive and the crucible cracked during solidification. In the second experiment, the 2 w/o niobium alloy (C 203) was successfully melted in a beryllia crucible and cast, under a helium atmosphere, into a 1-1/8-in.-diameter cylindrical zirconite mold. After solidification, the still hot ingot was removed and stress-relief annealed 1 hr at 2000 F. Visual, radiographic, and metallographic examination on sections through this ingot showed it to be sound and free of cracks.

Hardness, Structure, and Oxidation Resistance

Iodide Chromium-Base Alloys

The hardness of each of the binary screening alloys was determined in both the as-cast condition and after annealing for 24 hr at 2000 F and water quenching. These data

~~SECRET~~

TABLE 4. HARDNESS- AND OXIDATION-TEST DATA FOR IODIDE CHROMIUM-BASE SCREENING ALLOYS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hardness, VHN		Weight Change After 100 Hr in Air at 2000 F, mg per cm ²	
		As Cast	Annealed at 2000 F ^(a)	Gain	Loss ^(b)
C 180	100 Cr	139	128	10.5	13.9
C 128	0.5 Y	133	128	0.37	1.12
C 129	0.75 Y	130	128	0.73	0.93
C 130	1 Y	132	126	0.36	0.57
C 131	1.5 Y	133	127	0.26	0.26
C 132	2 Y	132	129	0.92	0.86
C 133	1 Nb	205	196	7.1	9.0
C 120	1.62 Nb ^(c)	271	245	10.0	22.0
C 181	2 Nb	255	215	--	--
C 134	2.5 Nb	307	242	18.8	21.7
C 135	5 Nb	352	286	36.6	25.3
C 136	7.5 Nb	387	321	44.8	34.6
C 137	10 Nb	435	370	53.2	56.5
C 138	1 Ta	156	143	7.6	5.2
C 139	2.5 Ta	213	195	10.8	3.2
C 121	4.91 Ta ^(c)	287	236	11.0	5.9
C 182	5 Ta	275	212	--	--
C 140	7.5 Ta	343	254	13.6	3.8
C 141	10 Ta	371	273	14.0	4.6
C 142	1 Mo	140	131	9.3	11.1
C 185	2.5 Mo	163	174	12.9	23.4
C 122	4.38 Mo ^(c)	253	240	5.2	+5.2
C 183	5 Mo	235	234	--	--
C 186	7.5 Mo	281	298	6.7	17.4
C 143	10 Mo	315	314	7.1	+5.9
C 144	20 Mo	433	419	5.2	+4.8
C 145	1 W	141	142	5.2	12.4
C 187	2.5 W	154	170	18.0	30.2
C 123	5.21 W ^(c)	201	201	5.9	9.5
C 184	5 W	200	206	--	--
C 188	7.5 W	236	218	20.0	27.1
C 146	10 W	274	276	16.9	14.4
C 147	20 W	398	398	6.0	15.3

TABLE 4. (Continued)

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hardness, VHN		Weight Change After 100 Hr in Air at 2000 F, mg per cm ²	
		As Cast	Annealed at 2000 F(a)	Gain	Loss(b)
C 148	1 V	150	143	8.3	14.3
C 149	5 V	185	186	9.6	10.6
C 150	10 V	230	226	8.2	10.0
C 151	20 V	292	280	>350	>200
C 152	1 Ru	128	133	12.7	20.2
C 153	5 Ru	207	215	9.0	9.1
C 154	10 Ru	279	286	13.0	17.8
C 155	20 Ru	355	360	23.8	24.6
C 156	1 Re	136	133	15.7	20.8
C 157	5 Re	152	156	5.7	14.8
C 158	10 Re	182	186	13.5	+5.0
C 159	20 Re	245	247	8.6	16.9
C 164	1 Fe	165	219	6.5	15.9
C 165	5 Fe	265	285	9.2	14.7
C 166	10 Fe	358	362	10.7	18.7
C 167	20 Fe	441	436	19.8	34.7
C 168	1 Mn	131	138	10.6	14.9
C 169	5 Mn	127	138	7.0	17.2
C 170	10 Mn	131	140	15.1	20.3
C 171	20 Mn	144	139	14.4	26.1
C 172	1 Zr	139	149	5.3	15.7
C 173	5 Zr	187	193	12.5	+0.38
C 174	10 Zr	273	266	8.5	+3.2
C 175	20 Zr	465	433	20.0	+14.1
C 176	1 Ti	182	177	14.9	31.6
C 177	5 Ti	338	329	20.0	41.6
C 178	10 Ti	440	459	23.0	47.0
C 179	20 Ti	608	729	17.8	33.7
C 189	4 Ta, 0.5 Y	250	262	4.9	7.1
C 190	4 Ta, 1 Y	262	258	7.8	0.34
C 191	4 Ta, 1.5 Y	243	247	8.5	+2.3
C 192	4 Ta, 2 Y	242	243	5.8	+1.2
C 193	2 Nb, 1 Y	251	254	6.1	3.4

(a) Water quenched after 24 hr at 2000 F under a hydrogen atmosphere.

(b) After wire brushing to remove loosely adherent surface oxide.

(c) Composition determined by analysis.

SECRET

are listed with the alloy compositions in Table 4 and are plotted as a function of alloy composition in Figure 2.

With the exception of the yttrium and manganese alloys, increasing the amount of each of the binary addition elements resulted in alloys of progressively greater hardnesses. Yttrium, in amounts up to 2 w/o, and manganese, in amounts up to nominally* 20 w/o, showed no significant effect on the hardness of chromium.

Generally, on an equivalent weight per cent addition basis, the hardening effect of the various addition metals on as-cast chromium increases in the following order: yttrium, manganese, rhenium, vanadium, zirconium, tungsten, ruthenium, molybdenum, iron, tantalum, titanium, and niobium.

With the exception of the niobium and tantalum alloys, the 2000 F annealing treatment had no significant effect on the hardness of the as-cast screening alloys. The hardnesses of these two binary alloy series, at compositions of 5 w/o and greater, were reduced on the order of 25 to 75 VHN after heat treatment. Metallographic examination showed that this hardness decrease resulted primarily from the precipitation at 2000 F of additional amounts of the Cr_2Nb and Cr_2Ta phases in these respective binary alloys.

On the basis of metallographic studies on these and the other heat-treated alloy samples, the extent of the solid solubility of the various binary addition elements in chromium at 2000 F was determined as shown in Table 5. The data for the iron, molybdenum, titanium, tungsten, and zirconium alloys are consistent with the published phase relationships for these respective binary-alloy systems. Figure 3 shows representative two-phase structures of the types observed in these alloys.

The results of the 100-hr oxidation tests at 2000 F on the screening-alloy series are also given in Table 4. Pertinent observations concerning the specific effects of the alloy additions are summarized as follows:

TABLE 5. EXTENT OF SOLID SOLUBILITY OF VARIOUS BINARY ALLOYING ADDITION ELEMENTS IN CHROMIUM AT 2000 F

Addition Element	Range of Solid Solubility, w/o	Addition Element	Range of Solid Solubility, w/o
Yttrium	<0.5	Ruthenium	10-20
Zirconium	<1	Rhenium	10-20
Niobium	<1	Vanadium	>20
Tantalum	1-2.5	Iron	>20
Manganese	1-5(a)	Molybdenum	>20
Titanium	5-10	Tungsten	>20

(a) Because of the relatively high vapor pressure of molten manganese, it is doubtful that the nominal manganese contents were realized in these arc-melted alloys.

*Because of the relatively high vapor pressure of molten manganese, it is doubtful that the nominal manganese contents were realized in these arc-melted alloys.

500X

N44706

- a. 1 w/o Niobium (Ingot C 133), Showing Fine Precipitate of Cr₂Nb Particles in Chromium-Solid-Solution Matrix

500X

N44708

- b. 10 w/o Niobium (Ingot C 137), Showing Primary Chromium Solid Solution (Large Dendrites), Containing Fine Particles of Cr₂Nb, Plus an Interdendritic Mixture of Chromium Particles in Cr₂Nb

FIGURE 3. REPRESENTATIVE MICROSTRUCTURES IN IODIDE CHROMIUM-BASE NIOBIUM ALLOYS, AFTER WATER QUENCHING FROM 2000 F

Etchant: 10 per cent oxalic acid.

SECRET

FIGURE 2. HARDNESSES OF VARIOUS IODIDE CHROMIUM-BASE ALLOYS, AS CAST AND AFTER ANNEALING FOR 24 HR AT 2000 F

SECRET

Binary additions of (1) 0.5 through 2 w/o yttrium, (2) 5 through 20 w/o of molybdenum or zirconium, and (3) 1 through 10 w/o tantalum were the only additions significantly effective in improving the 2000 F oxidation resistance of chromium. Of these addition metals, yttrium is by far the most effective. As shown by the weight-change data, yttrium in amounts as low as 0.5 w/o markedly lowers the oxidation rate and scaling loss of chromium. Moreover, as shown by the data in Table 6, yttrium is also very effective in reducing the amount of nitrogen pickup by chromium exposed to air at 2000 F for 100 hr. The data in Tables 4 and 6 indicate that the optimum amount of yttrium to minimize the oxidation rate and nitrogen pickup of chromium is about 1 w/o.

TABLE 6. EFFECT OF YTTRIUM IN REDUCING THE NITROGEN CONTAMINATION OF CHROMIUM EXPOSED FOR 100 HR IN AIR AT 2000 F

Ingot	Nominal Alloy Content	Nitrogen Content, w/o
	(Balance Chromium), w/o	
C 180	100 Cr	0.36
C 128	0.5 Y	0.012
C 129	0.75 Y	0.016
C 130	1.0 Y	0.002
C 131	1.5 Y	0.009
C 132	2.0 Y	0.076

The chief virtue of the molybdenum and zirconium additions is that both result in alloys with fairly adherent surface oxides. With the exception of the molybdenum, yttrium, and zirconium alloys, all of the other binary alloys tested at 2000 F were relatively free-scaling.

Vanadium additions of 1 through 10 w/o had no significant effect on the weight changes of chromium in the oxidation test. The 20 w/o vanadium alloy, however, formed a liquid oxide at 2000 F and showed very high weight gains indicative of catastrophic oxidation.

Additions of 1 through 20 w/o of manganese, rhenium, ruthenium, or tungsten resulted in alloys which generally showed slightly inferior oxidation resistance at 2000 F to unalloyed chromium.

On the other hand, binary additions over the same composition range of niobium and titanium resulted in alloys with significantly higher oxidation rates and scaling losses. In this regard, the effectiveness of yttrium as a ternary addition in combination with niobium and tantalum is of extreme interest. Thus, the oxidation data for the ternary niobium-yttrium and tantalum-yttrium alloys show that yttrium definitely improves the oxidation resistance of these binary alloys. While the performance of the ternary alloys is not so good as that for the binary yttrium alloys, the ternary alloys show oxidation resistance superior to that of unalloyed chromium at 2000 F.

Lunex Chromium-Base Alloys

As noted earlier, the base material for this study was shifted to iodide chromium when it was learned that the Lunex chromium (the intended base material) was contaminated with 0.13 w/o carbon. Prior to this changeover, the evaluation of 28 binary screening alloys made using the Lunex chromium was in process. Table 7 shows the hardness and 2000 F oxidation-resistance data which had been obtained on these alloys.

Comparison of these data with those given in Table 4 for the analogous iodide chromium-base alloys showed the following points of interest:

- (1) The Lunex chromium and the alloys prepared from it generally showed slightly higher hardnesses.
- (2) Yttrium additions of 1 to 5 w/o to the Lunex chromium reduced its hardness to the level of that obtained in the iodide chromium.
- (3) The effects of molybdenum, niobium, tungsten, and yttrium on the oxidation resistance of the Lunex chromium base were generally similar to those obtained using the iodide chromium base. The most notable exception here was that the oxidation products formed on the 10 and 20 w/o molybdenum alloys made with Lunex chromium were free-scaling.

The structures of the Lunex chromium-base alloys were complicated by the presence of numerous carbides in the base material, as illustrated in Figure 1. Molybdenum and tungsten additions up through 20 w/o had no significant effects on this structure. Both carbides and Cr_2Nb particles were observed in the Lunex chromium-1 w/o niobium alloy quenched from 2000 F. With additions of 5 w/o niobium and greater, the niobium and carbon apparently combined to form a lower melting phase based on the compound Cr_2Nb . Thus, each of the as-cast 5, 10, and 20 w/o niobium alloys contained an appreciable volume of a eutectic mixture of this phase and the terminal chromium solid solution. In contrast, only traces of the eutectic mixture were observed in the as-cast 10 w/o niobium alloy made with iodide chromium (see Figure 3). The indicated eutectic composition for the ternary chromium-0.13 w/o carbon-niobium alloys was placed between 10 and 20 w/o niobium on the basis of metallographic examination of the as-cast samples.

Fabricability

Screening Alloys

Table 8 lists the conditions used to study the workability of the sheathed iodide chromium-base screening alloys and the results obtained.

Unfortunately, the choice of hot-working temperatures for the niobium and tantalum alloys was too low, those alloys being the first in this series to be fabricated. On the other hand, the fabrication temperatures for some of the other alloys were obviously too high. Thus, partial liquation occurred from the interaction of the 20 w/o vanadium,

~~SECRET~~

23

TABLE 7. HARDNESS- AND OXIDATION-TEST DATA FOR LUNEX CHROMIUM-BASE SCREENING ALLOYS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hardness, VHN		Weight Change After 100 Hr in Air at 2000 F, mg per cm ²	
		As Cast	Annealed at 2000 F ^(a)	Gain	Loss ^(b)
C 180	100 Cr ^(c)	139	128	10.5	13.9
C 76	100 Cr	153	167	2.4	6.1
C 77	1 Y	141	139	1.3	0.18
C 78	2 Y	133	143	1.6	0.00
C 79	5 Y	131	142	1.5	0.06
C 80	1 Mo	182	190	17.2	29.9
C 81	5 Mo	274	277	12.6	+7.4
C 82	10 Mo	336	341	11.2	14.4
C 83	20 Mo	459	447	9.1	19.3
C 84	1 Nb	203	191	23.4	28.3
C 85	5 Nb	251	305	26.5	48.9
C 86	10 Nb	463	406	--	--
C 87	20 Nb	724	697	--	--
C 88	1 W	166	175	20.7	42.5
C 89	5 W	221	231	17.6	37.6
C 90	10 W	329	301	12.2	32.5
C 91	20 W	416	--	--	--
C 92	1 Fe	171	--	--	--
C 93	5 Fe	296	--	--	--
C 94	10 Fe	381	--	--	--
C 95	20 Fe	438	--	--	--
C 99	1 Mn	144	--	--	--
C 100	5 Mn	142	--	--	--
C 101	10 Mn	155	--	--	--
C 102	20 Mn	144	--	--	--

(a) Water quenched after 24 hr at 2000 F under a hydrogen atmosphere.

(b) After wire brushing to remove loosely adherent surface oxide.

(c) Iodide chromium control sample.

~~SECRET~~

SECRET

24

TABLE 8. FABRICATION DATA FOR IODIDE CHROMIUM-BASE SCREENING ALLOYS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hot-Working Temperature, F	Sheath Material	Type of Hot Working ^(a)	Reduction in Area in Working ^(b) , per cent	Workability Rating ^(c)
C 180	100 Cr	2300	Nickel	S	73	E
C 128	0.5 Y	2300	Nickel	S	74	E
C 129	0.75 Y	2300	Nickel	S	75	E
C 130	1 Y	2300	Nickel	S	74	E
C 131	1.5 Y	2300	Nickel	S	75	E
C 132	2 Y	2300	Nickel	S	75	E
C 133	1 Nb	2300	Nickel	S	66	G
C 120	1.62 Nb ^(d)	2200	Stainless steel ^(e)	S	46	P
C 181	2 Nb	2300	Nickel	S	55	G
C 134	2.5 Nb	2300	Nickel	S	58	P
C 135	5 Nb	2300	Nickel	S	36	NG
C 136	7.5 Nb	2300	Nickel	S	30	NG
C 137	10 Nb	2300	Nickel	S	22	NG
C 138	1 Ta	2300	Nickel	S	66	E
C 139	2.5 Ta	2300	Nickel	S	68	G
C 121	4.91 Ta ^(d)	2200	Stainless steel ^(e)	S	44	P
C 182	5 Ta	2300	Nickel	S	60	G-P
C 140	7.5 Ta	2300	Nickel	S	55	G-P
C 141	10 Ta	2300	Nickel	S	60	NG
C 142	1 Mo	2300	Nickel	S	72	E
C 185	2.5 Mo	2300	Nickel	R	64	E
C 122	4.38 Mo ^(d)	2200	Stainless steel ^(e)	S	46	P
C 183	5 Mo	2650	Molybdenum	R	66	E
C 186	7.5 Mo	2650	Molybdenum	R	61	E
C 143	10 Mo	2300	Nickel	S	47	NG
C 144	20 Mo	2300	Nickel	S	42	NG
C 145	1 W	2300	Nickel	R	66	E
C 187	2.5 W	2300	Nickel	R	65	E
C 123	5.21 W ^(d)	2200	Stainless steel ^(e)	S	49	P
C 184	5 W	2650	Molybdenum	R	71	E
C 188	7.5 W	2650	Molybdenum	R	69	E
C 146	10 W	2400	Molybdenum	R	54	NG
C 147	20 W	2650	Molybdenum	R	50	NG
C 148	1 V	2300	Nickel	R	56	P
C 149	5 V	2300	Nickel	R	53	NG
C 150	10 V	2650	Molybdenum	R	44	NG
C 151	20 V	2650	Molybdenum	R	--	NG ^(f)
C 152	1 Ru	2300	Nickel	R	66	E
C 153	5 Ru	2650	Molybdenum	R	68	E
C 154	10 Ru	2400	Molybdenum	R	66	P
C 155	20 Ru	2650	Molybdenum	R	68	E

SECRET

~~SECRET~~

25

TABLE 8. (Continued)

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hot-Working Temperature, F	Sheath Material	Type of Hot Working ^(a)	Reduction in Area in Working ^(b) , per cent	Workability Rating ^(c)
C 156	1 Re	2300	Nickel	R	66	E
C 157	5 Re	2300	Nickel	R	62	E
C 158	10 Re	2300	Nickel	R	62	E
C 159	20 Re	2650	Molybdenum	R	49	NG
C 164	1 Fe	2300	Nickel	R	64	E
C 165	5 Fe	2650	Molybdenum	R	67	E
C 166	10 Fe	2650	Molybdenum	R	65	E
C 167	20 Fe	2650	Molybdenum	R	66	E
C 168	1 Mn	2300	Nickel	R	66	E
C 169	5 Mn	2300	Nickel	R	67	E
C 170	10 Mn	(g)	--	--	--	--
C 171	20 Mn	2300	Nickel	R	57	E
C 172	1 Zr	2300	Nickel	R	45	P
C 173	5 Zr	2300	Nickel	R	45	P
C 174	10 Zr	2650	Molybdenum	R	66	P
C 175	20 Zr	2650	Molybdenum	R	--	NG
C 176	1 Ti	2300	Nickel	R	52	E
C 177	5 Ti	2650	Molybdenum	R	--	NG
C 178	10 Ti	2650	Molybdenum	R	--	NG ^(f)
C 179	20 Ti	2650	Molybdenum	R	--	NG ^(f)
C 189	4 Ta, 0.5 Y	2650	Molybdenum	R	67	E
C 190	4 Ta, 1 Y	2650	Molybdenum	R	68	E
C 191	4 Ta, 1.5 Y	2650	Molybdenum	R	67	E
C 192	4 Ta, 2 Y	2650	Molybdenum	R	67	E
C 193	2 Nb, 1 Y	2650	Molybdenum	R	--	NG ^(f)

(a) S designates swaging; R designates rod rolling.

(b) Actual reduction of alloy after hot working in sheath to an over-all total reduction of 75 per cent.

(c) E designates excellent; sound rod with no apparent defects.

G designates good; rod separated into two or three pieces which showed no surface defects.

P designates poor; rod separated into two or three pieces which showed transverse cracks.

NG designates no good; alloy cylinder broke into many pieces with many transverse cracks.

(d) Composition determined by analysis.

(e) Type 304.

(f) Partial liquation resulted from reaction of these alloys with their sheath material during fabrication.

(g) Ingot broke during attempted grinding to cylindrical shape.

~~SECRET~~

~~SECRET~~

26

10 and 20 w/o titanium, and 2 w/o niobium-1 w/o yttrium alloys with their respective sheath materials during fabrication.

Nevertheless, these experiments provided a good insight into the comparative fabricability of the alloys studied. On the basis of this work, the workable ranges of alloy content in the 12 binary systems were defined approximately as follows:

<u>Addition Element</u>	<u>Maximum Alloy Content for Good Workability, w/o</u>	<u>Addition Element</u>	<u>Maximum Alloy Content for Good Workability, w/o</u>
Vanadium	<1	Molybdenum	7.5-10
Zirconium	<1	Tungsten	7.5-10
Titanium	1-5	Rhenium	10-20
Yttrium	>2	Manganese	>20 ^(a)
Niobium	2-4	Iron	>20
Tantalum	2.5-5	Ruthenium	>20

(a) Because of the relatively high vapor pressure of molten manganese, it is doubtful that the nominal manganese contents were realized in these arc-melted alloys.

The results of similar fabrication experiments on a limited number of the Lunex chromium-base screening alloys are given in Table 9. With the exception of a single, unalloyed control sample (C 125), none of these alloys fabricated to sound material under the conditions used. While the fabrication temperatures were admittedly low, the workability of these alloys appeared decidedly inferior to that for the analogous iodide chromium-base alloys. This is evident from a comparison in Tables 8 and 9, of the reductions in area and workability ratings for the following analogous alloys which were fabricated under identical conditions:

<u>Nominal Alloy Content (Balance Chromium), w/o</u>	<u>Iodide Chromium Base</u>			<u>Lunex Chromium Base</u>		
	<u>Reduction in Area</u>			<u>Reduction in Area</u>		
	<u>Ingot</u>	<u>in Working, per cent</u>	<u>Workability Rating^(a)</u>	<u>Ingot</u>	<u>in Working, per cent</u>	<u>Workability Rating^(a)</u>
100 Cr	C 75	46	E	C 76	39	NG
	C 80	73	E	C 125	71	G
5 Mo	C 122	46	P	C 81	53	NG
5 W	C 123	49	P	C 89	42	NG

(a) See Table 8.

Large Alloy Ingots

All fabrication experiments on the large ingots were conducted without the use of sheath materials to explore the feasibility of developing a suitable fabrication process for semicommercial- or commercial-size ingots.

~~SECRET~~

TABLE 9. FABRICATION DATA FOR LUNEX CHROMIUM-BASE SCREENING ALLOYS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Hot-Swaging Temperature, F	Sheath Material	Reduction in Area in Working ^(a) , per cent	Workability Rating ^(b)
C 75	100 Cr ^(c)	1650	Mild steel	46	E
C 180	100 Cr ^(c)	2300	Nickel	73	E
C 76	100 Cr	1650	Mild steel	39	NG
C 125	100 Cr ^(d)	2300	Nickel	71	G
C 77	1 Y	1650	Mild steel	47	NG
C 78	2 Y	1650	Mild steel	47	NG
C 79	5 Y	1650	Mild steel	49	NG
C 80	1 Mo	2200	Stainless steel ^(e)	49	NG
C 81	5 Mo	2200	Stainless steel	53	NG
C 82	10 Mo	(f)	--	--	--
C 83	20 Mo	2200	Stainless steel	32	NG
C 84	1 Nb	2200	Stainless steel	41	NG
C 126	1 Nb ^(d)	2300	Nickel	33	NG
C 85	5 Nb	2200	Stainless steel	33	NG
C 86	10 Nb	(f)	--	--	--
C 87	20 Nb	(f)	--	--	--
C 88	1 W	2200	Stainless steel	51	NG
C 89	5 W	2200	Stainless steel	42	NG
C 90	10 W	2200	Stainless steel	40	NG
C 91	20 W	(f)	--	--	--
C 127	2 Mo, 1 Y ^(d)	2300	Nickel	--	NG

(a) Actual reductions of alloys after swaging to sheathed reductions of 56 per cent; samples C 125, C 126, and C 180 received sheathed reductions of 75 per cent.

(b) E designates excellent; sound rod with no apparent defects.

G designates good; rod separated into two or three pieces which showed no surface defects.

NG designates no good; alloy cylinder broke into many pieces with many transverse cracks.

(c) Iodide chromium control samples.

(d) Alloy samples ground from lithium-treated consumable-electrode arc-melted ingots.

(e) Type 304

(f) Ingots broke during attempted grinding to cylindrical shape.

~~SECRET~~

28

Initially, baseline experiments were carried out using the unalloyed iodide chromium ingots to explore various fabrication procedures. It was found that these ingots were amenable to fabrication by press forging or extrusion.

In the press-forging work, a 2-in.-round ingot (C 197) was forged on alternate sides using graded reductions of 1/16 to 1/8 in. to a 1-1/4-in.-square section, using frequent reheats to 2450 F. After this degree of reduction, the billet was finished to a sound 1 by 1-3/4 by 4-in. slab using three fairly heavy presses. This billet, however, developed severe transverse cracks during subsequent attempts to flat roll it to sheet using 10 per cent reductions per pass at 1650 F and 1830 F. Additional experiments confirmed the poor rolling characteristics of the press-forged slab in this temperature range but showed that excellent quality sheet could be obtained by rolling this material at temperatures from 1350 through 1525 F.

Extrusion of a 1-1/2-in.-diameter unalloyed chromium ingot (C 196) was carried out at 2450 F using a glass lubricant and an extrusion speed of 80 in. per min. Under these conditions, the chromium extruded readily under a maximum pressure of 92,000 psi into a 1/2-in.-diameter bar (reduction ratio of 9:1) of excellent quality. Samples of the extruded bar were successfully rod rolled at 1470 F and also flat rolled to excellent 0.060-in.-thick sheet at temperatures from 1350 through 1800 F.

Experiments in the fabrication of large chromium-base-alloy ingots were limited by virtue of the fact that no sound, crack-free alloy ingots were prepared until late in the program. Nevertheless, some success along these lines was achieved.

The 2-in.-diameter 1 w/o titanium alloy (C 198) was successfully press forged at 2600 F, using graded reductions of 1/16 to 1/8 in., to a 1-1/4-in.-square billet. While some end cracks developed, a sound 1-3/4-in. length of this billet was salvaged. This sample, however, developed deep transverse cracks on the first attempted pass through a diamond-oval-grooved rolling mill, using a preheating temperature of 2600 F.

Attempted press forging of a 1 w/o yttrium ingot (C 194) at 2200 F was unsuccessful due to the opening up of the end cracks present on the as-cast ingot. However, several block samples cut from the center portion of this ingot were subsequently flat rolled directly to good 0.060-in.-thick sheet at temperatures from 2200 to 2400 F.

Similarly, attempts to heal the cracks in the 4 w/o tantalum ingot (C 195) by extrusion were unsuccessful. In this experiment, a preheating temperature of 2600 F was used with conditions otherwise the same as those for the extrusion of the unalloyed chromium ingot. Very little deformation of the 4 w/o tantalum ingot was obtained even though a maximum extrusion pressure of 210,000 psi was reached on this ingot before the automatic pressure cutoff occurred. The presence of cracks in the ingot, the low extrusion speed, and the high attempted reduction ratio are all believed to be major reasons for the failure in this attempted extrusion.

In the last series of experiments, six block samples were cut from the induction-melted and cast 2 w/o niobium ingot (C 203) to study the effect of temperature on this alloy's workability. These samples were flat rolled to 50 per cent total reductions, using a 10 per cent reduction per pass, at 1600, 1800, 2000, 2200, 2400, and 2600 F. All of these samples behaved similarly and developed severe transverse cracks after

~~SECRET~~

~~SECRET~~

29

reductions of 10 to 20 per cent in thickness. Earlier work with a screening alloy of the same composition (see Table 8, Ingot C 181) showed that good quality rod could be obtained from this material by swaging it in a nickel sheath at 2300 F. Comparison of this finding with the results of the above experiment showed that the choice of hot-working temperature is clearly secondary to the method selected for hot working.

Hot Strength

Bend tests were conducted on samples of most of the fabricable screening alloys at 2000 and 2300 F. The results of this work are given in Table 10 which lists the fiber stresses calculated to produce an elongation of 0.5 per cent in the outer fibers of the individual alloys.

Generally, increasing additions of tungsten, molybdenum, niobium, tantalum, iron, manganese, ruthenium, and rhenium to chromium result in progressive increases in its strength at 2000 F. At this temperature, the maximum resistance to deformation was that shown by the 2 w/o niobium alloy which had an indicated strength of over 2-1/2 times that of unalloyed chromium.

At 2300 F, tungsten and molybdenum appear to be the most effective strengtheners for chromium. Maximum bend resistance was shown by the 7.5 w/o tungsten and 5 w/o molybdenum alloys, both of which had indicated strengths about twice that of unalloyed chromium. Binary additions of 2 w/o niobium, 10 w/o rhenium, 7.5 w/o tantalum, and 1 w/o iron also improve the 2300 F strength of chromium, but to lesser degrees. On the other hand, binary additions of 1 w/o zirconium or up through (nominally) 20 w/o manganese or ruthenium do not significantly improve the strength of chromium at 2300 F. Similarly, binary yttrium additions up through 2 w/o do not significantly affect the strength of chromium at 2000 or 2300 F. Further, the limited data on the ternary alloys in Table 10 indicate that yttrium additions to a 4 w/o tantalum-alloy base weaken and embrittle this alloy at 2000 and 2300 F.

DISCUSSION AND CONCLUSIONS

While no difficulties were encountered with the preparation of small alloy ingots using cold-mold arc-melting techniques, these methods do not appear suitable for the preparation of chromium-base alloys in sizes of 2 lb or larger. This stems from the susceptibility of the alloy ingots to develop radial cracks during solidification as a result of the severe thermal stresses imposed on them.

Induction melting and casting does appear feasible as a means of preparing large alloy ingots. In this regard, the present work confirms the experiences of earlier investigators with induction melting and casting of chromium-base alloys.^(1,2) However, additional work will be necessary to determine which refractories and mold materials are most suitable for chromium-base alloys.

~~SECRET~~

TABLE 10. RESULTS OF HOT-BEND TESTS ON VARIOUS IODIDE CHROMIUM-BASE ALLOYS

Ingot	Nominal Alloy Content (Balance Chromium), w/o	Stress to Produce 0.5 Per Cent Elongation in Outer Fiber of Bend Sample, psi	
		2000 F	2300 F
C 180	100 Cr	19,000	13,000
C 145	1 W	31,000	18,000
C 187	2.5 W	33,000	17,000
C 184	5 W	--	16,000
C 188	7.5 W	--	27,000
C 142	1 Mo	--	19,000
C 185	2.5 Mo	44,000	17,000
C 183	5 Mo	--	26,000
C 186	7.5 Mo	--	21,000
C 133	1 Nb	24,000	14,000
C 181	2 Nb	51,000	23,000
C 139	2.5 Ta	39,000	--
C 140	7.5 Ta	--	18,000
C 164	1 Fe	28,000	19,000
C 165	5 Fe	--	4,600
C 166	10 Fe	--	6,700
C 167	20 Fe	--	4,600
C 168	1 Mn ^(a)	26,000	14,000
C 169	5 Mn ^(a)	27,000	13,000
C 171	20 Mn ^(a)	--	13,000
C 152	1 Ru	27,000	12,000
C 153	5 Ru	37,000	14,000
C 155	20 Ru	14,000	9,000
C 156	1 Re	32,000	14,000
C 157	5 Re	35,000	15,000
C 158	10 Re	40,500	23,000
C 172	1 Zr	--	9,300
C 176	1 Ti	31,000	18,000
C 190	4 Ta, 1 Y	--	6,000 ^(b)
C 191	4 Ta, 1.5 Y	26,000 ^(b)	--
C 192	4 Ta, 2 Y	27,000 ^(b)	8,500 ^(c)

(a) Because of the relatively high vapor pressure of molten manganese, it is doubtful that the nominal manganese contents were realized in these arc-melted alloys.

(b) Sample fractured at bend stress slightly greater than that required to produce 0.5 per cent elongation.

(c) Sample fractured at fiber stress indicated which produced a calculated elongation of 0.4 per cent.

~~SECRET~~

31 and 32

Because of the difficulties associated with their melting, relatively little effort was given to the development of a fabrication procedure for large alloy ingots. Nevertheless, the work with the smaller ingots has shown that cast chromium-base alloys can be successfully hot worked by imposing some form of lateral constraint on the ingots during hot working. In this regard, extrusion, rod rolling or swaging, and closed-die press forging appear to offer the most promising methods for hot working.

Of all the fabricable alloys tested, the best combinations of oxidation resistance and strength, in the range of 2000 to 2300 F, were found in binary alloys containing 5 w/o molybdenum, 10 w/o tungsten, and 2 w/o niobium. This work confirmed the original findings of GE-ANPD regarding the extremely beneficial effect of yttrium on the oxidation resistance of chromium and showed that such arc-melted alloys have inherently good hot workability.

On the basis of this work, it is believed that optimum elevated-temperature properties in a chromium-base alloy could be realized through the use of molybdenum and/or tungsten as primary solid-solution strengtheners along with smaller amounts of niobium and/or iron as auxiliary strengtheners. It is further anticipated that a small addition of yttrium to such an alloy would substantially improve its oxidation resistance.

REFERENCES

- (1) Parke, R. M., and Bens, F. P., Symposium on Materials for Gas Turbines, ASTM, 1946, p 80.
- (2) Sully, A. H., Brandes, E. A., and Provan, A. G., "The Properties of Cast Chromium Alloys at Elevated Temperatures", J. Inst. Metals, 81 (1952-53), 569-584.

DJM/JWR/RIJ:bwl

~~SECRET~~