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ARC MELTING AND PROPERTIES OF TWO CHROMIUM-BASE ALLOYS

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by

A.M.Filippi

WESTINGHOUSE ASTRONUCLEAR LABORATORY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-10485
John Merutka, Project Manager
Wm. D. Klopp, Research Advisor

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FINAL REPORT

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NASA Lewis Research Center
Cleveland, Ohio
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FOREWORD

The work described herein was done at the Astronuclear Laboratory, Westinghouse Electric Corporation, under NASA Contract NAS 3-10485. Technical guidance was provided by Project Manager, Mr. John Merutka, and Research Advisor, Mr. William Klopp, of the Materials and Structures Research Division, NASA-Lewis Research Center.

ABSTRACT

An investigation of double consumable electrode arc melting, and the properties of material converted from ingots prepared by this melting technique, was made on two high strength chromium alloys, Cr-2Ta-0.1C-0.05Zr and Cr-7Mo-1.75Ta-0.095C-0.085Y. Consumable electrode arc melting proved unsuited for producing crack free ingots of these two compositions, although a sound low strength Cr-0.2Y ingot, and several of pure chromium, were prepared by the process. Investigation of changes in melting technique failed to uncover a procedure which would yield sound ingot of the two high strength alloys. Ingot examination revealed evidence that cracking occurred during solidification, and is related in part to the compositions selected for melting.

Although sound ingots of the alloys were not produced, some sound 3/8 inch diameter alloy rod was made and evaluated. The as-cast ingots were clad with unalloyed molybdenum or mild steel, hermetically sealed, extruded at 2200-2600°F and swaged at 2000-2400°F to final diameter. Microstructure, high temperature strength and oxidation behavior, and low temperature ductility were evaluated. Where property comparisons were made, the data obtained were similar to those reported on material prepared by induction melting.

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SUMMARY

An investigation of the use of consumable electrode arc melting techniques to produce ingot of two chromium-base alloys, Cr-7Mo-1.75Ta-0.095C-0.085Y and Cr-2Ta-0.1C-0.05Zr*, was made. The problem of gross ingot cracking was encountered and was observed to occur during or immediately after solidification. Examination of the as-cast structure of both alloys revealed that a large amount of precipitation occurred at grain boundaries and cracking followed intergranular paths. Both TaC and $Cr_{23}C_6$ were identified as the precipitating phases in the ingots with the $Cr_{23}C_6$ phase being found almost exclusively in the grain boundaries. Interpretation of the results of precipitate phase identification indicates severe liquidus depression occurs by carbon enrichment of the liquid as solidification proceeds. Hence, the combination of freezing point depression, and ingot stress due to thermal gradients and shrinkage, is believed to cause ingot failure by "hot tearing". Prevention of this mode of failure by varying cooling rate, ingot size, or melt agitation met with little success.

Although the ingot cracking problem was not overcome, some sound rod for property evaluation was successfully produced from the cracked ingots. Fabrication involved hermetic encasement in steel or molybdenum and processing to rod by hot extrusion and swaging. The yield of sound rod obtained with this approach was greatest for the Cr-2Ta-0.1C-0.05Zr composition.

Tensile properties at 1900°F, stress rupture properties at 2100°F, and reaction in air at 2400°F, were examined on both alloys. Impact and tensile ductile-to-brittle transition temperatures** were determined for the Cr-2Ta-0.1C-0.05Zr composition.

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*Compositions given in weight percent.

** Referred to as DBTT in latter text

Surface recession and weight gain appeared similar for both alloys exposed in air at 2400°F for 24 hours with a weight increase of 20-25 mg/cm^2, and a surface recession of 0.001 inch to 0.0015 inch, being typical. The Cr-2Ta-0.1C-0.05Zr composition exhibited a 1900°F yield strength of 37 ksi*, and a 3-4 hour rupture life at 2100°F and 15 ksi. The Cr-7Mo-1.75Ta-0.095C-0.085Y alloy had a 60 ksi yield strength and a 10-25 hour rupture life under equivalent conditions. An impact DBTT of about 600°F and tensile DBTT of room temperature were observed for the Cr-2Ta-0.1C-0.05Zr alloy in the wrought plus stress relieved condition.

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*Stress in kips per square inch, ksi, and temperature in Fahrenheit Degrees, °F, are used in the text. Common and international system units are used to describe data in tables and figures.

I. INTRODUCTION

The ever continuing requirement for advanced jet engine capability has spurred investigation of chromium as a base for high temperature alloys. Its high melting point, good oxidation resistance relative to the other group VIa and the group Va metals, intermediate density, and high elastic modulus, are attributes which suggest a strong potential for such application. The development of chromium as an engineering alloy base, however, has progressed slowly due principally to lack of low temperature ductility; a problem which is generally worsened by interstitial element contamination or intentional alloying. Because of this, engineering applications of chromium base alloys to date have been few. Alloying studies, however, continue to improve low temperature ductility, and increase elevated temperature strength.

Recent chromium base alloy development studies undertaken at the General Electric Co.(1) and at the Aeronautical Research Laboratories in Australia(2), have revealed that excellent high temperature properties are displayed by alloys strengthened through solid solution additions of molybdenum or tungsten, and/or dispersions of refractory carbide precipitates. Two promising alloys developed from the programs were Cr-2Ta-0.1C-0.05Ta, and Cr-7Mo-1.75Ta-0.095C-0.085Y. Strength is imparted in both compositions by formation of a precipitate dispersion of tantalum carbide. The Cr-7Mo-1.75Ta-0.095C-0.085Y composition is also solid solution strengthened by the molybdenum addition. Yttrium and zirconium impart special properties, the former serving to lower nitrogen embrittlement during elevated temperature air exposure, the latter causing morphological change in the TaC precipitate favoring improved high temperature strength.

The Cr-2Ta-0.1C-0.05Zr composition has been examined by Ryan using rod extruded from small non-consumable arc melted heats(2). Improved stress-rupture behavior without major increase in tensile DBTT is characteristic of this alloy, as compared to pure chromium. Study of the Cr-7Mo-1.75Ta-0.095C-0.085Y composition was made by Clark on rod produced from small induction melted heats(1). The molybdenum content of this alloy is reflected both in improved elevated temperature strength, and loss of ductility (increased DBTT), as compared to the Cr-2Ta-0.1C-0.05Zr alloy.

These two compositions were selected by NASA to be consumable arc melted, converted to rod, and tested in this program. A major purpose of the study was to determine whether the inherently clean consumable arc melting technique might yield material of superior properties compared to that produced by induction melting, the technique widely used to prepare chromium alloy ingots for development studies(1,3-6). Examination of whether reduction in oxygen and nitrogen contaminant levels of the basic metal might be effected by consumable arc melting, was also a study objective.

It was originally intended that 3 inch diameter arc cast ingot of each alloy be produced on this program, converted to 3/8 inch diameter rod by extrusion and swaging, and evaluated for property comparison with the data reported by Ryan, and by Clark. Furthermore, ingots of each alloy were to have been produced using three different types of chromium base material, to examine whether final product properties might reflect differences in starting material chemistry. However, fabrication and property characterization of the program alloys was accomplished only to a limited extent, because cast ingots could not be produced without serious cracking. Consequently, major effort was devoted to exploration of techniques to eliminate or minimize the ingot cracking problem.

Variation of melt parameters such as ingot size, cooling rate, molten pool agitation, melt current type, and electrode make-up, were examined in an attempt to find a procedure whereby sound ingots could be produced. The mechanism of ingot cracking was also studied.

II. MATERIALS AND EXPERIMENTAL PROCEDURES

Base Material

Electrolytic chromium from two sources, the Union Carbide Corporation and the Australian Department of Supply, and iodide chromium produced by the Materials Research Corporation, were supplied by NASA for use on this program. The impurity content of these materials as reported by the suppliers is displayed in Table 1. Also shown in the table are the results of Westinghouse check analysis for carbon, oxygen and nitrogen content.

Reasonable agreement was obtained between vendor and check analysis for the level of interstitial element contamination in these materials. The electrolytically produced Union Carbide chromium contained carbon, oxygen, and nitrogen each in the range approximately 50 to 80 ppm. A lower carbon and nitrogen level, around 10 ppm, and higher oxygen content, 200 ppm, is characteristic of the Australian material. The high purity of the iodide produced chromium is reflected in the low content of these elements, each of which is below 10 ppm.

To further characterize these materials, each was consolidated by tungsten-inert-gas arc melting in a water cooled copper mold. Photographs of these buttons are shown in Figure 1 along with the starting materials, and as-cast microstructures and hardness. The surface appearance and microstructure of the iodide material is consistent with its high purity. The appearance of the buttons produced from the electrolytic chromium paralleled that of the starting materials, with the "powdery deposit" Australian chromium yielding a duller as-cast surface. On the other hand, the Australian chromium produced a cleaner microstructure in comparison to the Union Carbide material. Hardness of the as-cast buttons followed microstructural cleanliness.

TABLE 1 - Analysis of Starting Material[1]

Element	Electrolytic Chromium Flake				Iodide Chromium	
	Union Carbide[2]		Australian		Matls. Research Corp.	
	Supplier	Check	Supplier[3]	Check[5]	Supplier[4]	Check[5]
C	70	52	10	10	.001	<5
O	50	69	210	200	6-7	6
N	80	64-71	10	7-10	.1-.5	4
H	10	--	--	--	.1-.2	--
S	90	--	10	--	--	--
Fe	90	--	3-4	--	14-23	--
Ni	--	--	3	--	.3-.5	--
Cu	--	--	3	--	.1	--
Ag	--	--	4	--	--	--
Si	--	--	4	--	10	--
Al	--	--	1	--	.3	--
Mg	--	--	2	--	.2	--
B	--	--	--	--	--	--
Na	--	--	<3	--	--	--
Ca	--	--	--	--	.1-.3	--
Pb	--	--	2	--	--	--
V	--	--	--	--	.3	--
Mn	--	--	.5	--	.1	--

1. PPM by weight
2. Supplier and Westinghouse check analysis obtained on a sample removed by the supplier from a 105 lb. lot of electrolytic flake.
3. Analysis reported to be representative of six approximately 10 lb. lots of electrolytic flake. Representative sample not supplied.
4. Minimum and maximum contamination level given in the analytical results of three lots of iodide chromium weighing a total of 50 lbs. Representative samples not supplied.
5. Analysis obtained on a sample chosen arbitrarily for one lot.

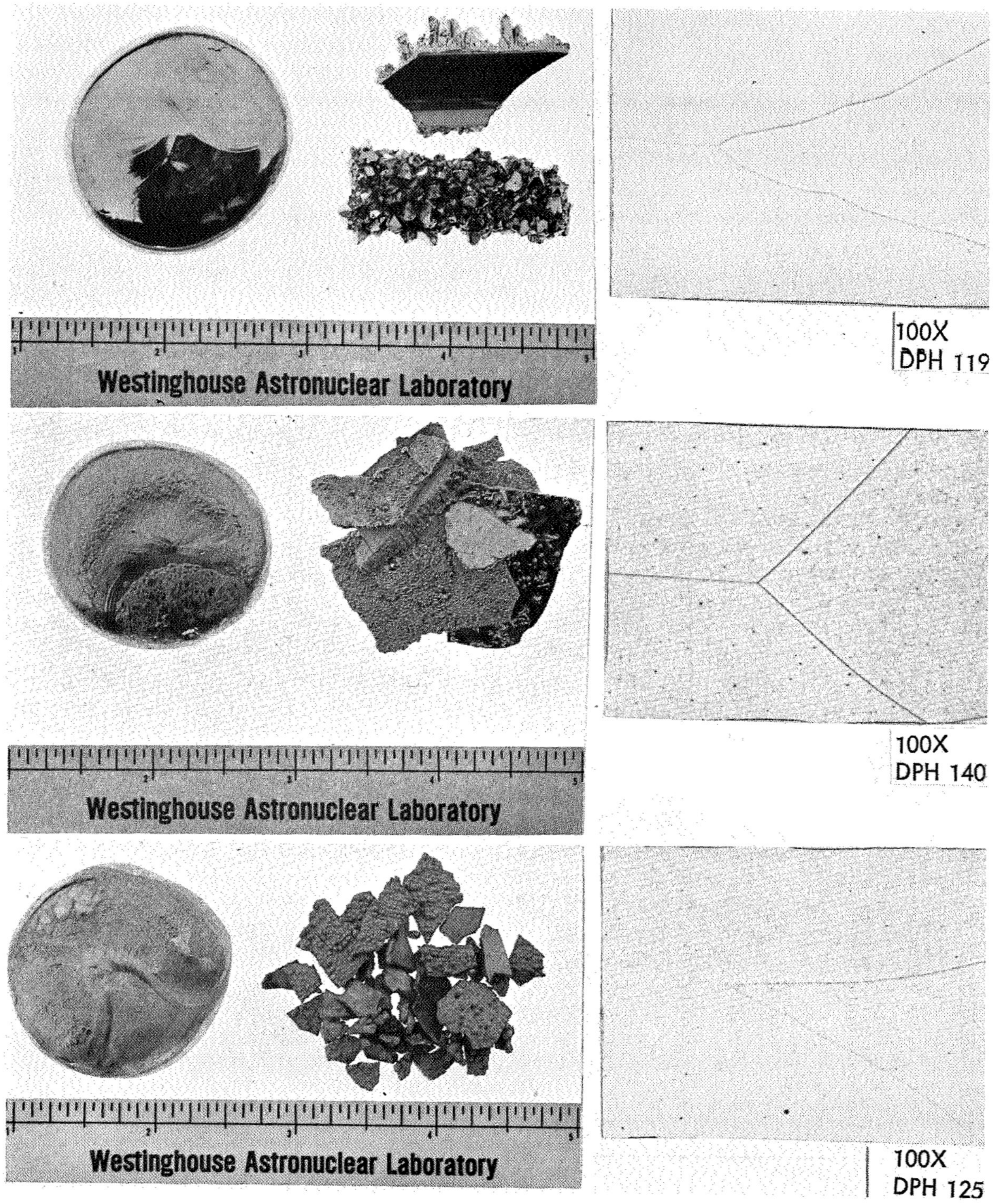


FIGURE 1 - Non-Consumable Arc Melted Buttons, Starting Materials, and As-Cast Microstructures of Chromium Sources.
Top: Iodide Chromium from Materials Research Corp.
Middle: Electrolytic Chromium from Union Carbide Corp.
Bottom: Electrolytic Chromium produced in Australia

It was an original intention of this program to produce arc cast ingot of the study alloys using as base material these individual chromium types. This would allow judgement of whether alloy properties would reflect differences in the base material. However, difficulty with cracking was encountered in the initial ingots, and study of all three chromium types was replaced by melting experiments in which only Union Carbide material was used.

Alloy Additions

Tantalum, molybdenum, and zirconium alloy additions were purchased as strip and foil of the highest purity commercially available. Yttrium was procured as a 1 inch thick rectangular cross-section ingot, and formed by hot and cold rolling to 0.003 inch foil for use in alloying. Graphite cloth was used to add carbon to the alloys.

Consumable Electrode Fabrication

Bars suitable for use in consumable electrodes were prepared by non-consumable arc melting in a water cooled copper trough mold, under 1/2 atmosphere of high purity argon gas. Both pure chromium, and chromium alloyed to the program compositions, were consolidated in this manner to produce bars nominally 3/8 inch x 1 inch x 22 inch, and weighing about 4-1/2 pounds. Alloyed bars were produced by placing the proper amount of each alloy addition uniformly along the length of the mold, and melting them with the chromium charge. The trough mold was slowly moved beneath the arc of the tungsten tip electrode to effect consolidation of the charge into a bar. The electrode entered the melt chamber through a ball joint seal, which provided some freedom in positioning and moving it during melting. Photographs of the equipment used for this purpose are shown in Figure 2a through 2c.

Preparation for trough melting involved chamber evacuation 1×10^{-5} torr (max.), a check of the chamber leak rate, backfill with 1/2 atmosphere of high purity argon (<5 ppm oxygen and nitrogen), and melting of a titanium "getter" button. Typical chamber leak rates obtained

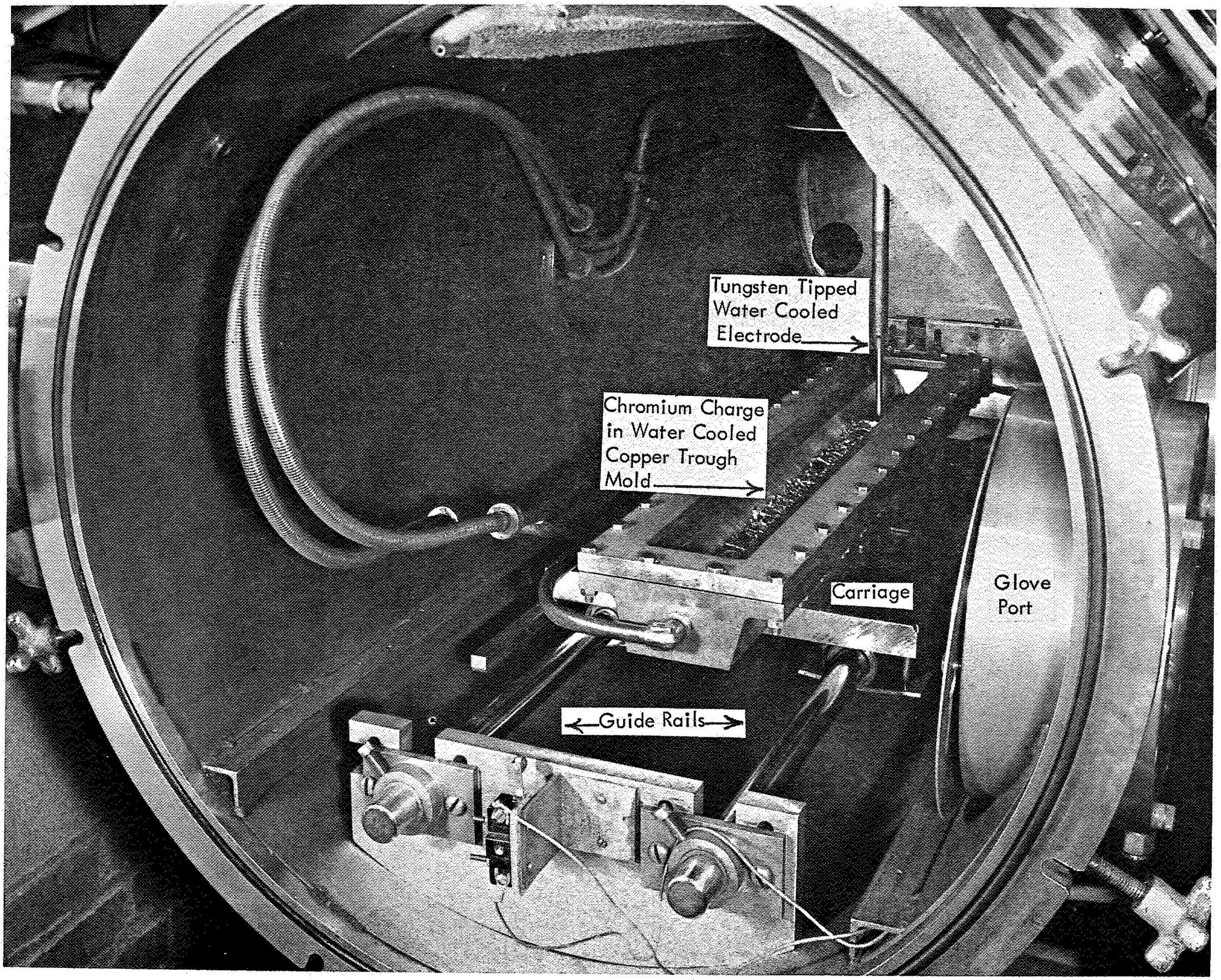


FIGURE 2a - View Into the Trough Melting Chamber. The Charge of Electrolytic Chromium has been Melted Over a Short Distance from One End.

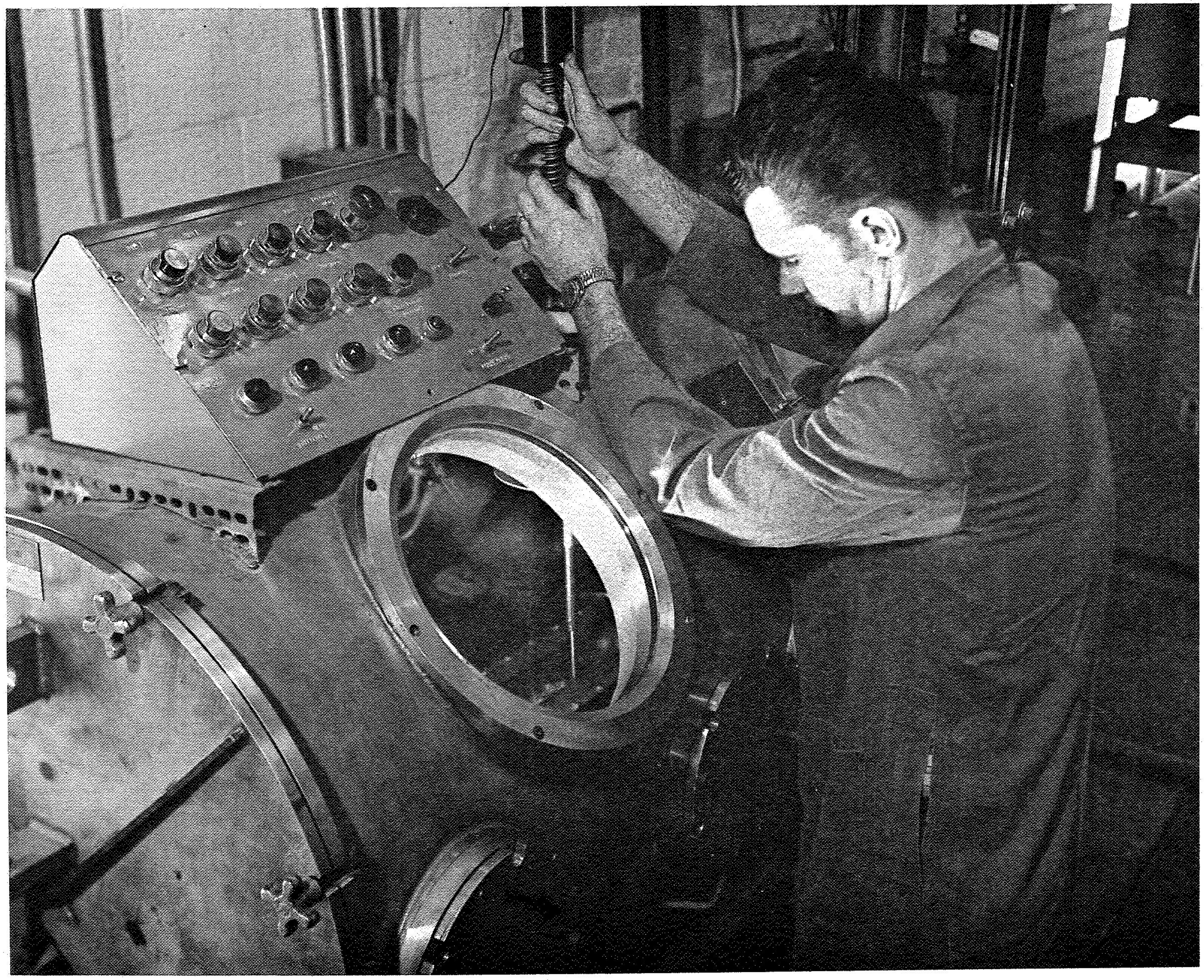


FIGURE 2b - Operator Trough Melting a Charge of Chromium

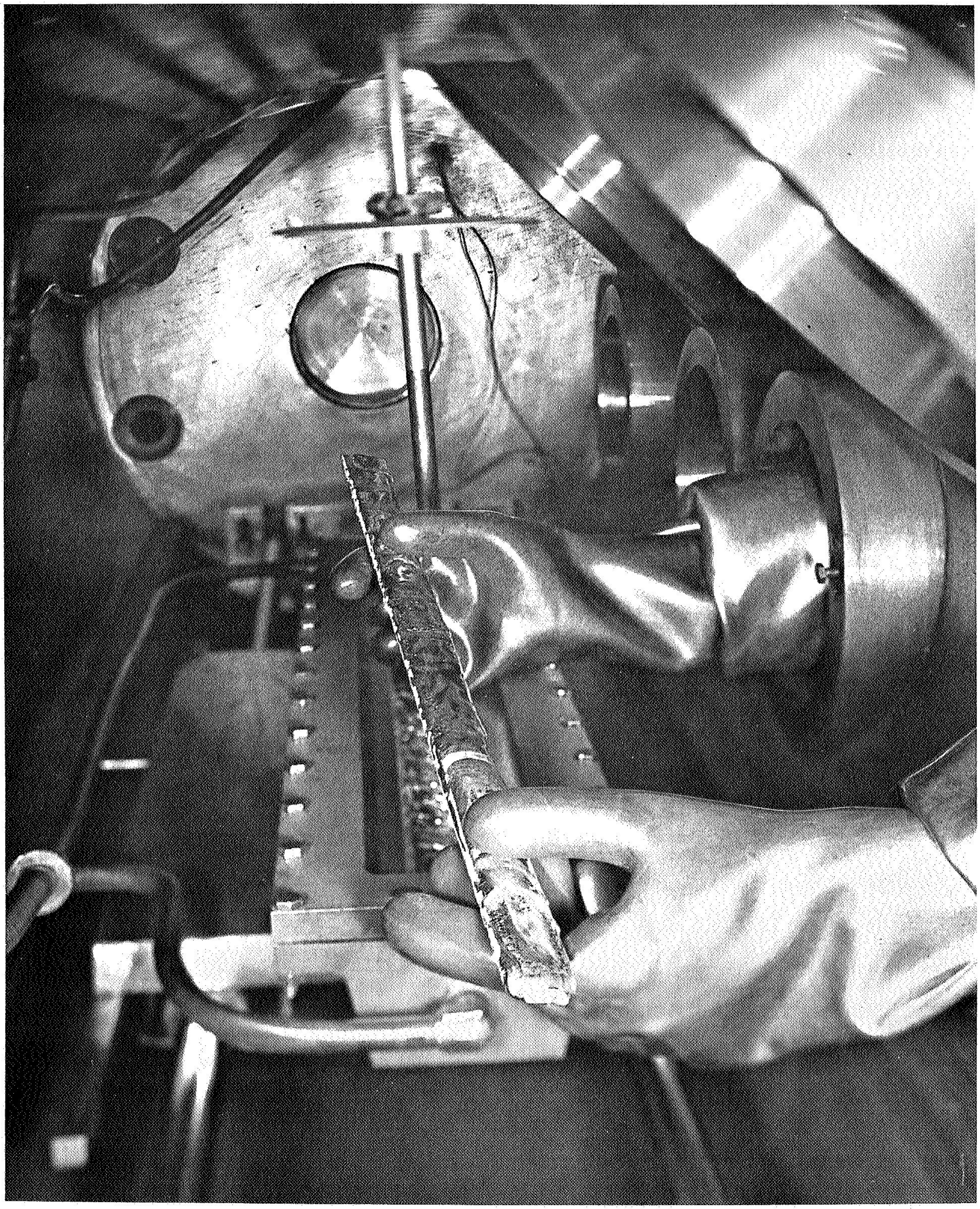


FIGURE 2c - Bar of Chromium Removed for Inspection After Trough Melting in preparation for melting a charge of Australian produced chromium were in the range 0.2 to 0.5 micron/minute, while 0.01 to 0.1 micron/minute rates were characteristic with Union Carbide material. (Melt chamber volume: 15.5 cubic feet). The Australian material was porous compared to the Union Carbide chromium, and presumably yielded more adsorbed gas during leak rate evaluation.

Chemical analysis for oxygen and nitrogen content was obtained on two 4-1/2 pound trough melted bars, one of Australian, and one of Union Carbide chromium, to determine whether contamination is avoided in this melting practice. The results are shown in Table 2 along with analysis of the base materials. A slightly higher oxygen level was detected after trough melting the Union Carbide chromium, as compared to the starting material analysis. The result of 106 ppm, however, was considered reasonably close to the values obtained for analysis of the starting material, and to be within the concentration range likely in the 105 pound lot of electrolytic flake. The oxygen and nitrogen levels detected in the bar produced from Australian chromium were below that observed in the base material. Perhaps some of these elements are "slagged" to the surface during melting, and removed in subsequent pickling prior to chemical analysis. The dull surface appearance of melted Australian chromium suggests this possibility (refer to Figure 1). It was concluded from these results that oxygen or nitrogen pick-up does not occur in the trough melting procedure.

Electrodes for initial consumable arc melting were assembled by stacking trough melted bars together, and TIG welding. Filler for joints was provided by using flakes of the electrolytic materials. Alloy additions were either added to the trough melt charge, or sandwiched between bars of pure trough melted chromium. Electrodes for remelt were assembled from first melt ingots by TIG butt welding, using pure chromium for filler material. Nickel or carbon steel adapters were welded to both first and second melt electrodes to provide suitable coupling to the arc furnace stinger. A photograph of typical trough melted bars, and a first melt electrode is displayed in Figure 3. An electrode assembled from first melt ingots for remelting, is shown in Figure 4.

TABLE 2 - Level of Oxygen and Nitrogen in Trough Melted Chromium[1]

Element	Australian Chromium		Union Carbide Chromium	
	Flake[2]	Melt	Flake[2]	Melt
Oxygen	200-210	105	50-69	106
Nitrogen	7- 10	< 5	64-80	67

1. PPM by weight
2. Sampling details given in Table 1

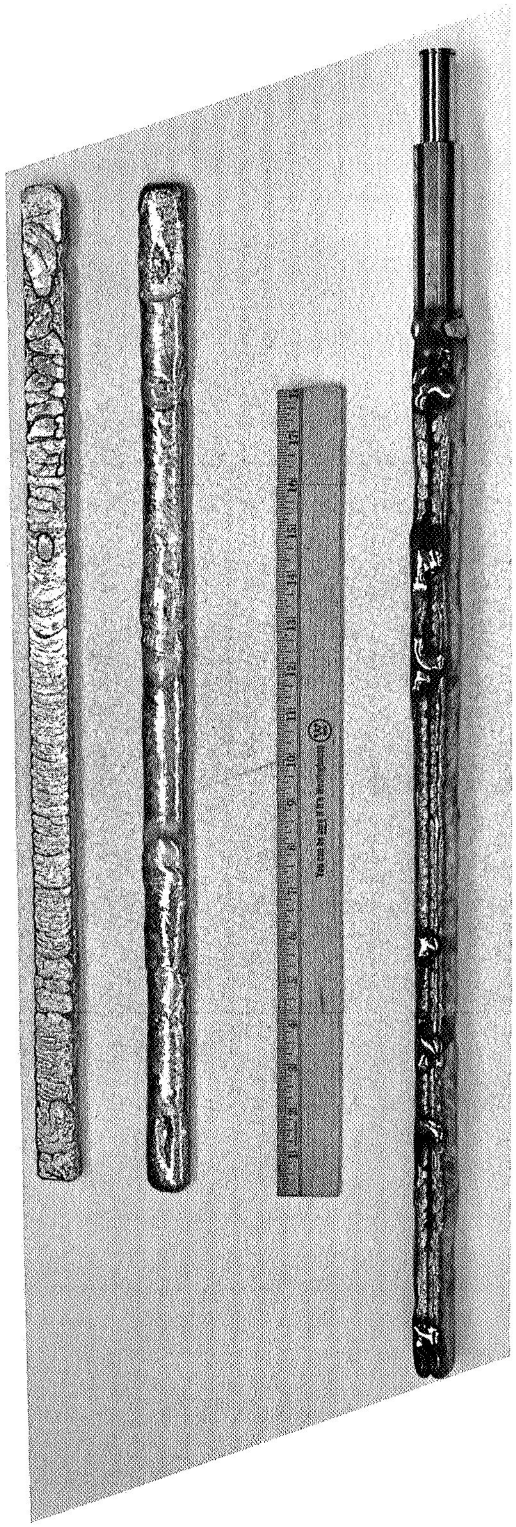


FIGURE 3 - Trough Melted Bars and an Assembled First Melt Electrode

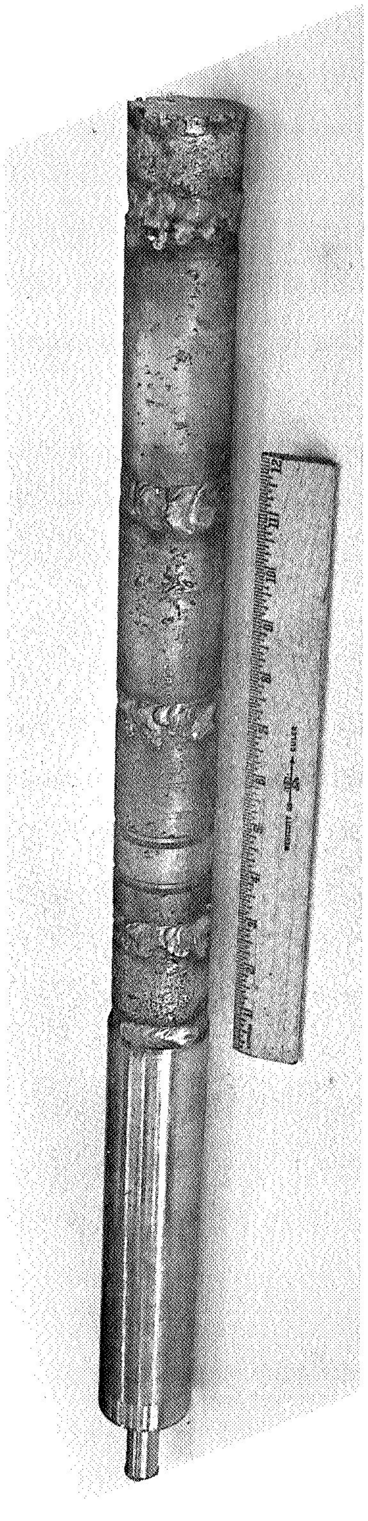


FIGURE 4 - Assembled Second Melt Electrode

Consumable Arc Melting

Consumable electrode melting was performed in a Westinghouse built unit of about 6000 amp stinger capacity, capable of melting with either AC or DC current. A range of crucible sizes in roughly 1/2 inch increments from 1.4 inch to 4 inch diameter can be used in the unit. Recent use of this furnace has been for casting tantalum, columbium and vanadium alloys for experimental programs similar to the type described herein(7,8,9).

The technique of chamber preparation used successfully to avoid contamination during melting Ta, Cb, and V alloys, were employed for melting chromium. Basically, this amounts to melting only if the chamber displays <0.2 micron/minute leak rate after evacuation to below 10^{-5} torr.

It was originally planned that double arc melting would be used to yield ingots for the program. This scheme involved producing 2 inch diameter castings weighing about 9 pounds using electrodes assembled from two trough melted bars, followed by assembly of two or three of these ingots into a remelt electrode and casting to 3 inch diameter.

It was decided that a simpler alloy would be melted to establish melting parameters and characteristics before one of the program compositions was attempted. To do this, an electrode of Cr-0.22Y was made up by sandwiching the alloy addition between two trough melted bars of Union Carbide chromium. A Cr-Y alloy was chosen to judge if yttrium loss would occur during melting, and/or whether presence of this element would induce arc instability.

Several attempts were made to melt the Cr-Y electrode under vacuum into a 2 inch diameter mold using AC power, but an arc could not be sustained for more than a few seconds. The electrode was, however, smoothly melted after introduction of 1/3 atmosphere high purity argon into the furnace. Melting was accomplished at 17-18 volts and 35 KW; these parameters yielding an excellent 5-1/2 inch long ingot. Because of this experience, all program alloy consumable arc melting was done in high purity argon. Both top and bottom of this ingot were chemically analyzed for yttrium and displayed 0.24% and 0.22%, respectively, indicating loss of the element would not occur.

The Cr-0.22Y ingot was also metallographically examined, then extruded to bar, and portions of this rolled to sheet and swaged to rod to gain chromium fabrication experience. Furthermore, bend and tensile DBTT were determined on the products in various heat treat conditions, to judge the adequacy of the specimens and annealing procedures intended for use on the program. Reaction of the alloy in air at 2400°F was also examined. Details of fabrication and evaluation are given in the Appendix.

Processing

Primary ingot fabrication was accomplished by both conventional and high velocity Dynapak extrusion to rod at 5:1 to 7:1 reduction. Induction heating in argon, and heating in a molybdenum wound resistance furnace purged with hydrogen, was used to achieve extrusion temperatures. Because the program ingots were cracked, those extruded were hermetically encased in either carbon steel or molybdenum. Cans of 1/4 inch wall thickness were used. End caps were tungsten arc welded to the cans inside an inert gas filled chamber. Electron beam welding of a plug into one end of the can assembly served to hermetically encase the billet.

Secondary fabrication to 3/8 inch diameter rod was achieved by swaging. The material was heated to swaging temperature under hydrogen in a molybdenum wound resistance furnace.

Property Evaluation

Mechanical Properties. Tensile and stress-rupture data were obtained using a specimen of 1 inch gauge length and 3/16 inch gauge diameter shown in Figure 5. Also displayed in this figure is the micro izod sample used for impact tests.

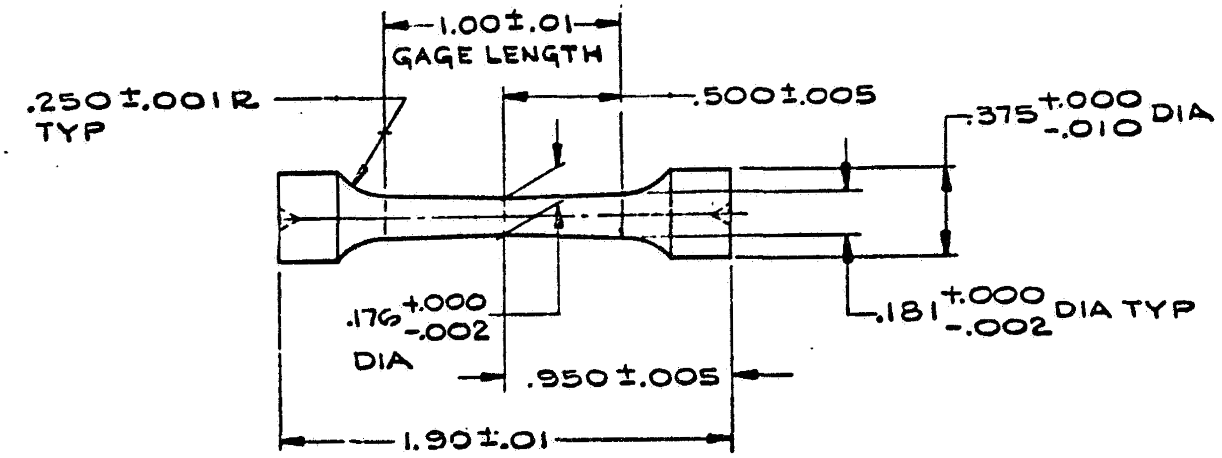
Elevated temperature tensile and stress-rupture tests were performed in vacuum of 10^{-5} torr (max.), measured at temperature. Impact and tensile data for DBTT determination were obtained in air. A strain rate of 0.03 inch/minute was used for tensile tests.

Heat Treatment. Samples were encapsulated in evacuated quartz tubing for heat treatment. A silicon carbide glo-bar resistance furnace was used for annealing at temperatures below 2300°F, with temperature determined by Pt-Pt/Rh thermocouple. Higher temperature annealing was accomplished in a cold wall tantalum resistance heated vacuum furnace. Optical pyrometer measurement on a black body emitter placed adjacent to the sample, corrected for sight port absorption, was used to determine temperature for anneals at and above 2300°F.

Metallography. Initial steps in sample preparation for microstructural inspection included grinding from 120 through 600 grit paper, and rough polishing on canvas cloth using one micron alumina abrasive. Final preparation was done by acid polishing on Frostmanns cloth, using jewlers rouge abrasive suspended in a weak chromic acid solution. Structure was developed by etching electrolytically in a 10% solution of hydrochloric acid in water.

Samples were prepared for macrostructural examination by grinding through 600 grit paper, and etching in a strong solution of hydrochloric and sulfuric acids in water.

Phase Identification. Precipitates were extracted from 10 gram samples by matrix dissolution in 300 ml of 10% bromine-methanol solution. Standard Debye X-ray diffraction patterns were obtained on extractions, using a 114 mm. Seimens camera, and nickel filtered



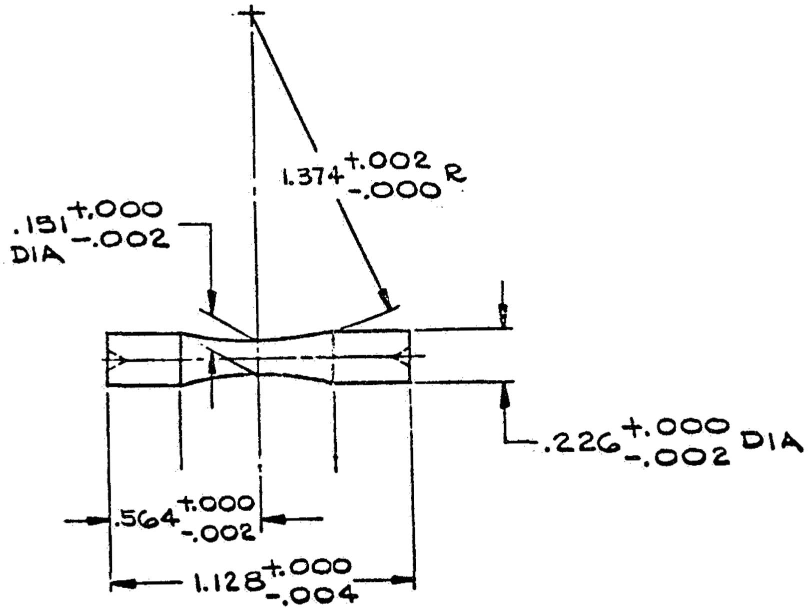


FIGURE 5 - Tensile and Impact Samples Used for Property Evaluations

copper radiation. Extracted phases were also redispersed on carbon replicas, and examined by electron microscopy and diffraction on a JEM 6A unit. Samples were prepared by drying drops of precipitate slurry, obtained by ultrasonic dispersion in amyl acetate, on glass slides, then transferring the residue to parlodian and carbon replicas using the standard two stage technique.

Oxidation Testing. A high temperature model Stanton Thermobalance was used to obtain air exposure data at 2400°F. This system consists basically of a vertically mounted platinum wound resistance furnace, and a weighing system. Sample weight change and temperature can be automatically recorded on the unit as a function of exposure time. Samples were held for exposure on a pad of platinum gauze placed in the bottom of alumina crucibles. Surface preparation was done by grinding from 120 through 600 grit paper, and solvent cleaning.

III. EXPERIMENTAL PROGRAM AND DISCUSSION

Consumable Arc Melting

Initial Melts. Initial program alloy melting involved study of the Cr-2Ta-0.1C-0.05Zr composition. First melt electrodes of this composition were assembled by stacking two alloyed trough melted bars. Five of these electrodes were produced, three using Union Carbide chromium as the base material, and two of Australian chromium base.

Each electrode was melted to 2 inch diameter ingot using AC current. Typical melting parameters were 18-25 volts, 35-45 KW, and 1.1 pound/minute melt rate. A photograph showing two of these ingots is displayed in Figure 6. Cracking was noted in the vicinity of the shrinkage pipe on each of the five ingots, some of which can be seen on those shown in Figure 6.

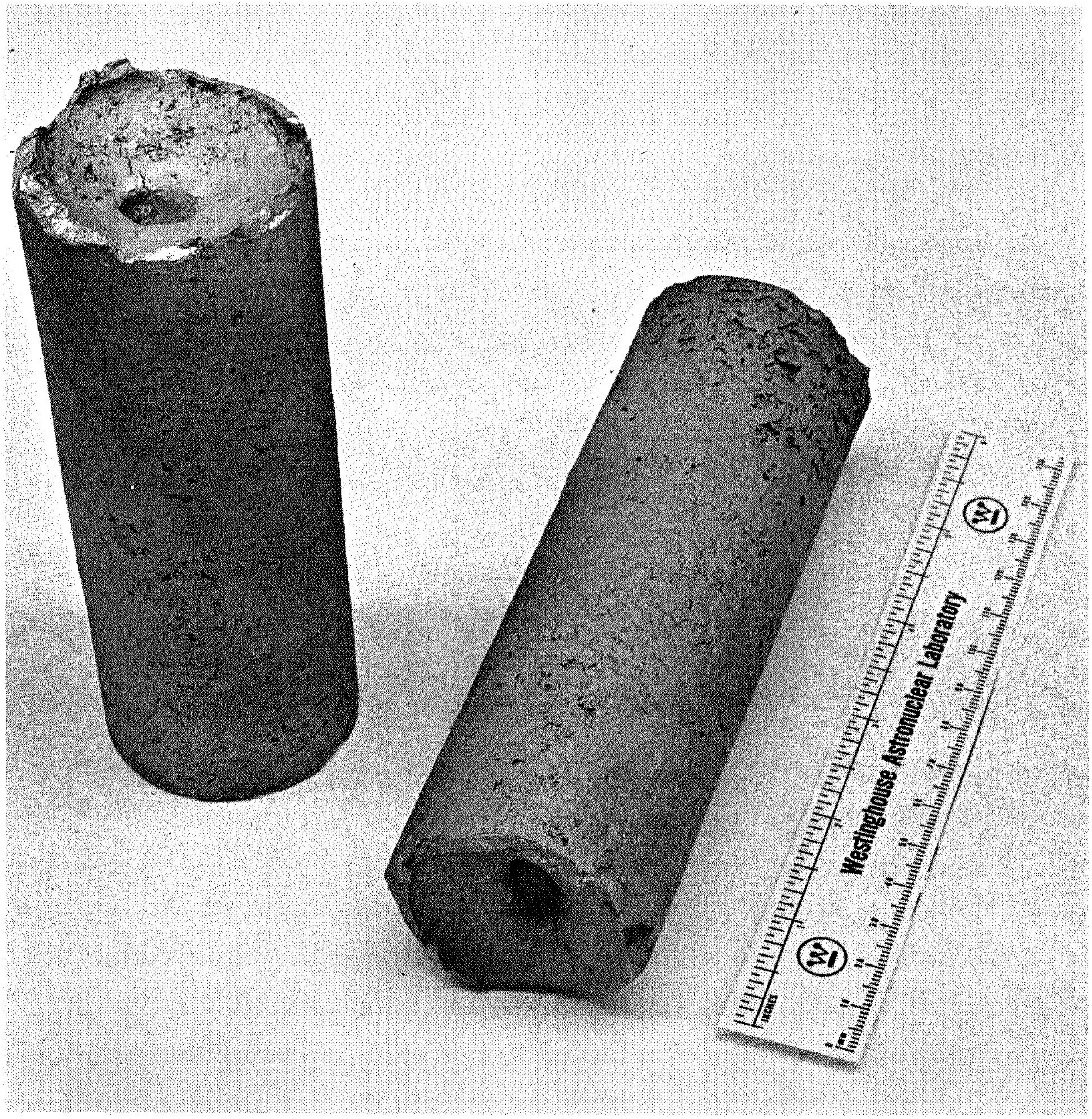


FIGURE 6 - Two First Melt Ingots

The ingots containing Union Carbide chromium, and those containing Australian chromium, were assembled into two separate second melt electrodes. Remelt of the alloy containing Australian chromium was made into a 2.6 inch diameter mold using AC current. Melting was done at a rate of 1.3 pounds/minute, employing 30 volts and 60 KW. This melt parameter data is given in Table 3 along with that for other ingots which will be discussed subsequently.

The ingot produced, Consumable Arc Melt-297*, contained a number of radial crack branches at both top and bottom locations. Removal of several 1/4 inch thick slices from the ingot ends failed to uncover sound material, indicating that cracking was not superficial, but instead probably continued throughout the entire casting length.

Remelt of the alloy containing Union Carbide chromium was made into a 3 inch diameter mold at 30 volts and 75 KW, using AC current. The ingot, CAM-298, was also found to be cracked badly at both top and bottom, indicating that differences in the two electrolytic chromium materials had no relationship to the condition.

The extent of cracking was such that conversion of these ingots to rod was not attempted. Both, however, were analyzed for oxygen and nitrogen content at top and bottom locations, to determine whether the melting practice employed may have resulted in contamination. These analytical results are listed in Table 4 where comparison is made to the levels observed in the base materials. Ingot CAM-298 was analyzed for major alloy content, and the results are also shown in the table. The alloy additions used, and the method for distributing them in the trough melts, was identical for both ingots CAM-297 and CAM-298. Because of this, analysis for major alloy content on only one ingot was considered a satisfactory check of alloying procedures. (Analytical results for ingots yet to be discussed are also given in Table 4.)

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*Referred to as CAM in latter text.

TABLE 3 - Melting Data

Heat No.	Composition (w/o)	Ingot Size		Steady State Melting Parameters		
		Dia. (in)	Length (in)	Volts	Kilowatts	Melt Rate (lb/min)
CAM-297	Cr-2Ta-0. 1C-0. 05Zr	2. 6	5. 5	30	60	1. 3
CAM-298	"	3. 0	5. 0	30	75	1. 5
CAM-425	"	3. 0	6. 0	25-30	75	2. 0
CAM-426	Cr-7Mo-1. 75Ta-0. 095C-0. 085Y	3. 0	7. 3	20-23	75	2. 2
CAM-447	Cr-2Ta-0. 1C-0. 05Zr	1. 4	4. 5	23-28	20	0. 6
CAM-448	"	2. 0	5. 5	30	--	1. 1
CAM-449	Cr-7Mo-1. 75Ta-0. 095C-0. 085Y	2. 0	5. 5	20	50	---
CAM-451	"	1. 4	4. 5	25	25	0. 6
CAM-452	Cr-2Ta-0. 1C-0. 05Zr	2. 3	4. 0	25-30	50	2. 0
CAM-459	"	1. 4	4. 5	30	30	0. 9

TABLE 4 - Analysis of Arc Melted Ingots

Heat No.[1]	Nominal Composition (w/o)	Sample[2] Location	Level of Element Detected[3]						
			O	N	C	Ta	Mo	Zr	Y
CAM-297	Cr-2Ta-0. 1C-0. 05Zr	T	230	<5					
		B	280	<5					
CAM-298	Cr-2Ta-0. 1C-0. 05Zr	T	100	55	0. 130	1. 90		0. 05	
		B	220	42	0. 120	2. 13		0. 04	
CAM-425	Cr-2Ta-0. 1C-0. 05Zr	T	77		0. 110	1. 50		0. 04	
		B		59	0. 092	1. 72		0. 04	
CAM-426	Cr-7Mo-1. 75Ta-0. 095C-0. 085Y	T	28	60	0. 120	1. 95	7. 13		0. 10
		B	56	62	0. 120	1. 71	7. 12		0. 08
CAM-447	Cr-2Ta-0. 1C-0. 05Zr	T	120	66	0. 101	2. 06		0. 04	
		B	95	47	0. 105	2. 06		0. 04	
CAM-448	Cr-2Ta-0. 1C-0. 05Zr	T	180	53	0. 105	2. 07		0. 04	
Union Carbide Cr		--	50-69	64-80					
Australian Cr		--	200-210	7-10					

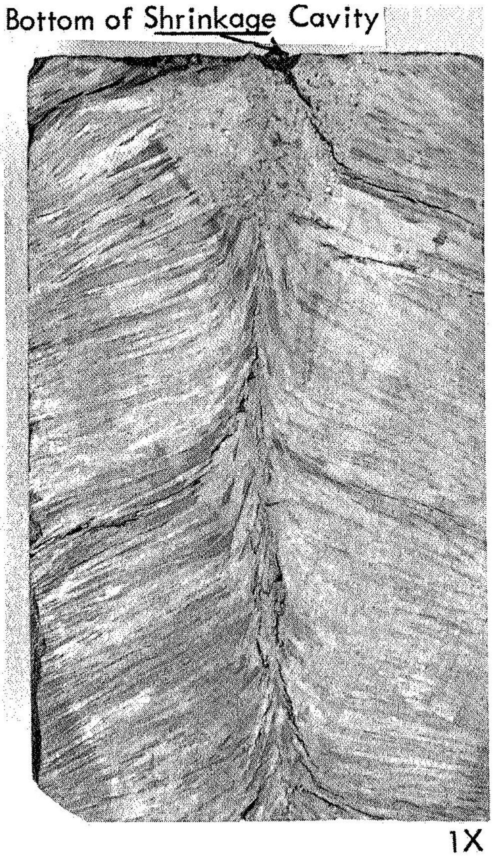
1. Australian chromium used as the base for CAM-297.
   Union Carbide chromium used in the other heats.
2. T = Ingot Top B = Ingot Bottom
3. Oxygen and Nitrogen: Analysis in PPM by weight.
   C,Ta,Mo,Zr,Y: Analysis in weight percent.

Oxygen and nitrogen contents observed on CAM-297 are reasonably consistent with the amounts in the Australian produced electrolytic base material. Nitrogen content on CAM-298 is consistent with that characteristic of the Union Carbide electrolytic base material, as is the oxygen level detected at the ingot top. A slightly high oxygen content, 220 ppm, was detected at the bottom of CAM-298 which may have resulted by contamination from the starting pad. Overall, the analytical results indicate that consumable arc melting neither significantly increased or decreased contamination levels, as compared that in the base materials.

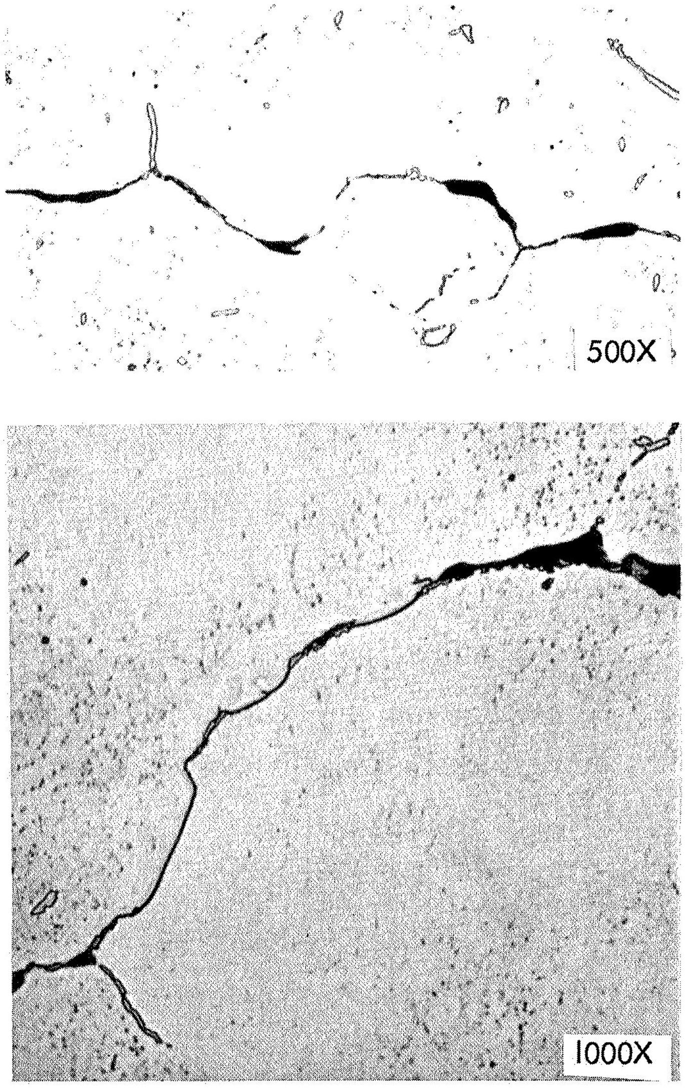
Major alloy element content of CAM-298 is reasonably close to the aimed for nominal Cr-2Ta-0.1C-0.05Zr composition. Furthermore, the distribution of these elements is uniform, indicating the procedure used to make the additions was adequate.

Examination was made of the cracked condition in ingot CAM-297. Photographs of the structure observed are displayed in Figure 7. Cracking was found to be generally associated with grain boundaries, with wide cracks developing along the radial columnar grain boundaries, and the centerline casting defect formed by impingement of these grains. Some crack branches observed in the macroetched axial ingot section shown in Figure 7 extend through to the ingot side, as well as its top and bottom. Actually, cracking was not obvious on the as-cast surface of either ingots CAM-297 or CAM-298. Ingot CAM-297 had been machined on the diameter about 1/4 inch below the as-cast size exposing these cracks, prior to sectioning axially.

Variation of Melting Technique. Shrinkage during solidification, and temperature gradients in a casting, are two conditions which may cause ingot cracking. Materials sensitive in this respect must, therefore, be cast using a method which avoids rapid temperature changes, and insures molten metal is continually fed into any solidification cavity. Because of this, five techniques promoting these conditions were introduced into the practice used to produce the next two consumable arc castings (CAM-425 and 426). These were:



Macroetched Axial Plane Section



Typical Cross Section Crack Microstructure

FIGURE 7 - Cracked Condition of Ingot CAM-297

1. Striking the melt at a power level too low for melting to begin, and holding at this level for a period of time to allow the starting pad to "heat-up". In this manner, the degree of thermal shock is reduced when power is increased to start melting, and molten alloy is deposited on the pad.
2. Use of a thick starting pad to allow longer retention of heat.
3. Lowering the mold stool immediately at the end of the melt to break cooling contact with the ingot bottom.
4. Withdrawal of the argon from the chamber at the end of the melt.
5. Slowly reducing power toward the end of the process to a level well below that used during steady state melting. In this manner, the molten pool is reduced slowly allowing time for metal to fill the shrinkage void. ("Hot topping").

Two second melt electrodes, one of each program composition, were cast to 3 inch diameter ingot employing these techniques. Melting was accomplished using direct rather than alternating current. It was thought that DC melting might produce a higher starting pad temperature during the initial melting period when the arc is sustained at a power too low for melting to occur. In AC practice, a power distribution of equal parts exists at the electrode, and the molten pool. With DC melting a power partition favoring the molten pool tends to develop, especially for the higher melting point metals.

Each second melt electrode consisted of three butt joined 2 inch diameter by 4-1/2 pound first melt ingots. The first melts were produced from consumable electrodes comprised of two trough melted bars of Union Carbide chromium, between which each alloy addition was sandwiched. To further assist production of sound castings, lengths of 2 inch diameter unalloyed arc cast Union Carbide chromium were welded to both ends of the second melt electrodes. The purpose of this was to form a composition less sensitive to cracking under thermal stress at both ends of the melt. This technique has been utilized successfully to eliminate cracking in some molybdenum base alloys(10). The electrode shown in Figure 4 is one which was used in this investigation.

The 3 inch diameter castings of both program compositions (CAM-425 and CAM-426) cracked badly, in spite of the introduction of the melting procedures outlined above. Of the two compositions, ingot cracking appeared most pronounced in the Cr-7Mo-1.75Ta-0.095C-0.085Y alloy (CAM-426). A photograph showing this ingot, and the cracked condition observed at top and bottom positions, is given in Figure 8. One observation made during this melt, which proved important in latter analysis of the cracking problem, was that the crack at the ingot top formed shortly after termination of melting, before the ingot had cooled below "red heat".

Chemical analysis was obtained for major metallic alloy additions, and interstitial element levels, at top and bottom locations of both CAM-425 and CAM-426. The results are displayed in Table 4. Major alloy element contents were reasonably close to the nominal composition levels, except for tantalum which was slightly high on CAM-426 top (1.95 w/o), and low at CAM-425 top and bottom (1.72 w/o and 1.52 w/o). A reason for the tantalum deviation from nominal could not be found. A weighing error, or mix-up of the individual ingot tantalum changes, may have occurred.

Oxygen and nitrogen levels detected on CAM-425 and 426 compared well with that characteristic of the Union Carbide electrolytic chromium, indicating contamination was avoided on both melts.

An examination of whether a pronounced reduction of ingot cooling rate might eliminate cracking was also made. In the experiment, an electrode of the Cr-2Ta-0.1C-0.05Zr composition, consisting of two alloyed trough melted bars of Union Carbide chromium, was melted into a mass of molybdenum of sufficient size to absorb the energy of casting and solidification, without itself melting. The molybdenum heat sink used was a 2-1/4 inch ID x 3 inch OD crucible, 1 inch thick at its bottom.

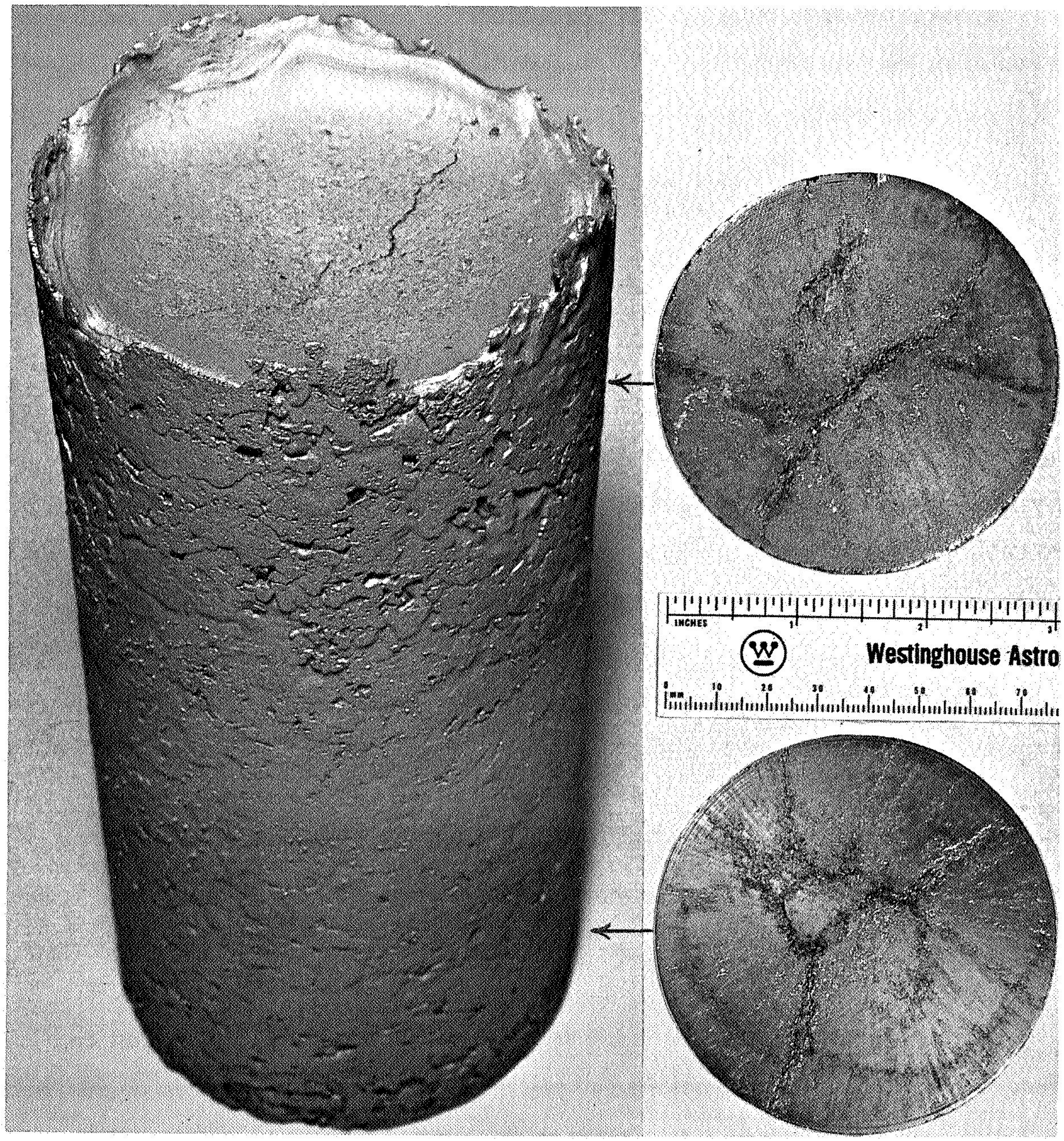


FIGURE 8 - Condition at Top and Bottom of CAM-426

The casting was made into the crucible which was held inside a 3 inch diameter arc furnace mold. A plasma spray coating of ZrO_2, 0.025 inch thick, was applied to this outside diameter of the crucible to reduce radial heat transfer to the arc furnace mold, which was water cooled as a safety precaution. The five melting procedures introduced into CAM-425 and CAM-426 were used on this melt.

Casting of this ingot, CAM-452, was smoothly accomplished into the molybdenum heat sink, and for at least a minute after its completion, molten liquid was noted to be shimmering in the crucible. By comparison, total solidification is observed immediately at the end of a melt of this size when arc cast into a conventional water cooled copper mold. The ability of the molybdenum crucible to slow the cooling rate was further noted by the time during which the ingot remained above "black heat", ten minutes, compared to about two minutes for similar size chromium heats cold mold consumable arc cast. In spite of this, gross cracking occurred in this casting, as can be seen in Figure 9, where the condition at the ingot top just below the solidification pipe is displayed.

Hardness of this ingot was noted to be higher than that observed on the similar composition heat CAM-425, and compared closer to CAM-426, the Cr-7Mo-1.75Ta-0.095C-0.085Y alloy. It follows, therefore, that some molybdenum pick-up occurred from the crucible. As previously noted in discussion of CAM-425 and CAM-426, ingot cracking is most pronounced in the alloy containing 7% molybdenum, thus, contamination from the heat sink may have contributed to cracking of CAM-452.

Effect of Ingot Size. A few ingots smaller than 3 inch diameter were cast and examined to determine whether cracking had any relationship to melt size. To do this, both 1.4 inch and 2 inch diameter consumable arc castings were prepared of each program composition, and compared visually with each other and the larger ingots previously discussed. Two alloyed trough melted bars were assembled to form electrodes for the 2 inch diameter ingots, while one sufficed for the 1.4 inch diameter melts. Union Carbide chromium was used for

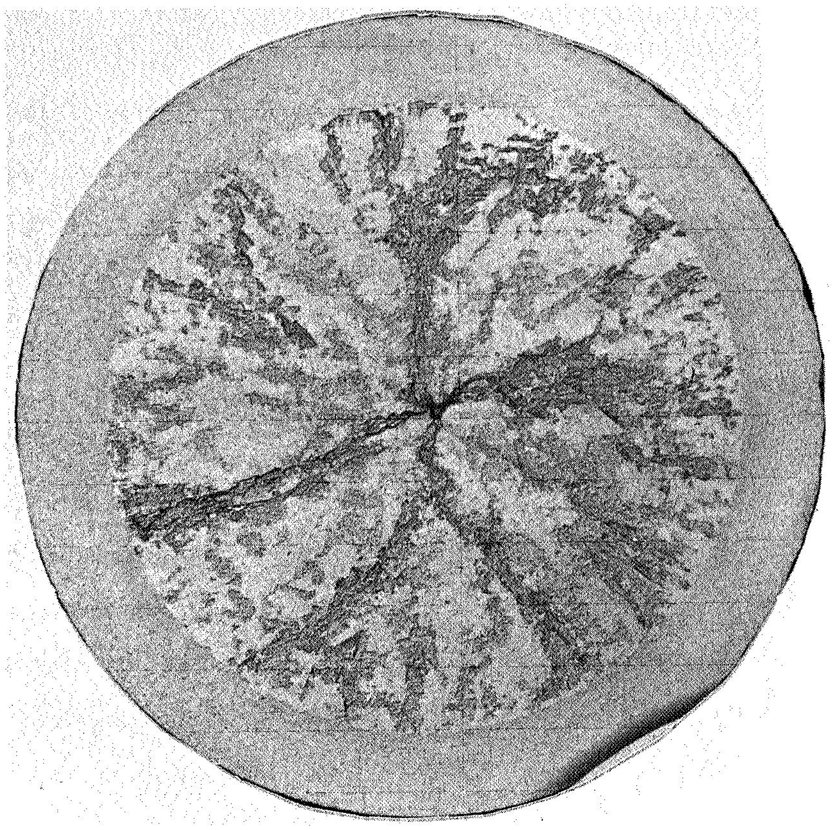


FIGURE 9 - Condition at the Top of CAM-452

the base in the ingots. Melting was done employing those procedures introduced into the practice used for CAM-425 and 426.

Each of these ingots (CAM-447, 448, 449, and 451) cracked severely, and no relationship between the degree of cracking and ingot size was observed. The cracked condition typically observed at the ends of ingots of each of these sizes, is shown in Figure 10.

Both ingots of the Cr-2Ta-0.1C-0.05Zr composition, CAM-447 and 448, were analyzed for major alloy element and oxygen and nitrogen levels, as a check on alloying and melt procedures. The data shown in Table 4, indicates good conformance to nominal composition, and little melt contamination.

Effect of Stirring. The association of ingot cracks with columnar grain boundaries, and the centerline casting defect, was noted on CAM-297 (Figure 7). Because of this, an examination was made of whether refinement of the as-cast structure might provide a more torturous path for cracks to follow, and in doing so possibly eliminate them. To do this, a 1.4 inch diameter ingot of the Cr-2Ta-0.1C-0.05Zr composition was produced employing magnetic melt stirring to induce grain refinement. The electrode used consisted of a single trough melted bar of Union Carbide chromium alloyed to this composition. Consumable melting was accomplished with direct current. The five melting procedures introduced into practice for CAM-425 and 426 to minimize the effects of thermal shock and shrinkage, were not employed on this melt.

A 1000 coil solenoid wrap of 14 gauge insulated copper wire placed around the mold and excited with 7 amps DC reversed in polarity every 5 seconds, was used to induce stirring of the molten pool during casting. This procedure developed a maximum magnetic field of 200 gauss reversed in direction every 5 seconds, as measured on the mold axis.

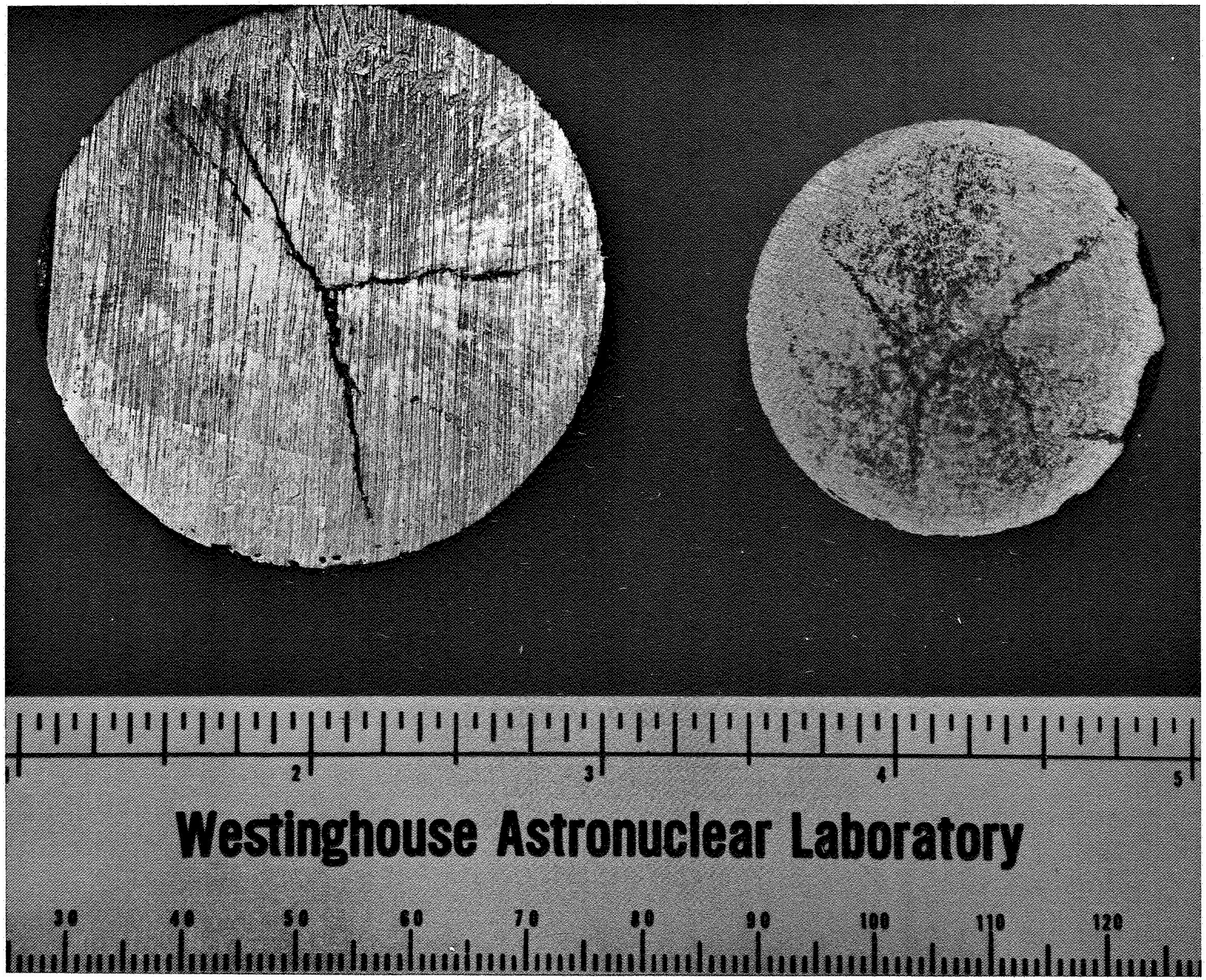


FIGURE 10 - Condition at the Ends of 1.4" and 2" Diameter Ingots

The ingot cast under these conditions, CAM-459, was sectioned on an axial plane, and prepared metallographically for macro-examination. A photograph showing the structure of this section is given in Figure 11. Grain refinement was certainly induced at the core of the casting, eliminating the centerline defect and associated cracking. Grain boundary cracking did occur, however, both in the fine central structure and the columnar grain case, as can be seen in Figure 11.

Analysis of Ingot Cracking

Background. Consumable electrode arc melting has been used on other studies for consolidation of chromium and chromium-base alloys(4,11-13). Generally success, as measured by the melting of sound ingot, has been achieved only for relatively pure chromium, or chromium scavenged with yttrium. For example, Allen et al(11) produced 4 pound consumable arc melted heats of binary chromium alloys containing up to 150 ppm carbon or nitrogen, or 500 ppm oxygen, without encountering the problem of ingot cracking. A total of thirty-five sound consumable arc cast 3 inch and 4 inch diameter ingots of unalloyed chromium and Cr-1Y, were produced by Fox(4). Although not discussed, ten sound 2 inch diameter by 4-1/2 pound ingots of unalloyed Union Carbide chromium were produced by consumable arc melting, for use as melt pads and electrode caps, on the program discussed herein. Furthermore, the Cr-0.22Y casting produced to establish melting parameters, was not cracked.

On the other hand, evidence of cracking was detected radiographically by Foster et al(12) on 3.5 inch diameter consumable melts of Cr-0.7Y, and it appeared to follow the solidification pattern characteristic of arc cast ingot, indicating an association with grain boundaries. Little success in producing sound consumable melted ingots of chromium alloys containing elements aimed at strengthening the base, was achieved by Maykuth and co-workers(13). Both electrolytic and high purity iodide chromium were investigated in this study as the base of binary alloys containing 1-2Cb, 7.5Mo, 1Y, 4-5Ta and 1Ti. Ingots of these compositions displayed cracks extending throughout their entire length. Reducing melt power and the

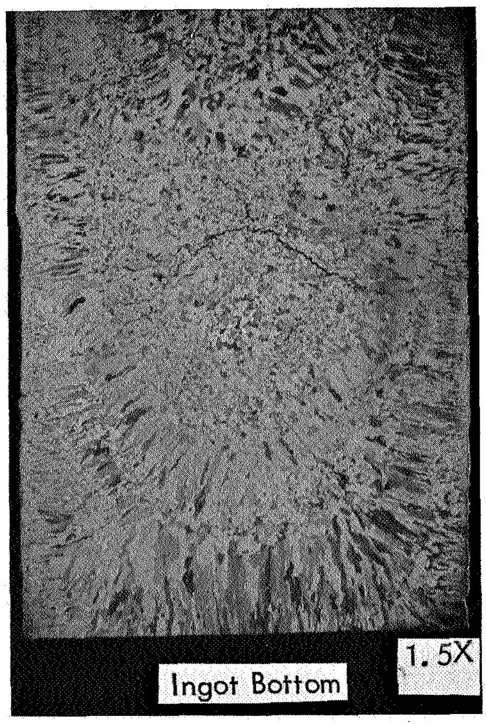


FIGURE 11 - Macroetched Structure of Axial Plane on CAM-459

mold cooling water flow during the latter stage of casting, and transferring the ingots while still hot to a furnace for 1 hour anneal at 2000°F, failed to eliminate cracking in the alloys. A conclusion from this work was that cold mold consumable electrode arc melting is not suitable for preparation of sound chromium alloy ingot containing greater than about 1% alloy addition. No indication was given, however, that any analysis of the cracking problem had been made.

The only literature reference found which indicated that sound ingot of a complex chromium-base alloy was produced by consumable arc melting, is the work of Northwood, Shaw and Smith(14) on Alloy "E"; Cr-2Ta-0.5Si-0.1Ti. In the study, 4 inch diameter by 10 inch long ingots of this alloy were consumable melted, successfully extruded to rod, and evaluated. No mention is made in the reported work of any direct observation of ingot cracking.

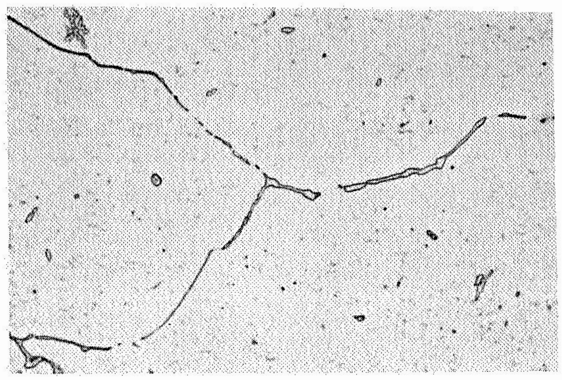
The ingots of Alloy "E" were produced at the Brightside Works of Jessop-Saville Ltd. in Sheffield, England. Contact was made with Jessop-Saville requesting experience information on consumable arc melting of chromium. In response, it was learned that Alloy "E" ingots required transferrence, within 7 to 10 minutes of the completion of casting, to a furnace at 1450 to 1800°F followed by furnace cooling, to prevent cracking(15). Although an explanation of how cracking would occur if the ingots were not annealed was not given, relief of thermal stress by brittle fracture after ductile-brittle transition during cooling, is the logical mode.

In subsequent work at Jessop-Saville intended to examine what property effect other strengthening additions made to the Cr-2Ta-0.5Si-0.1Ti composition would have, cracking of arc cast ingots was a problem. Cracking was particularly acute on highly alloyed compositions, and those containing tungsten or molybdenum; the investigators being "unable to obtain any sound material in alloys containing more than 2% tungsten or molybdenum". Mention was not made in the correspondence to specific compositions studied or any analysis of the cracking mechanism.

Present Study. The prior discussion has established that cracking of arc melted chromium alloy ingots has long been recognized. However, observations on the mode of cracking have been notably absent from the literature. High strength molybdenum alloys also exhibit ingot cracking, and in this case the cracking behavior has been studied in some detail(10,16). The cracks observed are often transgranular cleavage type, and cracking appears to be associated with the ductile-brittle transition behavior of the group VIa metals. It has been generally assumed that ingot cracking in chromium follows the same pattern, that is, cracking occurs at relatively low temperatures (in the DBTT range of the cast material) and is associated with thermal stresses encountered during cooling. Hence in this program melting techniques were selected which would minimize thermal gradients during melting and solidification. Initially, AC melting was chosen in preference to DC melting since the partition of power between electrode and pool is essentially equal, resulting in less pool superheat and consequently reduced thermal gradients. However, as described previously, the observed cracking was intergranular, and the cracks appeared to form during or immediately following solidification.

Metallographic examination made on both program alloys revealed a large amount of semi-continuous grain boundary phase, and finer matrix precipitate, in the as-cast structure. Photomicrographs of typical as-cast structures are displayed in Figure 12.

Bulk extraction of these precipitates were made on samples from CAM-425 and CAM-426, by matrix dissolution in a methanol-bromine solution. Analysis by x-ray diffraction uncovered the monocarbide of tantalum, and chromium carbide ($Cr_{23}C_6$), in both sample residues. The x-ray data are given in Table 5 where comparison is made to the ASTM standards.



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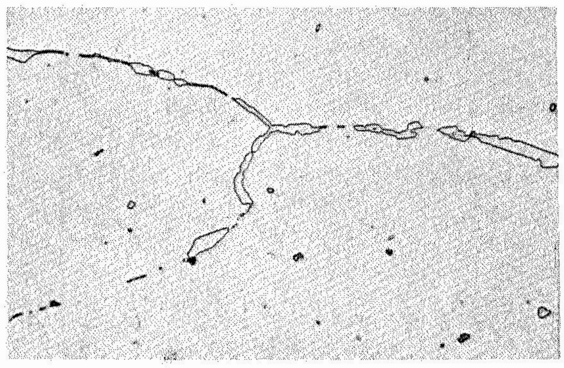

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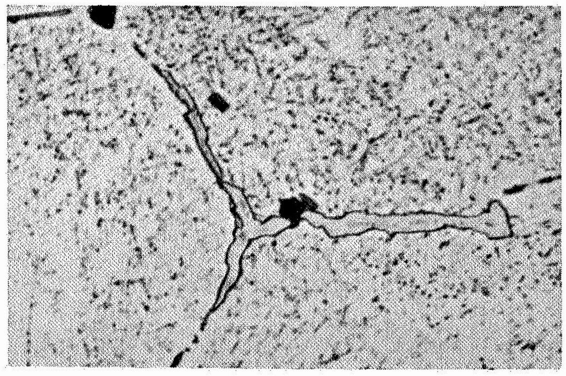


1000X

FIGURE 12 - Typical As-Cast Microstructure of the Program Alloys
Top: CAM-425
Middle & Bottom: CAM-426

TABLE 5 - Results of X-ray Diffraction Analysis of Extracted Precipitates

CAM-425 Extraction		CAM-426 Extraction		ASTM Standards			
				TaC		$Cr_{23}C_6$	
d	I	d	I	d	I	d	I
						3.20	W
						3.08	W
						2.66	W
2.57	S	2.55	S	2.57	S		
2.39	W	2.38	W			2.37	M
2.22	S	2.21	M	2.23	S		
2.15	W	2.18	W			2.17	M
2.05	W	2.05	M			2.05	S
1.88	W	1.88	W			1.88	M
		1.80	W			1.80	M
		1.77	W			1.78	M
						1.63	W
		1.61	W			1.61	M
1.57	M	1.56	M	1.575	S		
						1.49	W
						1.48	W
1.34	M	1.33	M	1.345	M		
						1.333	M
		1.29	W			1.293	M
1.28	W	1.28	W	1.285	M		
		1.27	W			1.256	S
1.22	W	1.23	W			1.231	S
1.18	W					1.191	S
		1.17	W			1.170	S
1.11	W	1.11	W	1.116	W		
				1.022	M		
				0.996	M		
				0.909	M		
				0.857	M		

Examination by electron microscopy uncovered two predominant precipitate morphologies in the residues extracted from both alloys. One of the morphologically distinguishable phases consisted of large irregular particles having many branches eminating from a central stem. Small thin platelets, which appeared generally square, were characteristic of the other major phase.

Electron diffraction patterns could not be obtained from any of the large precipitate particles since none displayed areas thin enough for penetration by the beam. A diffraction pattern was obtained, however, from a cluster of the thin platelet particles, the results of which compared well to the ASTM standard for the monocarbide of tantalum. This data is summarized in Table 6. Photographs of the two precipitate phases are displayed in Figure 13. It is deduced from identification of the platelet phase as TaC that the large irregular particles are the $Cr_{23}C_6$ phase. The appearance of each phase suggests that the TaC particles were formed by solid state reaction, and the $Cr_{23}C_6$ particles directly from the melt.

A summary of the phase identification results are given in Table 7 along with data reported by Clark(1) and by Ryan(2). The tantalum monocarbide compositions given in this table for the phase extracted from ingots CAM-425 and 426, was obtained by comparison of the diffraction data to that reported for the compound by Santoro(17). To simplify discussion this phase will be referred to as TaC.

Both Ryan and Clark noted only TaC in the program alloys after equilibration. Ryan, however, did find $Cr_{23}C_6$ and TaC present in the as-cast Cr-2Ta-0. 1C-0. 05Zr alloy. Furthermore, Ryan metallographically observed a massive grain boundary precipitate in this alloy as-cast, which disappeared upon annealing, indicating it to be the $Cr_{23}C_6$ phase.

TABLE 6 - Diffraction Data from Extracted Thin Platelet Phase

Extraction of Platelet Phase (d)	ASTM Standard for TaC (d)
2.59	2.57
2.22	2.23
1.57	1.575
1.34	1.345
--	1.285
1.11	1.116
1.01	1.022
0.99	0.996
0.90	0.909
0.85	0.857

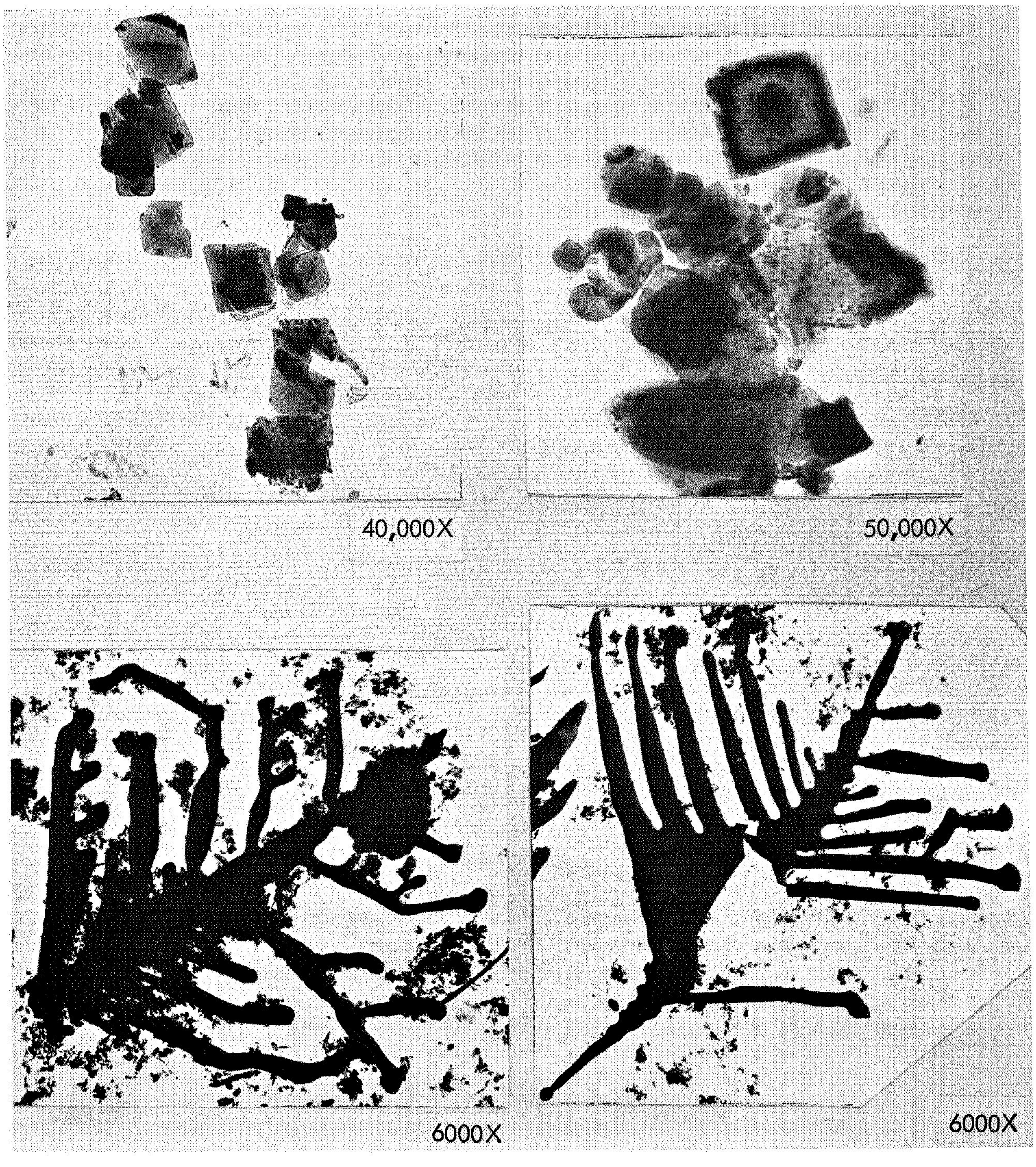


FIGURE 13 - Morphology Typical of Precipitate Phases in As-Cast Alloys. Top: TaC. Bottom: $Cr_{23}C_6$

TABLE 7 - Carbide Phases Identified in the Program Alloys

Alloy	Conditions	Phases Present	Heat or Ref.
Cr-7Mo-1.75Ta-.095C-.085Y	As Cast	$TaC_{.77}$, $Cr_{23}C_6$	CAM-426
Cr-7Mo-1.75Ta-.095C-.085Y	Worked and Annealed	$TaC_{.90}$	Reference 1
Cr-2Ta-.1C-.05Zr	As Cast	$TaC_{.81}$, $Cr_{23}C_6$	CAM-425
Cr-2Ta-.1C-.05Zr	As Cast	(TaC_x)[1], $Cr_{23}C_6$	Reference 2
Cr-2Ta-.1C-.05Zr	Cast and Annealed	(TaC_x)[1]	Reference 2

[1] Stoichiometry not reported.

Ryan suggests "sluggish scavenging" of carbon by tantalum as the reason for $Cr_{23}C_6$ formation in as-cast Cr-2Ta-0.1C-0.05Zr. Viewed on the basis of the data obtained on the program, it appears that only tantalum and carbon in solution within the solid formed as freezing proceeds, react to yield TaC. Since the solubility of carbon in the solid is low even at high temperatures, this results in failure to combine all the carbon present in the alloy as TaC. Furthermore, observation of the non-equilibrium $Cr_{23}C_6$ phase as a solidification precipitate, indicates formation as a consequence of carbon enrichment of interdendritic liquid occurring during freezing.

Carbon enrichment of interdendritic liquid would cause a wide separation of liquidus and solidus temperatures, and is considered a major factor contributing to the formation of intergranular casting cracks in the consumable electrode melted ingots. An approximate idea of the solidification range which would result, can be obtained by reference to the Cr-C binary system, reproduced in Figure 14(18). Freezing over a range of about 700°F is characteristic of a composition containing carbon at the 0.1 w/o level. It is felt that this wide solidification temperature range favors development of a "hot tearing" condition in the study alloys, in which ingot stress resulting from thermal gradients and shrinkage, is relieved by separation at a few remaining liquid grain boundaries.

The observation of cracking at the top of ingot CAM-426 prior to its cooling below "red heat", lends support to hot tearing as the failure mechanism. Furthermore, microscopic inspection of the surface of cracks formed in the ingots, revealed a contour suggestive of interdendritic separation. Photographs showing ingot crack surface features are displayed in Figure 15. The low magnification photograph, 20X, shown in the figure is typical of the macroscopic fracture appearance, and reveals the absence of signs of cleavage, or plastic deformation. At higher magnification the fracture detail observed reveals a surface containing closely spaced smooth rounded features. The rounded surface features are believed to be dendrite branches, their smooth appearance suggesting failure by separation through interdendritic liquid.

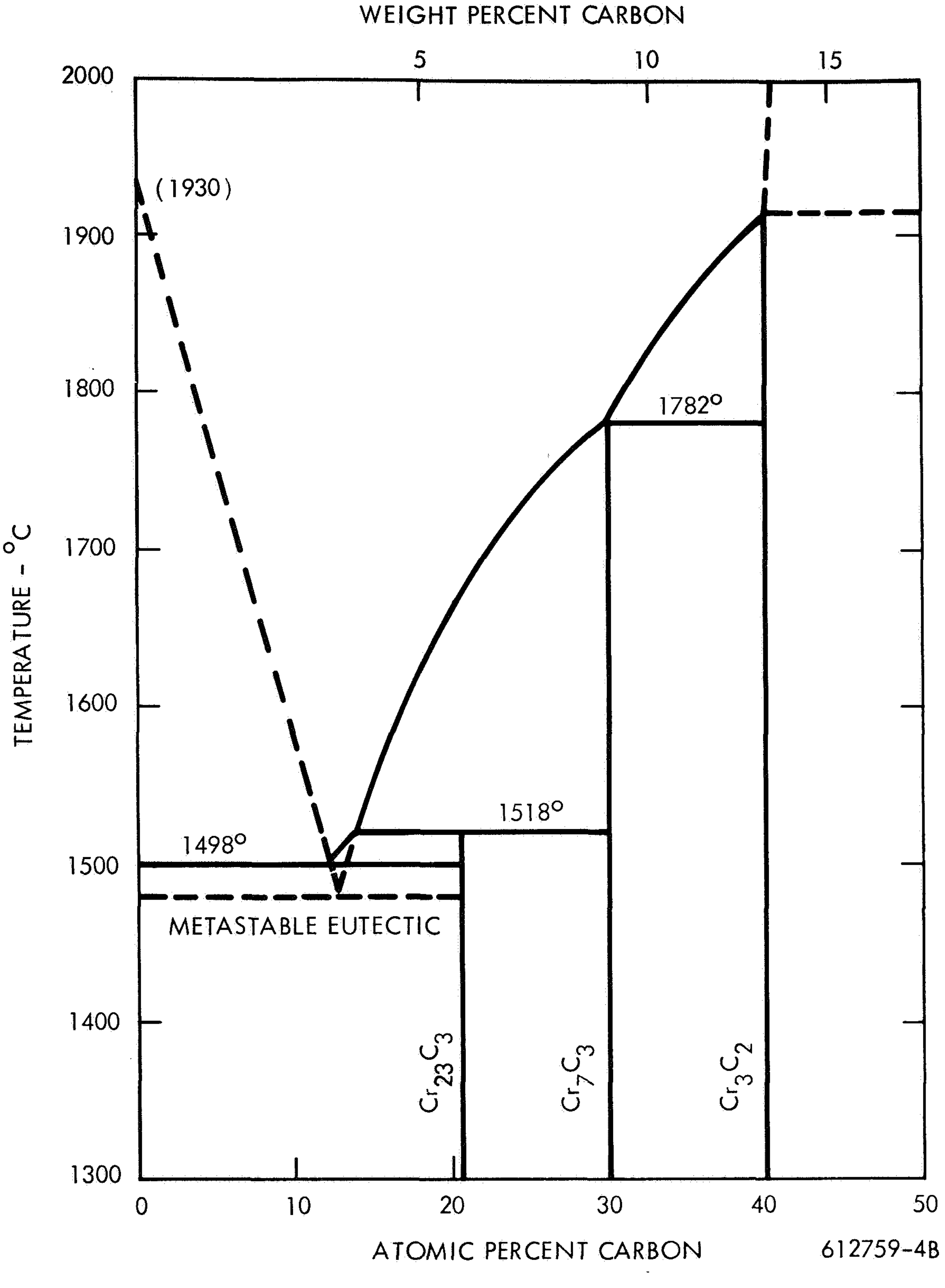
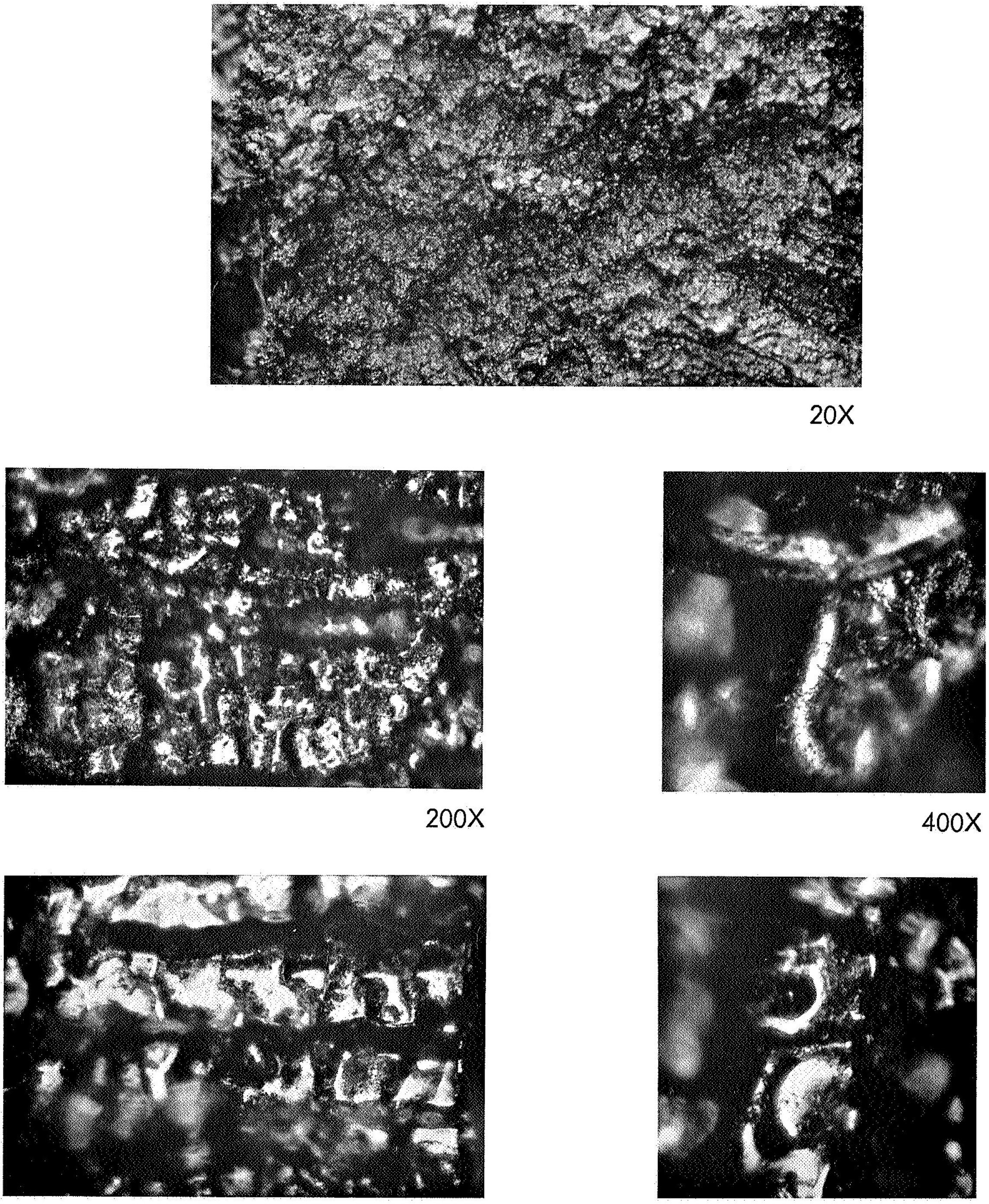


FIGURE 14 - The Cr-C Binary Phase Diagram (Ref. 18)



20X

200X

400X

200X

400X

FIGURE 15 – Ingot Crack Surface Fractography. Top: Macroscopic Fracture Appearance. Left: Typical Fracture Surface Detail. Right: Individual Smooth Fracture Surface Features.

Evaluation of Alloy Characteristics

Material Fabrication. One ingot, CAM-425, was hermetically encased in steel and processed through a high temperature autoclave cycle in an attempt to bond the cracked areas. A cycle during which the ingot was subjected to 28 ksi for 8 hours at 750°C, was used. Subsequent examination showed the process failed to effect any "healing" of the casting cracks. Because of this, all ingots processed to rod were canned to avoid any contamination through the crack network, and to assist bonding during the working operations. Conversion of ingot to test rod was attempted on CAM-425 (following autoclaving), 426, 447, 448 and 451. Initially CAM-425, 447, 448 and 451 were encased in steel, and Dynapak extruded to round bar at 2200°F. All, except CAM-451 which ruptured into several small pieces, were processed without extrusion failure. Of these ingots, only CAM-451 was of the Cr-7Mo-1.75Ta-0.095C-0.085Y composition. Because of this, heat CAM-426 of the same composition was sealed in molybdenum, and conventionally extruded at 2700°F. Success was achieved with this procedure in that extrusion failure did not occur.

The Cr-2Ta-0.1C-0.05Zr extrusions were fabricated to 3/8 inch rod by swaging at 2200°F to 2300°F without encountering fabrication problems. Difficulty was encountered, on the other hand, with fabrication of the molybdenum clad Cr-7Mo-1.75Ta-0.095C-0.085Y alloy. The expansion mismatch between the core and clad caused tensile failure of the chromium alloy when cooled from the swaging temperature. Fabrication of a major portion of this extrusion at 2300°F and 2600°F with molybdenum clad protection, resulted in a product containing numerous closely spaced transgranular cracks. Introduction of furnace cooling from 1800°F after swaging at 2400°F to 2600°F, avoided this problem, but only a small portion of the extrusion was processed in this manner.

All material produced and evaluated from these ingots received greater than 90% reduction in the overall process. Because of the cracked condition of the ingots, finish rod was carefully examined by etching and die penetrant inspection to insure sound materials was used for evaluation. Successful production of tensile and impact specimens from 3/8 inch rod to the dimensional requirements given in Figure 4, was also considered a test of material soundness.

Response to Heat Treatment. Hardness and microstructural change accompanying 1 hour heat treatment at 2000 to 2600°F, were examined on rod of the program alloys prepared from CAM-426 and CAM-447. This data is presented in Figure 15. Also included in Figure 15 is the as-fabricated hardness values of the rods of heats CAM-425 and CAM-448, and hardness data reported by Clark(1), and by Ryan(2).

The as-fabricated hardness level of the similar composition heats CAM-425, 447, and 448 were consistent, reflecting the close similarity of thermal-mechanical processing. Also, comparison of the hardness data with that given by Ryan and by Clark, show close agreement among this work.

Lack of any hardness or gross microstructural change with heat treatment of the Cr-7Mo-1.75Ta-0.095C-0.085Y alloy indicates it was hot worked, in the classical sense, during swaging at 2400°F to 2600°F. The lower swaging temperature, 2200°F to 2300°F, used to produce Cr-2Ta-0.1C-0.05Zr alloy rod, is reflected in the wrought as-fabricated structure. Recrystallization or change in hardness of this alloy did not occur in 1 hour for anneals up to 2400°F. One hour treatment at 2600°F did achieve recrystallization, resulting in a 30 point hardness drop, and a structure displaying elongated grains with their major axis along the direction of work.

Reaction in Air. Two samples, one of each alloy, were exposed in air at 2400°F for 24 hours, with weight gain continuously monitored during test. Both alloys changed similarly in weight, the Cr-2Ta-0.1C-0.05Zr composition gaining roughly 21 mg/cm^2, and the Cr-7Mo-1.75Ta-0.095C-0.085Y composition 24 mg/cm^2. Clark observed similar weight gain behavior for alloys of the Cr-7Mo-Y-(Refractory carbide) type, exposed under these conditions(1). The alloys also displayed identical parabolic behavior of weight gain with exposure time, 3.45 $mg/cm^2/hr^{1/2}$, after the first few hours of exposure. Plots of weight change against the square root of time for these tests are given in Figure 16.

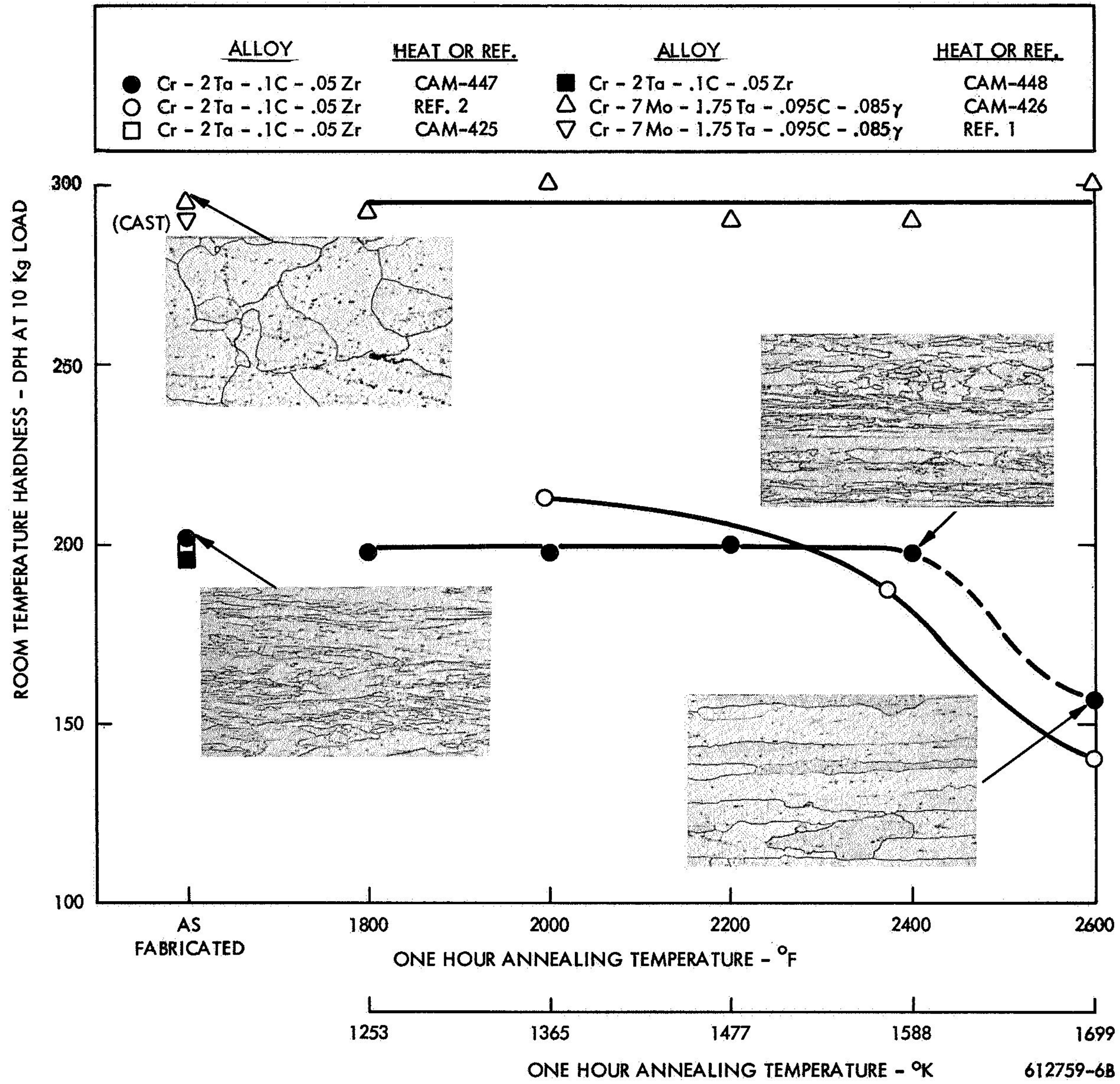


FIGURE 16 - Response of Hardness and Microstructure of the Program Alloys to Heat Treatment

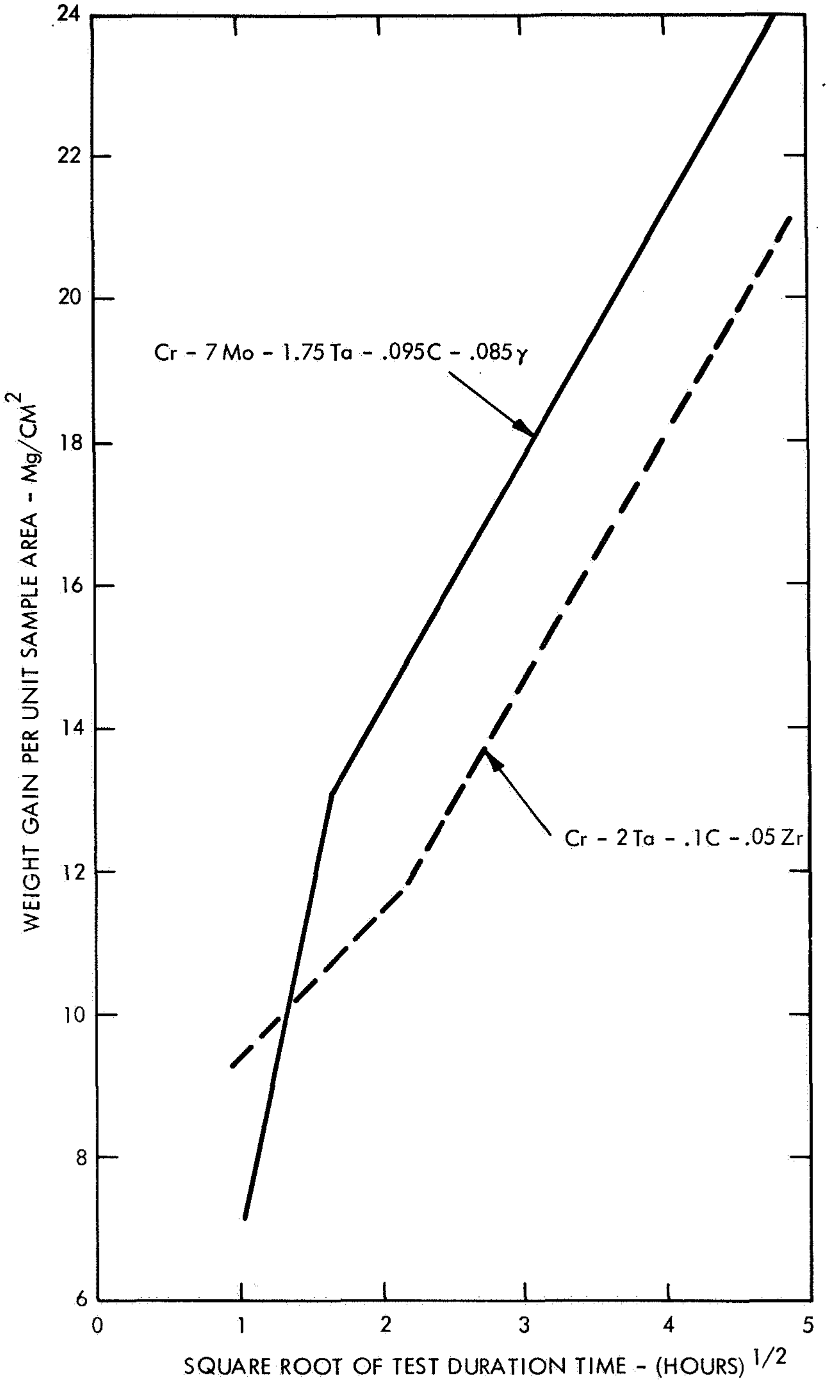


FIGURE 17 - Rate of Weight Increase of Program Alloys Exposed in Air at 2400°F

The green colored soft reaction product formed during sample exposure, was removed to determine surface recession. A recession of 0.0010 inch on the Cr-2Ta-0.1C-0.05Zr alloy, and 0.0015 inch of the other study alloy, were measured.

Metallographic examination of the Cr-2Ta-0.1C-0.05Zr alloy sample, revealed that an adherent and dense compound layer, about 10 mils in thickness, had developed under the soft outer surface product. Hardness values of from 1200 to 1800 KHN were measured on this layer. The level of hardness from beyond the hard layer to the sample center, corresponded close to that of unexposed material, measuring about 180 DPH. However, evidence of penetration of the hard layer phase beyond its interface with the alloy substrate, could be observed along grain boundary paths.

Examination of the Cr-7Mo-1.75Ta-0.095C-0.085Y sample uncovered formation of a massive continuous grain boundary phase through to the center of the 0.2 inch diameter specimen. A small amount of adherent, but porous surface compound, was also noted.

Photomicrographs of the oxidation samples are given in Figure 18. The reaction products noted in both samples metallographically appeared similar. Presumably, the phase is chromium nitride.

Mechanical Properties. Tensile properties at 1900°F, and stress rupture behavior at 2100°F were determined on both alloys. Impact and tensile DBTT was measured for the Cr-2Ta-0.1C-0.05Zr composition.

Evaluations were made for material after 1 hour heat treatment at 2000°F, and at 2400°F. As discussed under response to heat treatment, however, thermal-mechanical processing was such on the program alloys, that these anneals do not differ significantly in their effect on gross microstructure or hardness.

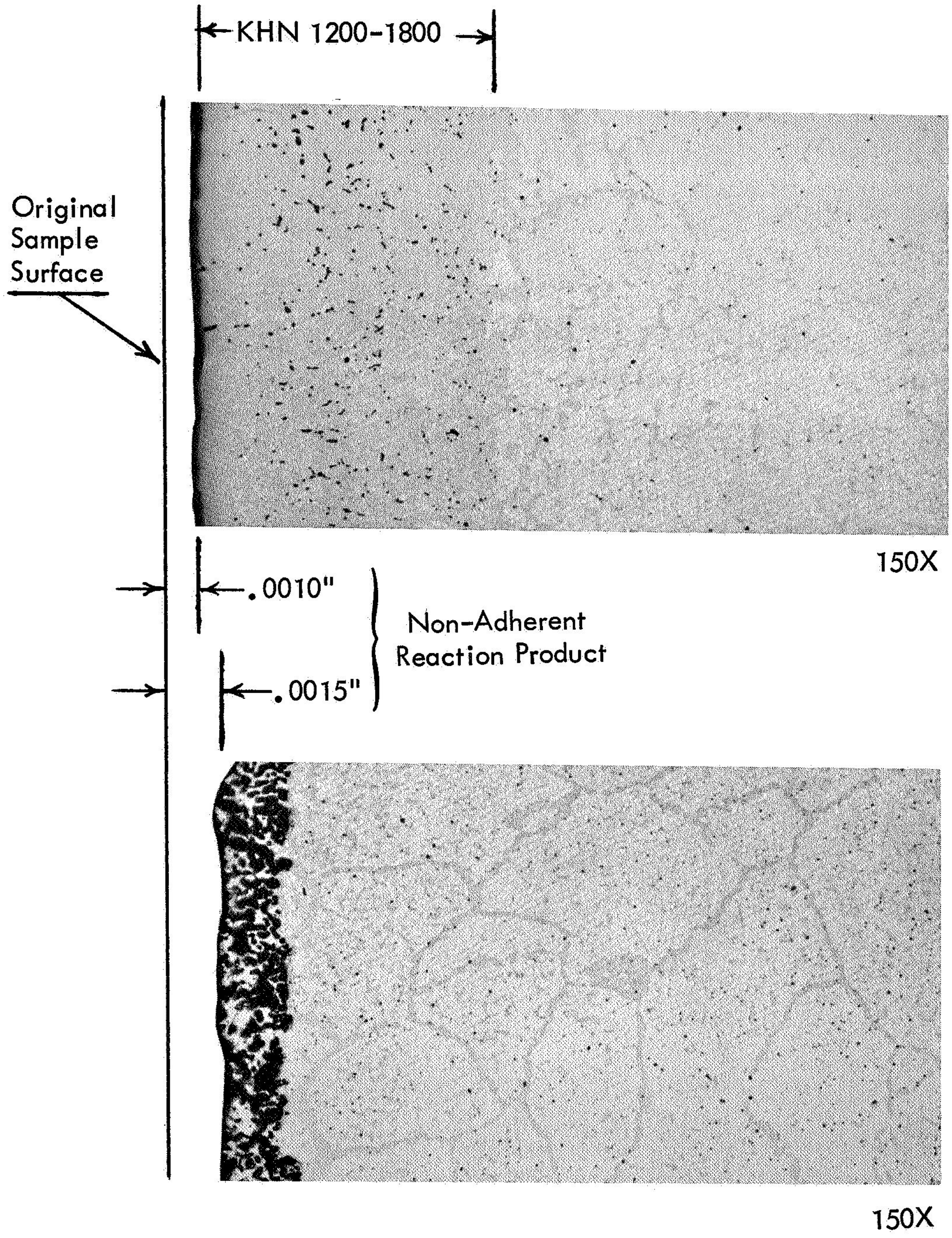


FIGURE 18 - Microstructural Characteristics of Program Alloys Exposed in Air for 24 Hours at 2400°F (Unetched). Top: Cr-2Ta-0.1C-0.05Zr Bottom: Cr-7Mo-1.75Ta-0.095C-0.085Y.

Stress rupture data obtained for both program alloys is shown in Table 8. Included in the table is data reported by Clark for the Cr-7Mo-1.75Ta-0.095C-0.085Y alloy(1). The results of rupture life obtained on this program, and reported by Clark for material in a comparable condition, display the scatter typical of short time stress rupture tests. Interpreted in an averaged sense, rupture life obtained for the Cr-7Mo-1.75Ta-0.095C-0.085Y composition on this program, is slightly lower than reported by Clark. Of the four similar tests, however, comparison of two can be made showing longer life for material produced in this program. Comparison of all the data for this alloy shows superior stress rupture behavior is displayed by material in the wrought condition.

Of the two study alloys the Cr-7Mo-1.75Ta-0.095C-0.085Y composition displayed superior stress rupture behavior, presumably reflecting the strengthening contribution of the molybdenum solid solution addition. By comparison, rupture life at about 12 ksi and 2100°F for the Cr-2Ta-0.1C-0.05Zr alloy, is similar to that for the composition containing 7% molybdenum tested at 15 ksi and 2100°F.

Stress dependency of rupture life at 2100°F is plotted for the Cr-2Ta-0.1C-0.05Zr alloy in Figure 19, along with data reported by Ryan(2) for material in similar condition tested at 1950°F. Test results at identical temperature and stress are not available for comparison on this alloy.

Pronounced loss of stress rupture strength occurs in the alloy with increase of test temperature from 1950 to 2100°F. This is not surprising, however, since 2100°F does represent nearly 70% of the absolute melting point of chromium, a level where conventional strengthening mechanisms begin to be overwhelmed by diffusional processes. For instance, although 2100°F is below the short time recrystallization temperature of the material, hardness measurement taken on the samples after testing indicated recrystallization had occurred. Perhaps major structural instability contributed to pronounced lowering of rupture strength of the alloy.

TABLE 8 - Stress Rupture Behavior of the Program Alloys at 2100° F (1421° K)

Heat or Ref.	Alloy	Condition[1]	Structure	Test Stress KSI	Test Stress $(N/M^2 \times 10^{-7})$	Life (Hr.)	Creep Rate %/Hr. (Min.)	Elong (%)	R.A. (%)
CAM-426	Cr-7Mo-1.75Ta-0.095C-0.085Y	1 hr/2000° F	Recrystallized	15	(10.34)	24.7	0.12	25.8	42.7
"	"	1 hr/2400° F	"	"	"	10.1	0.46	15.5	34.5
Ref. 1	"	"	"	"	"	49.9	0.033	29.2	-
"	"	"	"	"	"	17.6	-	32.8	-
"	"	1 hr/2000° F	Wrought	"	"	132.7	0.009	27.5	-
CAM-448	Cr-2Ta-.1C-0.05Zr	1 hr/2000° F	"	"	"	3.3	3.67	30.0	67.4
"	"	1 hr/2400° F	"	"	"	3.4	3.02	38.0	71.6
CAM-447	"	1 hr/2000° F	"	12	(8.27)	57.1	0.16	27.0	55.0
"	"	1 hr/2400° F	"	"	"	33.7	0.16	24.7	61.0

1. 2000° F: 1265° K
   2400° F: 1587° K

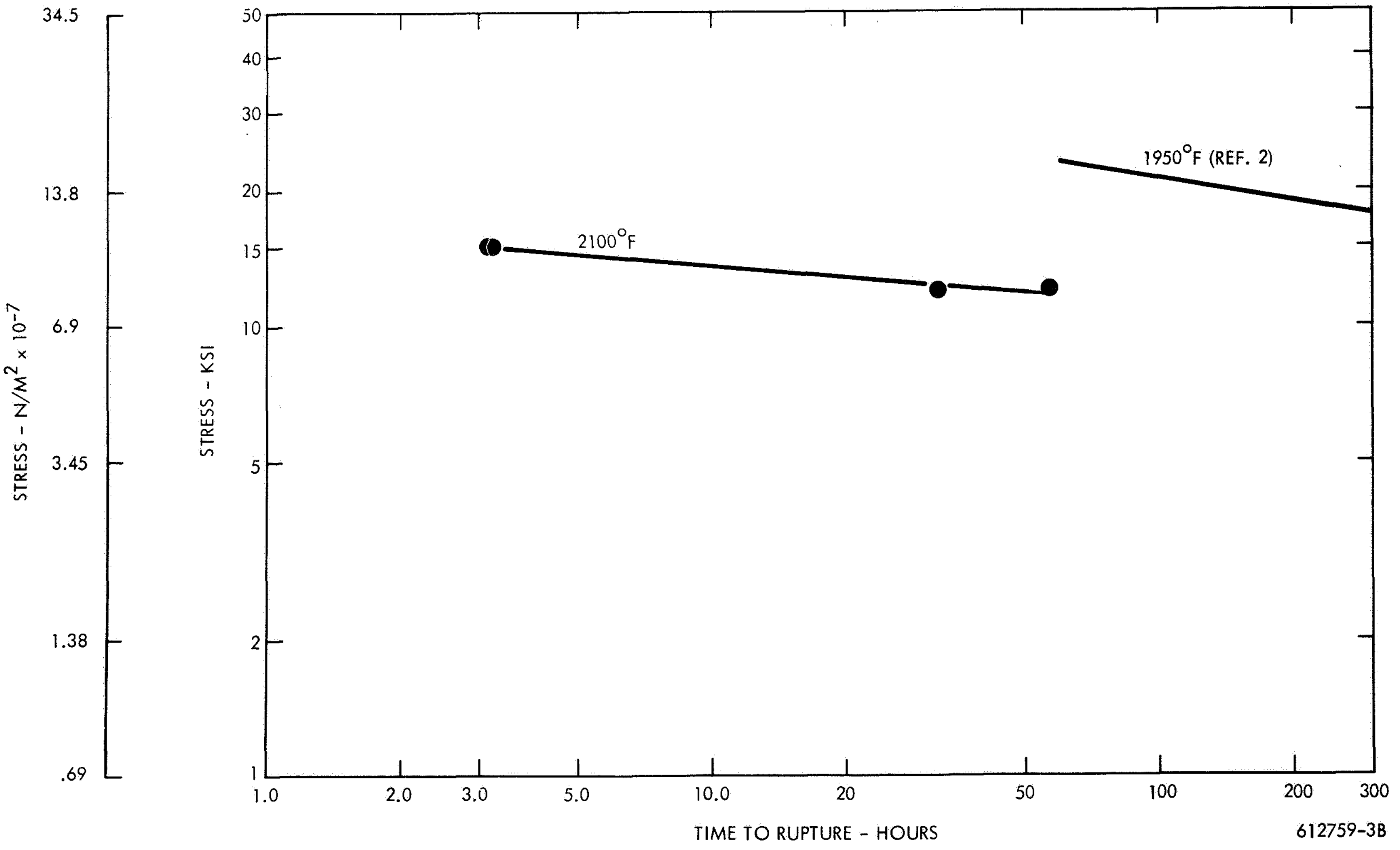


FIGURE 19 - Stress Dependency of Rupture Life for Cr-2Ta-0.1C-0.05Zr Alloy

Tensile properties at 1900°F are given for both alloys in Table 9. Data that Clark obtained for the Cr-7Mo-1.75Ta-0.095C-0.085Y alloy is also given in the table(1), and compares well with that obtained in this study. An ultimate tensile strength of 37 ksi for the Cr-2Ta-0.1C-0.05Zr alloy, compared to 60 ksi for the composition containing 7% molybdenum, shows the pronounced effect this addition also has on short time strength.

The results of tensile DBTT determination on the Cr-2Ta-0.1C-0.05Zr alloy are given in Table 10. A clear determination of tensile DBTT was obtained for the 1 hr/2000°F annealed condition, which using 5% minimum elongation as a criteria, occurs at about room temperature.

Only two tests were obtained for the 1 hr/2400°F annealed condition, one at room temperature, the other at 400°F. An elongation of 6.3% was achieved for the 400°F test, but the specimen break occurred at a shoulder fillet, indicating the possibility of premature failure due to presence of a flaw. The room temperature test for the 1 hr/2400°F condition was completely brittle, but by comparison 4.3% elongation and no reduction of area for the 1 hr/2000°F condition, does not represent markedly superior ductility. Perhaps the higher temperature anneal produces a higher tensile DBTT, but based on the limited data it is questionable to absolutely draw this conclusion.

The results of impact DBTT tests obtained on the Cr-2Ta-0.1C-0.05Zr alloy are given in Table 11 and plotted in Figure 20. Included in the figure is a photograph showing the tested samples. Transition from ductile-to-brittle behavior occurred somewhere in the temperature range 400°F to 800°F. At 400°F fracture occurred without any bending of the specimen, at 800°F impact caused the sample to bend without failure, and at two intermediate test temperatures partial bending and complete fracture both occurred. Energy absorbed followed the trend indicated by the shape of the tested samples, ranging from 4 inch-pounds at 400°F to 71 inch-pounds at 800°F. The value of 71 inch-pounds absorbed at 800°F is undoubtedly somewhat high, since because failure did not occur, the impacted portion of the specimen was not swept clear of the striking pendulum, and they rubbed together.

TABLE 9 - Tensile Behavior of the Program Alloys at 1900° F (1310° K)

Heat or Ref.	Alloy	Condition	Structure	Ultimate Strength		0.2% Yield Strength		Elong (%)	R.A. (%)
				KSI	($N/M^2 \times 10^{-7}$)	KSI	($N/M^2 \times 10^{-7}$)		
CAM-426	Cr-7Mo-1.75Ta-0.095C-0.085Y	1 hr/2000° F	Recrystallized	59.7	(41.0)	41.9	(28.8)	21.5	64
Ref. 1	"	1 hr/2000° F	Wrought	61.5	(42.3)	-	-	-	-
Ref. 1	"	1 hr/2400° F	Recrystalized	61.9	(42.6)	-	-	-	-
CAM-448	Cr-2Ta-.1C-0.05Zr	1 hr/2000° F	Wrought	37.4	(25.7)	36.4	(25.0)	23.0	87
CAM-447	"	1 hr/2000° F	Wrought	37.0	(25.5)	35.9	(25.4)	18.6	84

TABLE 10 - Low Temperature Tensile Behavior of Cr-2Ta-.1C-.05Zr Alloy

Condition[1]	Test Temp.		Ultimate Strength		0.2% Yield Strength		Elong.	R.A.
	(° F)	(° K)	KSI	($N/M^2 \times 10^{-7}$)	KSI	($N/M^2 \times 10^{-7}$)	(%)	(%)
1 hr/2000° F	400	(478)	72.7	(50.0)	52.2	(36.0)	24.4	65.8
"	200	(368)	82.4	(56.8)	53.5	(36.8)	15.9	25.5
"	R.T.	(296)	88.1	(60.8)	63.8	(43.8)	4.3	0
1 hr/2400° F	400	(478)	64.0	(44.0)	43.5	(30.0)	6.3[2]	0
"	R.T.	(296)	61.6	(42.4)	-	-	0	0

1. Heat: CAM-447
Structure: Wrought in both conditions

2. Fractured through specimen shoulder radius.

TABLE 11 - Impact Behavior of Cr-2Ta-.1C-.05Zr Alloy

Test Temperature		Energy Absorbed		Appearance of Tested Specimen
°F	(°K)	inch-lbs	(Joules)	
400	(478)	4	(0.45)	Fractured without bending
550	(560)	16	(1.81)	Fractured with some bending
650	(617)	26	(2.95)	Fractured with some bending
800	(699)	71	(8.01)	Bent without fracture

1. Condition: Annealed 1 hr/2000°F
   Structure: Wrought

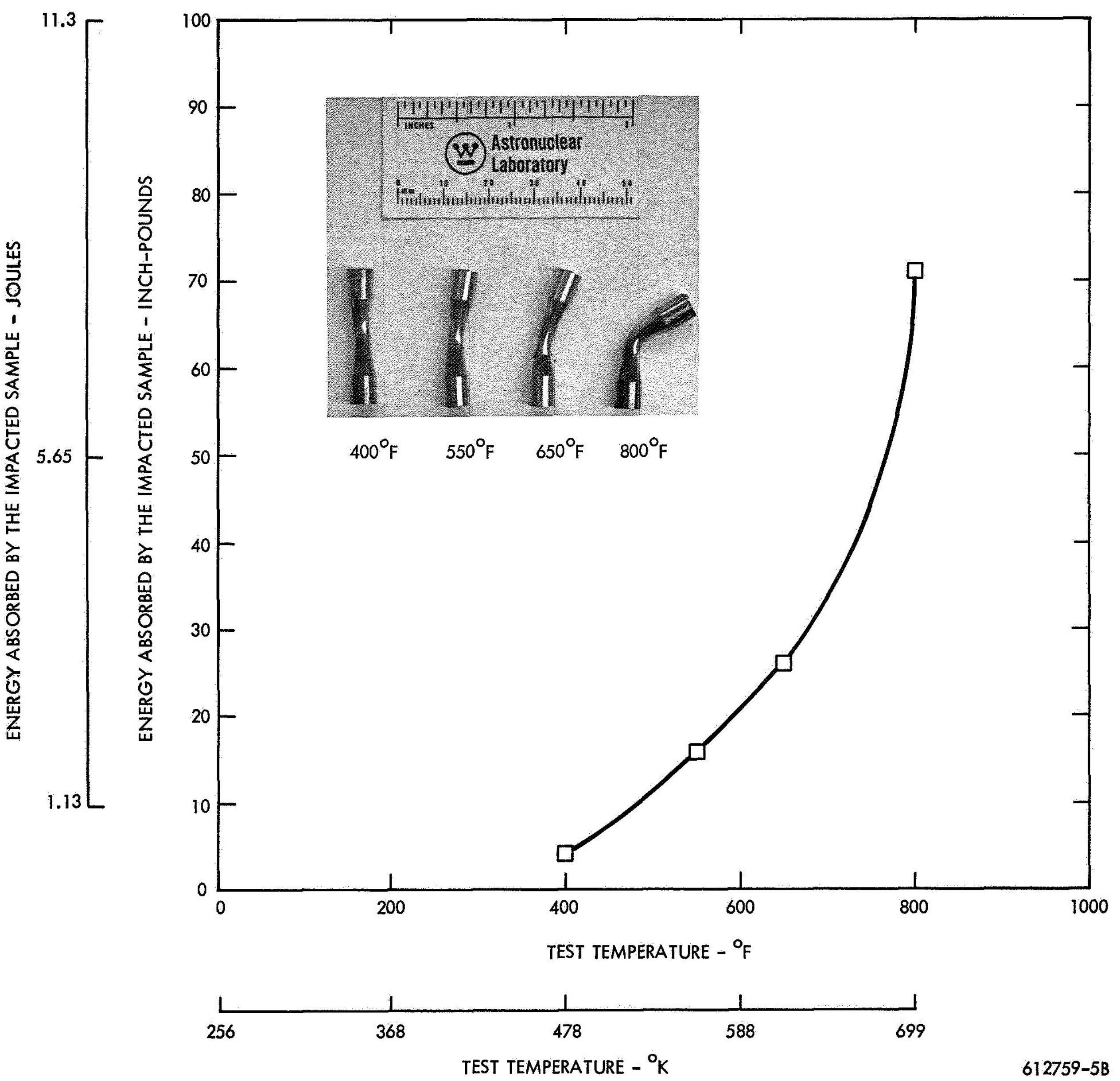


FIGURE 20 - Impact Toughness Behavior of the Cr-2Ta-0.1C-0.05Zr Alloy

IV. CONCLUSIONS

This study represents an effort to evaluate the consumable arc melting of two currently developed chromium-base alloys, and material fabricated from the ingots. Although major difficulty encountered with ingot cracking prevented accomplishing the original program scope, analysis of the failure mechanism was made, and some alloy properties were examined.

Conclusions arrived at from this work are:

1. Conventional cold mold consumable arc melting is unsuitable for producing ingots of the study compositions, because of their susceptibility to ingot cracking.

2. Ingot cracking appears to be related to the mechanism of solidification, which results in freezing over a large temperature range, causing a condition favorable to "hot tearing".

3. Sound bar stock can be produced from cracked ingots by extruding in evacuated cans, and swaging to final size.

4. The consumable arc melting practice employed on the program did not result in any significant changes in alloy contaminant levels, compared to that characteristic of the chromium used.

5. Because difficulty with ingot cracking limited the quantity of material produced from arc melted ingot, thorough comparison of final product properties with that for material produced from induction melted ingot, could not be made. Where comparison was made, however, properties were similar, indicating no benefit was derived from use of the cleaner melting technique.

While arc melting was not successful it is quite feasible to produce sound bar from cracked ingots providing care is taken to properly jacket the billets prior to extrusion. Electromagnetic stirring of the melt reduced cracking tendency, and more detailed studies of stirring, either by electromagnetic or ultrasonic techniques, coupled with methods to reduce the rate of cooling may offer potential to eliminate the cracking problem.

V. APPENDIX

Evaluation of Chromium-0.22Y Alloy

The consumable arc cast ingot of this composition was machined to 1-3/4 inch diameter, plasma spray coated with molybdenum, and high velocity Dynapak extruded at 1560°F through a 5/1 reduction to 0.84 inch diameter rod. A photograph of the extrusion is presented in Figure A-1. Portions of the extruded bar were swaged to 3/8 inch diameter rod, and rolled to 0.050 inch thick sheet. Secondary fabrication was accomplished at 2000°F.

Both tensile and bend DBTT evaluations were made on the Cr-0.22Y rod and sheet in the 1 hr/1800°F, 1 hr/2200°F, and 16 hrs/2400°F annealed conditions. Microstructures of the alloy in these conditions, and the mechanical property data are given in Figure A-2 and Tables A-1 and A-2. Transition in ductility occurred for the alloy between roughly room temperature and 400°F, and appears to be related to structure; the wrought and stress relieved condition exhibiting a lower DBTT than to the recrystallized condition.

Air exposure data obtained on a sample of as-swaged Cr-0.22Y rod held for 24 hours at 2400°F, is presented in Figure A-3. A weight gain of 19.4 mg/cm^2 resulted from the exposure, and weight change displayed a parabolic relationship with test time.

Surface recession of 0.0015 inch was measured on this sample by removing the soft non-adherent surface reaction product. Microstructural examination revealed that a hard adherent layer, 6 to 7 mils in thickness, had formed beneath the soft surface product. Some penetration of the hard reaction product along grain boundary paths, was also noted. Photomicrographs are given in Figure A-4.

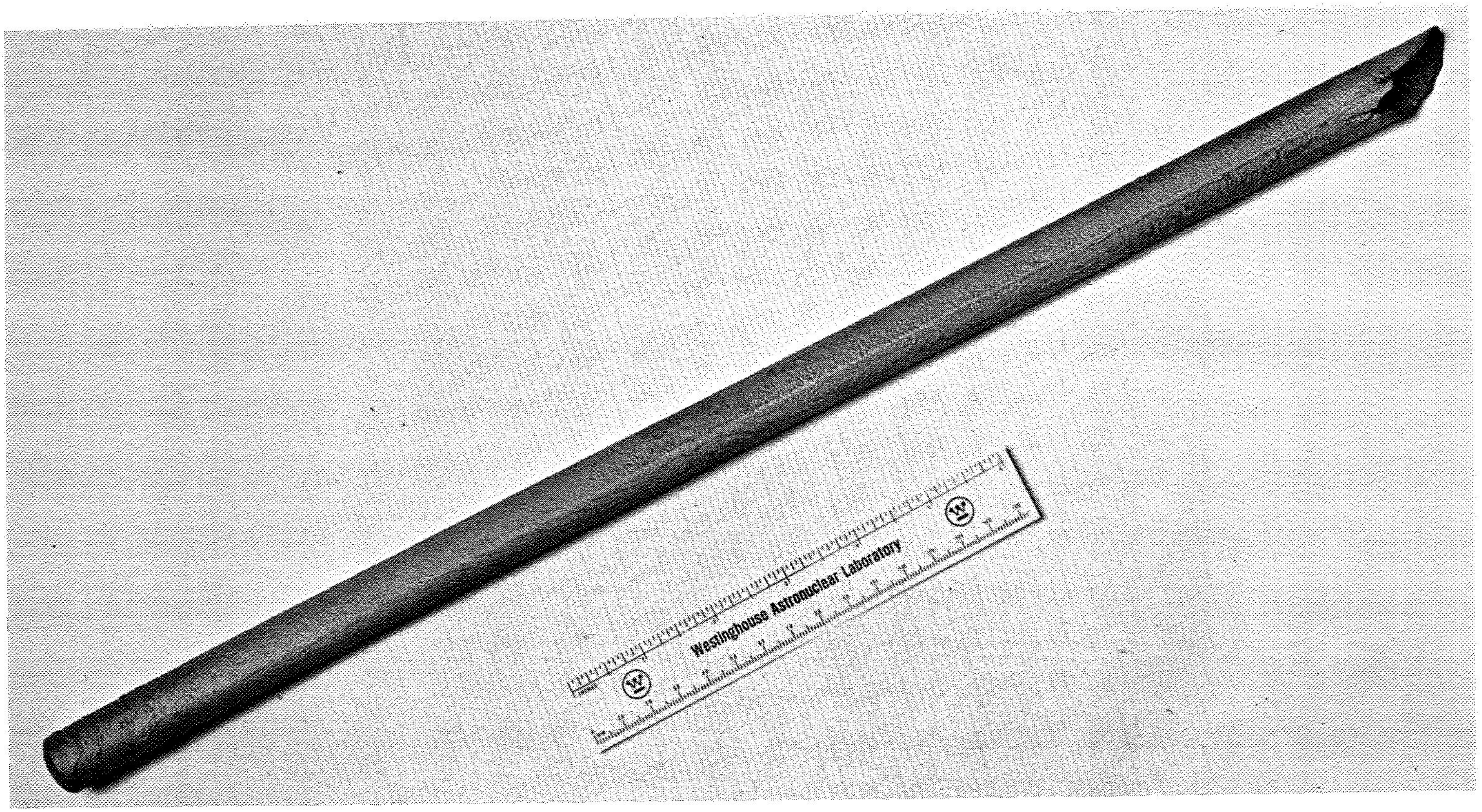


FIGURE A1 - As-Extruded Cr-0.22Y Bar

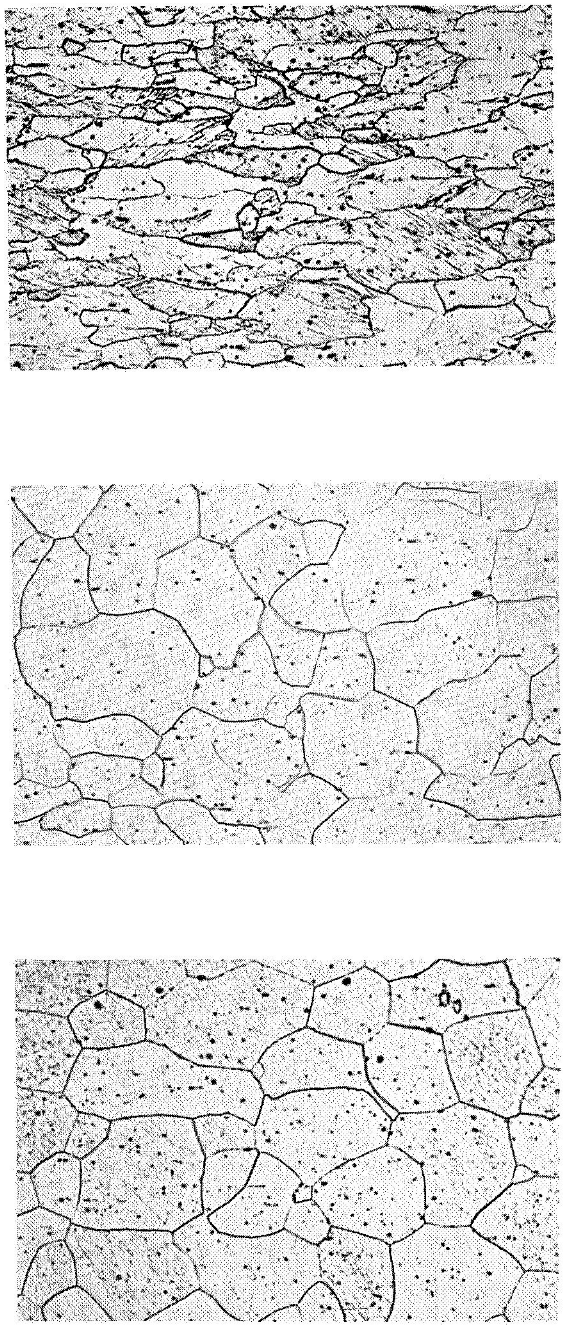


FIGURE A2 - Microstructures of Cr-0.22Y Alloy (100X)
Top: Annealed 1 Hr. at 1800°F
Middle: Annealed 1 Hr. at 2200°F
Bottom: Annealed 16 Hrs. at 2400°F

TABLE A1 - Bend Ductility Data for Cr-0.22Y[1]

Condition[2]	Test Temperature °F	(°K)	Bend Angle	Remarks
1 hr/1800°F	RT	(296)	0	Break
	200	(368)	95	Bend
	300	(423)	96	Bend
	500	(533)	90	Bend
1 hr/2200°F	RT	(296)	0	Break
	300	(423)	0	Break
	400	(478)	100	Bend
	500	(533)	100	Bend
16 hr/2400°F	200	(368)	30	Break
	300	(423)	90	Break
	400	(478)	102	Bend

1. Specimen: .040" x .5" x 2.5"
Punch: .160" radius
Support Span: .75"
Test Rate: 1"/min.

2. 1800°F: 1255°K
2200°F: 1478°K
2400°F: 1587°K

TABLE A2 - Tensile Data for Cr-0.22Y

Condition[1]	Test Temp.		Ultimate Strength		0.2% Yield Strength		Elong.	R.A.
	(°F)	(°K)	KSI	(N/M^2 x 10^{-7})	KSI	(N/M^2 x 10^{-7})	(%)	(%)
1 hr/1800°F	0	(255)	91.1	(62.9)	---	---	0	0
"	R.T.	(296)	61.7	(42.5)	44.9 61.7[2]	(30.9) (42.5)	47.2	52.0
1 hr/2200°F	-100	(200)	59.7	(41.1)	---	---	0	0
"	0	(256)	52.8	(36.4)	47.3	(32.6)	1.0	0.9
"	25	(269)	58.2	(40.2)	37.9	(26.1)	12.1	11.0
"	R.T.	(296)	49.4	(34.0)	25.9	(17.8)	31.6	24.6
16 hrs/2400°F	0	(256)	45.1	(30.0)	---	---	0	0
"	R.T.	(296)	31.2	(21.5)	24.2	(16.7)	0.6	0
"	200	(368)	42.7	(61.9)	11.8	(8.2)	19.9	17.3

1. 1800°F: 1255°K
   2200°F: 1478°K
   2400°F: 1587°K

2. Upper and lower yield points.

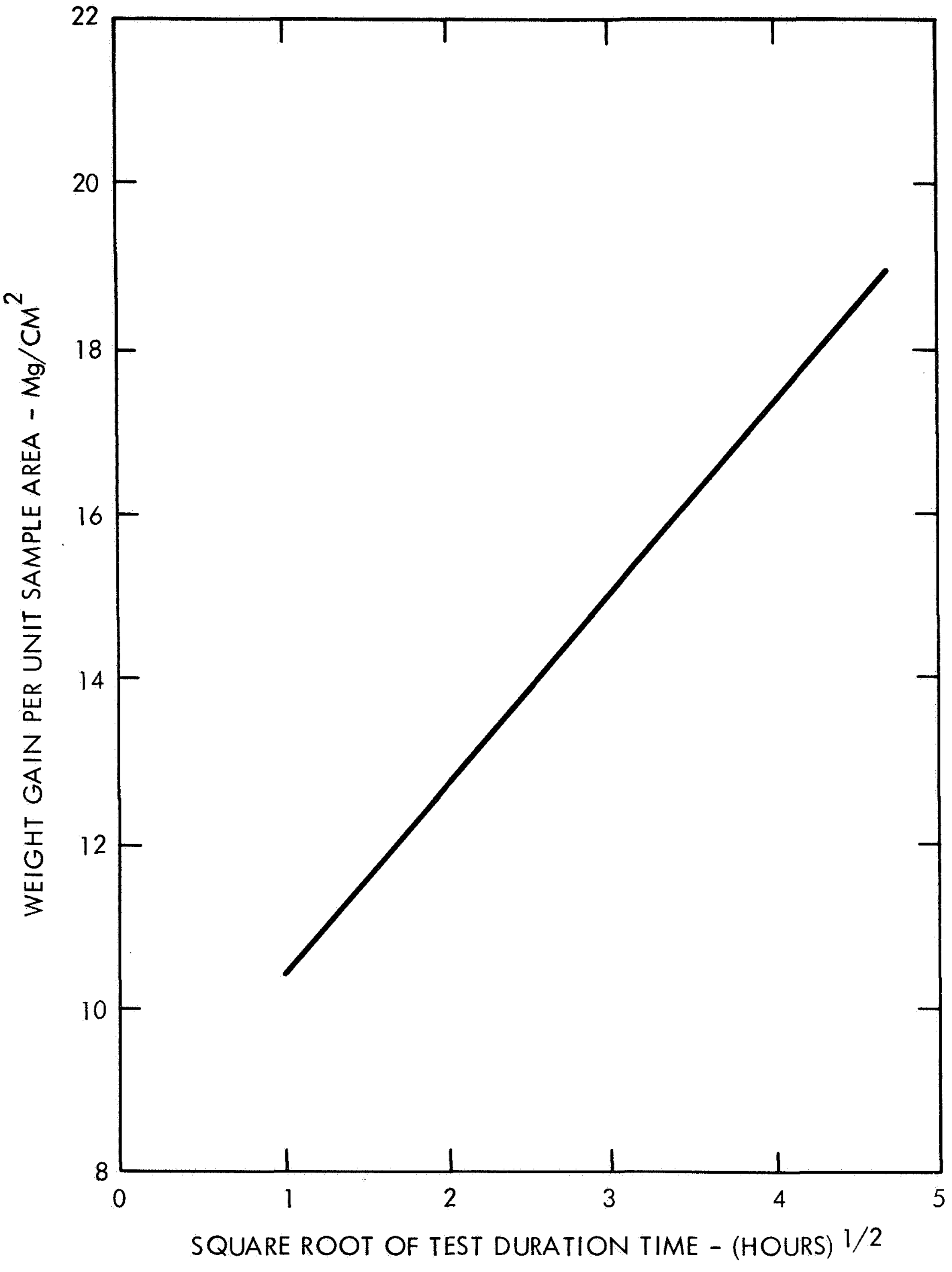


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FIGURE A3 - Rate of Weight Increase of Cr-0.22Y Alloy Exposed in Air at 2400°F

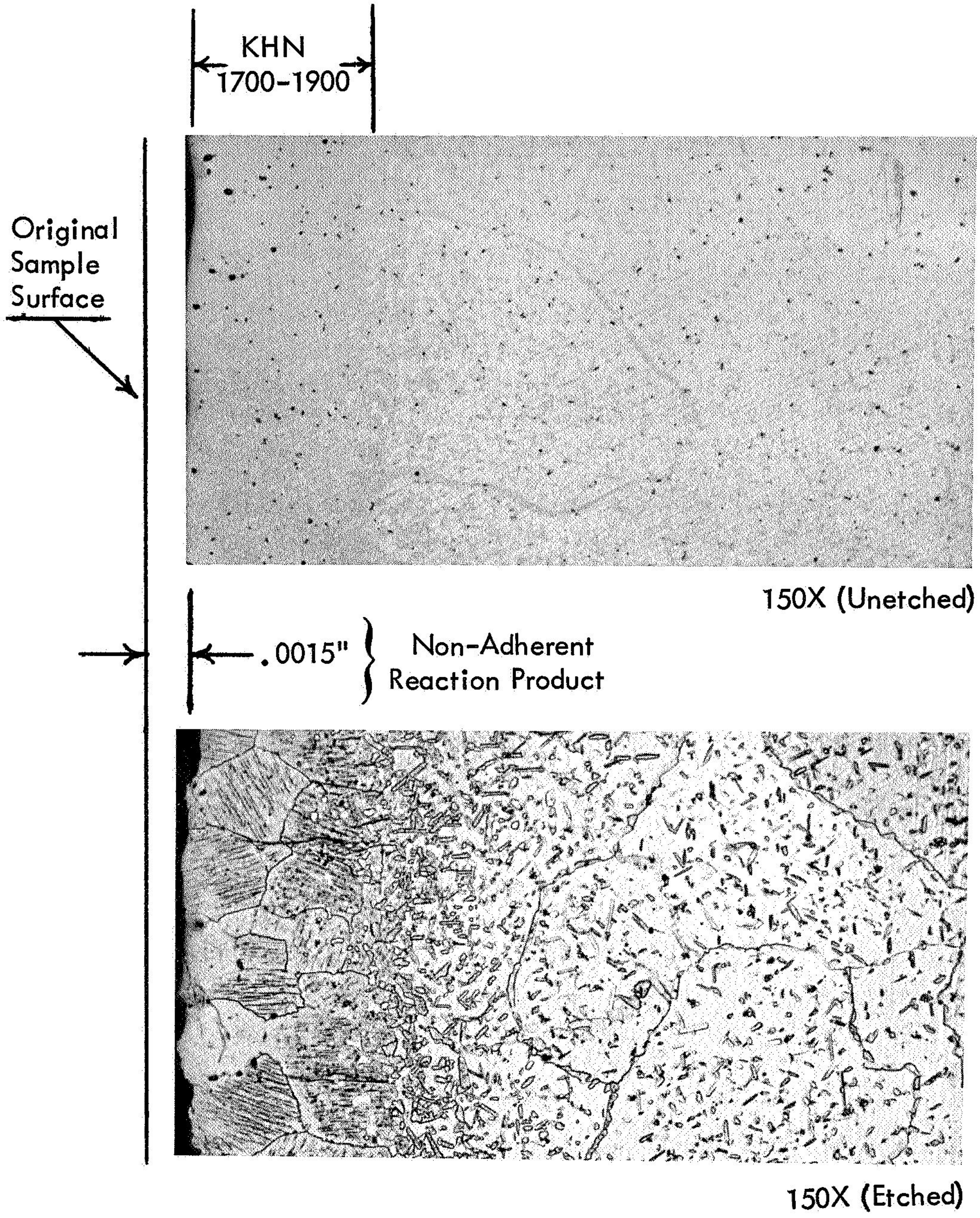

FIGURE A4 - Microstructural Characteristics of Cr-0.22Y Alloy Exposed in Air for 24 Hours at 2400°F

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