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PROTECTIVE CLADDINGS FOR HIGH STRENGTH CHROMIUM ALLOYS

FINAL REPORT

Prepared by
J. F. Collins

Approved by
R. G. Frank

September 15, 1971

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-13495
J. R. Stephens, Project Manager

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CONTRACT NAS 3-13495

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FOREWORD

The work described herein was performed at the Nuclear Systems Programs Department, General Electric Company, under the sponsorship of the National Aeronautics and Space Administration, Contract NAS 3-13495. Mr. J.R. Stephens,Refractory Metals Section, NASA-Lewis Research Center, functioned as Project Manager.

Mr. R.G. Frank, Manager, Nuclear Materials and Components, administered the program for the General Electric Company; Mr. J.F. Collins, Manager, Reactor Materials, directed the program and was principal investigator. Melting and fabrication of the clad sheet was performed by J. Hopkins and M. Kennedy; specimen preparation and evaluation were performed by G. Anderson and W.L. McCullough.

ABSTRACT

The application of a Cr-Y-Hf-Th alloy as a protective cladding for a high strength chromium alloy was investigated for its effectiveness in inhibiting nitrogen embrittlement of a core alloy. Cladding was accomplished by a combination of hot gas pressure bonding and roll cladding techniques. Based on bend DBTT, the cladding alloy was effective in inhibiting nitrogen embrittlement of the chromium core alloy for up to 720 ks (200 hours) in air at $1422^{\circ}K$ ($2100^{\circ}F$). A significant increase in the bend DBTT occurred with longer time exposures at $1422^{\circ}K$ or short time exposures at $1589^{\circ}K$ ($2400^{\circ}F$).

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I. SUMMARY

The effectiveness of a ductile chromium-base alloy (Cr-Y-Hf-Th) as a protective cladding for a high strength chromium-base alloy (Cr-Mo-Ta-Y-C) was investigated for high temperature service in air. The cladding alloy was induction melted and fabricated in the form of sheet with less than 100 ppm total oxygen and nitrogen content and the bend ductile-brittle transition temperature (DBTT) was determined to be 372°K (210°F). A process was developed for fabrication of clad composites by gas pressure bonding and hot rolling to thin sheet. The DBTT of the composite sheet was determined to be 700°-713°K (800-824°F). The effectiveness of the cladding in inhibiting nitrogen embrittlement of the core was determined by exposure of the composite sheet to flowing air at 1422°K and 1589°K. After 720 ks (200 hours) at 1422°K only partial penetration of the cladding had occurred and no increase in the DBTT resulted. Longer time exposures at 1422°K and short time exposures (72-360 ks) at 1589°K resulted in complete penetration of the cladding and significant increases in the DBTT of the composite sheet.

II. INTRODUCTION

The properties of chromium-base alloys are sufficiently attractive to make them potential candidates as structural materials for advanced airbreathing engines. Chromium combines a high melting point, 2148°K (3407°F) with good oxidation resistance and intermediate density. For this reason, the development of a chromium base alloy with superior high temperature creep resistance and adequate low temperature ductility has been the goal of researchers for well over a decade. Significant advances were made in enhancing the elevated temperature strength of chromium. The strongest alloys of chromium include a moderate refractory metal addition. Clark studied the influence of tungsten, molybdenum, and vanadium on the elevated temperature tensile strength of chromium and in concentrations of 3 to 6 atom percent found tungsten to be the most significant strengthener. He found tungsten to increase the tensile strength of chromium an average of 56 MN/m^2 (8100 psi) per atom percent at 1589°K (2400°F).(1)*

Molybdenum and tungsten greatly enhance the high temperature creep resistance of chromium. However, associated with this strength increase is a decrease in low temperature ductility (∿477°K [400°F] increase in the tensile DBTT). The impact ductile-brittle transition temperature apparently suffers an even larger increase to temperatures near 811°K (1000°F). The insertion of reactive metals with carbon which form a fine carbide dispersion, in combination with a solid solution strengthener, resulted in further escalation of the DBTT.

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*Refers to references listed in Section VII.

Sims and Clark,(2) Ryan(3) and more recently, Filippi(4) have intensively studied the influence of carbide and boride dispersions on chromium alloys. The reactive metal elements of Group IV-A and V-A combine with carbon (boron) to form a fine carbide (boride) dispersion in chromium. These dispersions were found to enhance the high temperature strength of chromium without seriously imparing low temperature ductility. Tantalum and columbium additions were particularly effective in this regard. Dramatic improvement in stress rupture strengths were achieved in tantalum and columbium containing alloys by Filippi through microstructural control by heat treatment. Cr-0.5Ta-0.5B, Cr-0.5Ta-0.5C and Cr-0.5Cb-0.5B alloys* were heat treated at 1811°K (2800°F) to form the $Cr_{23}C_6$ (Cr_4B) phase and aged for 10.8 ks (3 hours) at 1422°K (2100°F) to partially solution the $Cr_{23}C_6$ (Cr_4B) phase and precipitate the stable Group VB monocarbide (diboride). Further carbide (boride) interchange precipitation of the monocarbide (diboride) occurs under stress. The 360 ks (100 hour), 1422°K (2100°F) stress rupture strength of a Cr-0.5Ta-0.5C dispersion strengthened alloy in the heat treated conditon is of the same order of magnitude as the previously reported 360 ks (100 hour), 1422°K (2100°F) stress rupture strength of a Cr-4Mo-0.6Ta-0.4C alloy which contains a solid solution strengthener (4Mo) in addition to the TaC dispersoid, 110 MN/m^2 (16 ksi). Significantly, the Cr-0.5Ta-0.5C alloy has a lower tensile DBTT than the Cr-4Mo-0.6Ta-0.4C ($\sim$477°K [400°F] vs. 672°K [750°F]).

One of the major deterrents to the successful stabilization of the high strength chromium base alloys is the tendency toward nitrogen

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*Atom percent, unless otherwise noted.

embrittlement during high temperature exposure to air. Several approaches have been employed to alleviate this problem including protective coatings(5) and alloying.(6)

One of the most important alloying discoveries was that minor additions of the Group III-A metals (rare earths), particularly yttrium, increased the oxidation and nitridation resistance of chromium by several orders of magnitude.(7,8) Although the mechanism by which nitridation resistance is improved is not completely understood, the effect is reproducible.(7,9) Furthermore, yttrium additions also enhance the low temperature ductility of chromium, even after high temperature exposure.(9,10) One of the hypotheses explaining this phenomenon is that the yttrium improves the adherence of the surface oxide film, Cr_2O_3, preventing nitrogen penetration to some extent.(11)

It has been shown that thorium, hafnium, and titanium in combination with yttrium further lowers the oxidation and nitridation rate of chromium(1) and thereby increases the resistance to nitrogen embrittlement. Limited studies on a Cr-Y-Hf-Th alloy indicated that low weight gains due to oxygen and nitrogen pickup resulted after exposure over the temperature range 1089°K (1500°F) to 1589°K (2400°F) for times up to 360 ks (100 hours). In addition, this alloy has the added advantage of exhibiting room temperature ductility.(1) The combination of these two properties make this alloy attractive as a protective cladding for the high strength chromium alloys having poor nitridation/oxidation resistance.

The specific objective of this program is to determine the effectiveness of a ductile chromium alloy (Cr-Y-Hf-Th) as a protective cladding for high temperature air exposures of high strength chromium alloy cores.

The overall goal is the protection of a high strength chromium alloy from embrittlement by oxygen and nitrogen for approximately 7.2 Ms (2000 hours) at temperatures of 1422°K (2100°F) to 1589°K (2400°F).

The scope of the program includes: Phase I - cladding fabrication in the form of 0.635 cm (0.25 inch) and 0.27 cm (0.050 inch) thick sheet and determination of the bend ductile-brittle transition temperature (DBTT). Phase II - process development for preparation of composites rolled to 0.089 cm (0.035-inch) thickness and determination of the bend DBTT, and Phase III - evaluation of the properties of composite sheets after an exposure to air at 1422°K (2100°F) and 1589°K (2400°F). The bend DBTT was used as the primary criterion for cladding effectiveness.

III. Cr-Y-Hf-Th CLADDING ALLOY FABRICATION AND EVALUATION

A. Materials

Two grades of chromium were used in the program: Iodide crystals (99.996 w/o Cr) and H_2-reduced Grade A electrolytic flake (99.94 w/o Cr). Typical chemical analyses of the chromium crystals and flake as supplied by the vendors are reported in Table 1. The minor alloying additions required for the program were obtained in high purity form. Yttrium was obtained in the form of sponge (99.9 w/o Y); hafnium was obtained in the form of crystal bar (99.9 w/o Hf excluding Zr) and thorium was obtained in the form of reactor grade wrought plate (99.5 w/o Th). Typical chemical analyses of the minor alloying additions used also are reported in Table 1. It should be noted that since the total amount of alloying elements added to the chromium was less than 0.3 w/o (0.015 a/o), any one impurity element in the alloying additions has a negligible effect on the impurity concentration of the chromium alloy ingots.

B. Melting

The induction melting and casting procedures for the dilute chromium alloys of the Cr-Y-Hf-Th type had been established on previous NASA sponsored programs (1) and basically the same melting and casting procedures were followed in this program. However, the combined requirement of achieving a maximum total interstitial concentration of 150 ppm and specified concentrations of the reactive elements yttrium, hafnium, and thorium at levels of 0.17 w/o (0.1 a/o), 0.05 w/o (0.015 a/o) and 0.05 w/o (0.012 a/o), respectively proved to be difficult. A total of ten

TABLE 1

TYPICAL ANALYSES OF RAW MATERIALS USED IN MELTING OF CHROMIUM ALLOYS

Element	Purity	Form	Impurities, ppm										
			O	N	H	C	Fe	Ni	Al	Si	S	Ca	Other
Chromium	99.996%	Iochrome Crystals	20	1	< 1	10	30	2	< 1	1	1	0.3	(b)
Chromium	99.94%	H_2-reduced flake	42	84	< 8	58	75	25	10	70	75	5	-
Yttrium	99.9%	Sponge	890	8	-	-	20	-	5	50	-	10	400RE(c)
Hafnium(a)	99.9%	Crystal Bar	160	15	-	70	200	-	15	25	-	-	1.6% Zr
Thorium	99.5%	Reactor grade	-	220	-	1050	-	-	-	60	-	-	3B,15Be, 60Cu,4Mn

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(a) Purity refers to combined Zr and Hf contents.

(b) Other: Cu, 1; Mg, 0.2; Mn, 0.1; V, 1; K, 0.3; Cl, 0.7; Na, 0.4 (ppm).

(c) Rare Earth impurities, principally Yb, Ho, Ce, Pr, Nd.

heats of 3.2 kg (7-pounds) each were melted in order to produce four heats with chemistries that were acceptable to the program.

Melting of the Cr-0.17Y-0.05Hf-0.05Th (w/o) Cr-0.1Y-0.015Hf-0.012 Th (a/o) alloy was accomplished in an induction melting furnace which has the capability to melt under high pressure, 2.76 MN/m^2 (400 psi), as well as the capability to melt under vacuum. A photograph of the facility is shown in Figure 1. The furnace was designed to handle up to 5.5 kg (12 pounds) of steel in a 0.19 m^3 (6.8 ft^3) chamber. The power source for melting consists of a 30 kw, 4200 cycle motor generator set.

Evacuation of the chamber is accomplished by means of a 10.2 cm (4 inch), 0.75 m^3/s (750 l/s) diffusion pump equipped with a cold trap and backed up with a 0.057 m^3 (2 CFM) mechanical pump; initial evacuation was accomplished with a 0.43 m^3 (15 CFM) mechanical pump. The vacuum system was capable of achieving an initial pressure of 10^{-3} N/m^2 (10^{-5} torr) in the melting chamber prior to heating the charge in preparation for melting.

Melting Parameters

The melting and casting procedures that were ultimately utilized in the preparation of the Cr-Y-Hf-Th alloy ingots used for the fabrication of the composite sheet consisted of the following steps:

1. Clean and condition the facility, evacuate the chamber to $<1.3 \times 10^{-3}$ N/m^2 ($<1 \times 10^{-4}$ torr) and determine the leak rate. When properly conditioned, the leak rate is $<4.4 \times 10^{-3}$ N/m^2 ($<3.3 \times 10^{-5}$ torr) per minute.

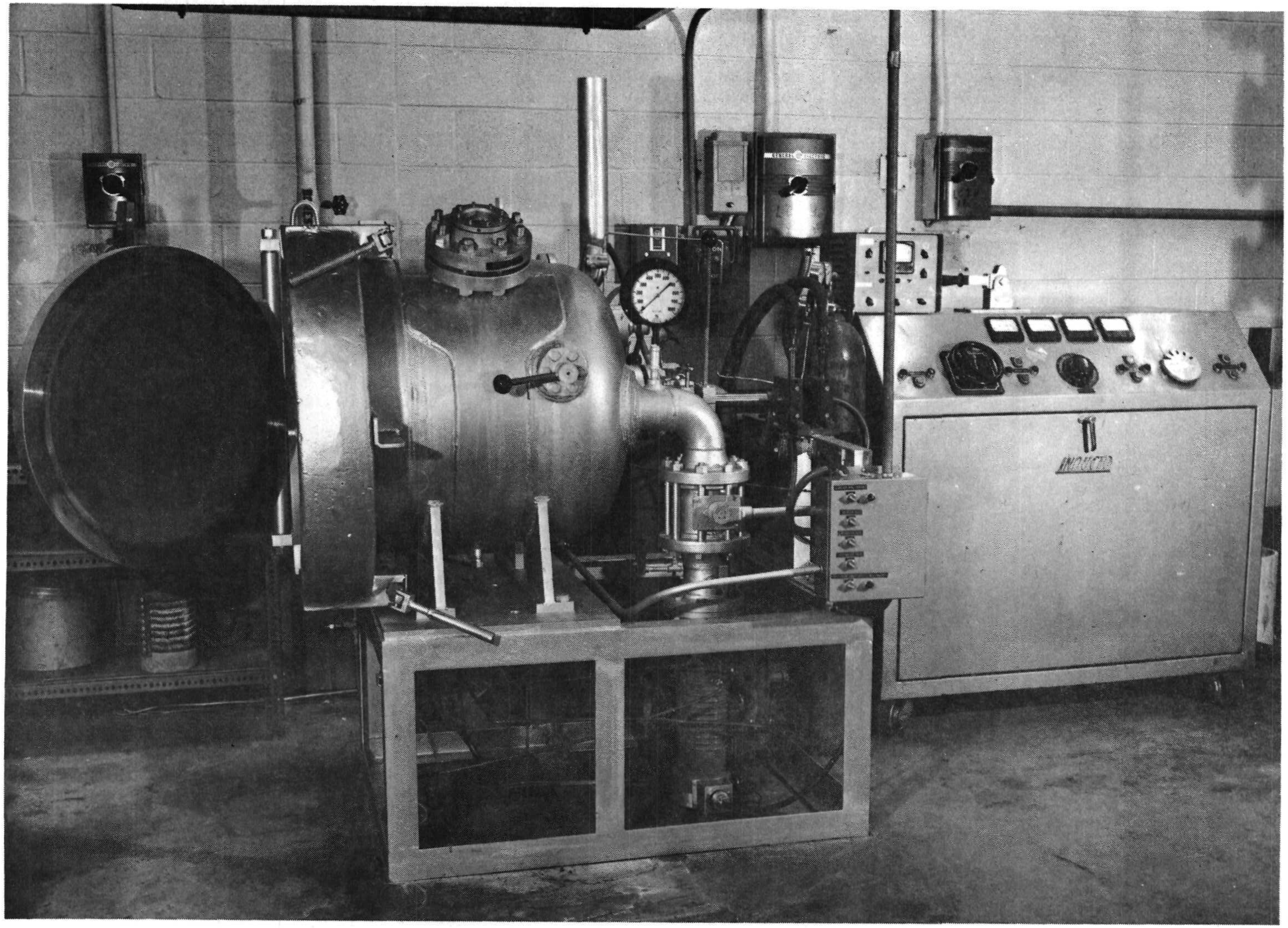

Figure 1 - Induction Melting Furnace with capability of melting under high pressure gas or vacuum.

(Neg. P69-3-1B)

2. Pack the Y_2O_3 or CaO stabilized ZrO_2 crucible in the induction coil; seal with a ZrO_2 collar. Install a 6.4 cm (2.5 inch) ID copper mold with a CaO stabilized ZrO_2 liner, hot top and tundish.

3. Bake-out the crucible at 922°-1033°K (1200°-1400°F) by inductively heating a molybdenum susceptor placed inside the crucible. The crucible is baked under a dynamic vacuum for a period of approximately 7.2 ks (2 hours) or until the pressure in the chamber reached approximately 10^{-2} N/m^2 (10^{-4} torr). Then the crucible is allowed to cool to room temperature under vacuum.

4. Place the preweighed chromium and yttrium charge in the crucible and the yttrium, hafnium and thorium charge in the hopper. Evacuate the chamber to a pressure of 10^{-2} to 10^{-3} N/m^2 (10^{-4} to 10^{-5} torr) and hold overnight, approximately 54 x 10^3s(∿15 hours). Determine the leak rate until a rate of <1.3 x 10^{-2} N/m^2 (<1 x 10^{-4} torr) per minute is achieved.

5. Heat the charge in the crucible to 1033°K (1400°F) and hold under vacuum to remove adsorbed gases; this is accomplished in about 900s(15 minutes). Introduce high purity argon gas to the chamber until a pressure of 6.7 x 10^4 N/m^2 (500 torr) is achieved, ∿420 s (7 minutes). A 99.99% argon gas is utilized with an oxygen concentration of <5 ppm.

6. Increase power to complete melting of the charge in the crucible in about 900 s (15 minutes). Adjust the power to maintain a stable super heat of 84^oK (150^oF) and hold for 900 s (15 minutes). The pressure at this time is approximately 8.2 x 10^4 N/m^2 (620 torr).

7. Add balance of yttrium and hafnium and thorium to charge and hold for 300 s (5 minutes). Note: no noticeable reaction was observed.

8. Cast 3.2 kg (7 pound) heat into a CaO stabilized ZrO_2 lined copper mold to yield a 5.1 cm (2 inch) diameter ingot, 8.9 cm (3.5 inches) long and weighing approximately 1.4 kg (3 pounds). The cast ingot is allowed to cool under a partial pressure of argon. Note: A considerable amount of slag remains in the crucible.

9. Remove the hot top from the ingot, cut a slice from the top and bottom of the ingot for analytical samples and inspect for internal defects using radiographic inspection techniques.

Crucible Materials

The initial two heats of the Cr-Y-Hf-Th alloy were melted in a CaO stabilized ZrO_2 crucible utilizing Grade A electrolytic flake (MP-16) and iochrome crystals (MP-17). Although no problems were encountered in the melting of either heat, analytical results revealed a high nitrogen content in MP-16 (Table 2). In an attempt to achieve a lower concentration of total interstitial elements, two additional heats (MP-18, MP-19) were melted using Y_2O_3 stabilized ZrO_2 crucibles and iochrome crystals. Melting procedures were the same as those utilized for heats MP-16 and MP-17. However, analytical results of samples taken from the top of these ingots showed oxygen concentrations of 680 for MP-18 and 1003 ppm for MP-19. To determine whether the high oxygen levels could be attributed to improperly prepared Y_2O_3 stabilized ZrO_2 crucibles, another heat (MP-20) utilizing iochrome crystals was melted in a CaO stabilized crucible. Again, an analysis of this heat showed a high

TABLE 2

CHEMICAL ANALYSES OF Cr-Y-Hf-Th ALLOY INGOTS AND SHEET

(INTERSTITIAL ELEMENTS)

Heat No.	Crucible	Chromium Grade	Sample Location	Analyses, ppm			
				O	N	H	C
MP-16	CaO/ZrO_2	Grade A	Ingot Top	35	126	1	234/24(a)
			" Bottom	50	107	1	199
MP-17	CaO/ZrO_2	Iochrome	" Top	59	55	2	114/19(a)
			" Bottom	42	52	1	139
MP-18	Y_2O_3/ZrO_2	Iochrome	" Top	680	28	7	-
MP-19	Y_2O_3/ZrO_2	Iochrome	" Top	1003	123	6	-
			" Bottom	983	35	6	-
MP-20	CaO/ZrO_2	Iochrome	" Top	850	32	-	-
MP-21	CaO/ZrO_2	Grade A	" Top	18	31	1	-
			" Top	30	52	2	100
			" Bottom	40	62	2	100
MP-22	CaO/ZrO_2	Grade A	" Top	23	27	5	-
MP-23	CaO/ZrO_2	Grade A	" Top	49	35	2	-
MP-24	CaO/ZrO_2	Grade A	" Top	26	35	3	-
MP-25	CaO/ZrO_2	Grade A	" Top	109	81	13	100
			" Top	27	22	-	80
MP-24	CaO/ZrO_2	Grade A	0.127 cm (0.050 in) Sheet	93	154	-	131
			"	140	94	2	42
			"	56	51	-	100
			"	140	90	-	42
			Avg.	107	97	2	79

(a) Recheck analyses on extruded alloy.

oxygen concentration of 850 ppm. An examination of the melting log indicated some difficulty was experienced in melting the large irregular pieces of iodide chromium because they could not be consolidated in the crucible readily (poor coupling) due to a bridging problem. As a result, a longer heating period was required for the melting of the iochrome crystals than when melting Grade A electrolytic flake. The oxygen analysis of heat MP-20 melted in a CaO stabilized crucible indicates that the longer heating time required for the melting of the iodide chromium is the major factor in the high oxygen content of the ingots.

All of the subsequent heats (MP-22 through MP-25) that were required to be melted in the program were melted with Grade A electrolytic flake in CaO stabilized ZrO_2 crucibles. The selection of CaO stabilized crucibles was based on work by Clark(12) where equivalent levels of interstitial impurities existed for the same alloy composition melted in both types of crucibles. The oxygen and nitrogen concentrations determined from samples taken from the top and bottom of ingots MP-22 to MP-25 were found to be within the acceptable range (Table 2). The total oxygen plus nitrogen contents of these heats were less than 100 ppm with the possible exception of MP-25. The carbon concentration was relatively high and the source of the carbon contamination was not determined.

Chemical Analysis

The analytical results for the principal alloying elements yttrium, hafnium and thorium and for trace impurity elements in the Cr-Y-Hf-Th alloy ingots are given in Tables 3 and 4. The retention of yttrium and thorium in the first series of heats (MP-16 to MP-21)was considerably below the desired composition. Improvement in retention of the reactive elements was achieved in three ways: (1) by reducing the holding time of the melt at superheat prior to the addition of the reactive elements to the melt from the hopper from 15 to 10 minutes as was the case for heat MP-21; (2) by reducing the holding time of the melt after the addition of the reactive elements to the melt from the hopper from 5 to 4 minutes as was done for heats MP-23, -24 and -25; and (3) by increasing the amounts of reactive elements added to the charge and to the melt from hopper (see Table 3). The procedure that was found to achieve an acceptable alloying concentration in the cast ingots consisted of placing 4000 ppm yttrium (12.7 g) in the crucible with the initial chromium charge and 3000 ppm yttrium (9.5 gm) in the hopper for subsequent addition to the melt before the hafnium and thorium. The thorium addition of 650 ppm (2.1 g) and the hafnium addition of 600 ppm (1.9 g) were encased in a small cube of chromium from a previous heat (MP-21), so that when it was added to the melt the thorium and hafnium would be carried below the surface. This was done to insure that these very small additions would not be lost in the slag and as a means of improving the retention of these addition elements.

Oxidation testing of heats MP-16, MP-17, MP-21, MP-22, MP-23 were conducted at 1422°K (2100°F) for times to 360 Ms (100 hours) in flowing air to supplement chemical analyses data for yttrium, thorium and hafnium. The results are presented in Table 5 and indicate good oxidation resistance in comparison to previous data obtained on another program.(1) For example, the specimen from MP-22 gained 0.30 mg/cm^2 in 360 Ms (100 hours)

TABLE 3

CHEMICAL ANALYSES OF Cr-Y-Hf-Th ALLOY INGOTS
(REACTIVE ALLOYING ELEMENTS)

	Amount Reactive Element Added, ppm				Analyses,[a] ppm			
	Y		Hf	Th				
Heat No.	Crucible	Melt	Melt	Melt	Sample Location	Y	Hf	Th
MP-16	3000	1000	500	500	Ingot Top	260/600[b]	1000	270
					" Bottom	260	1100	270
MP-17[c]	3000	1000	500	500	" Top	10/<100[b]	750	53
					" Bottom	10	800	57
MP-21	3000	1000	500	500	" Top	570	270	52
					" Bottom	540	250	57
MP-22	3000	3000	600	650	" Bottom	475 1500[d]	600	220
MP-23	4000	4000	600	650	" Bottom	2750	620	400
MP-24	4000	3000	600	650	" Bottom	1970	340	450
MP-25	4000	3000	600	650	" Bottom	1050	300	480

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[a] Analytical techniques used:

Y - X-ray fluorescence.
Hf - Gravimetric/emission spectrography.
Th - Colormetric or X-ray fluorescence.

[b] Recheck analysis on extruded alloy.

[c] MP-17 made with iodide chromium; others made with Grade A electrolytic chromium.

[d] Analysis obtained on oxidized surface.

TABLE 4

CHEMICAL ANALYSES OF Cr-Y-Hf-Th INGOTS AND SHEET

(METALLIC IMPURITIES)

Element	Analyses,[a] ppm						
	MP-16 Ingot		MP-17[b] Ingot		MP-21 Ingot		MP-24
	Top	Bottom	Top	Bottom	Top	Bottom	0.127 cm (0.050 in.) Sheet
S	ND<25[c]	ND<25	ND<25	ND<25	ND<25	ND<25	50
Al	150	100	30	10	70	70	30
Ca	ND<10[c]	ND<10	ND<10	ND<10	ND<10	ND<10	ND<10
Fe	70	50	1500	1500	50	50	20
Mo	50	50	ND<10	ND<10	50	50	-
Ni	ND<10[c]	ND<10	10	10	ND<10	ND<10	ND<10
Si	400	300	100	100	400	400	160
Ti	20	10	ND<10	ND<10	ND<10	ND<10	ND<10
Zr	4000	20	500	300	70	70	50

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[a] Analytical techniques used: S - Combustion/titrametric; all other trace elements by emission spectrographic analysis.

[b] MP-17 made with iodide chromium; others made with Grade A electrolytic chromium.

[c] ND < not detected, less than . . . .

TABLE 5

AIR OXIDATION OF Cr-Y-Hf-Th ALLOYS AT 1422°K (2100°F) FOR 360 Ms (100 HOURS)

Heat No.	Condition(a)	Retained Y, ppm	Total Weight Gain, mg/cm^2	Comments
MP-16	Extruded, Annealed	260-600	2.7 net(b)	Spalled mildly
MP-17	Extruded	10-20	3.0 gross 0.1 net	Spalled heavily
MP-21	As-cast	540	3.05 net	Spalled mildly
MP-22	As-cast	475	0.30	Adherent Oxide
MP-23	As-cast	2750	0.96	Adherent Oxide

(a) Approximately 5 gm samples with 5.5 cm^2 average area.

(b) Net weight of specimen after loss of spalled product.

at 1422°K (2100°F) vs. the published data of 1.9 mg/cm^2 obtained on a similar alloy.

From the chemical analyses of the four ingots MP-22, MP-23, MP-24 and MP-25, and supplemental oxidation tests of MP-22 and MP-23, heat MP-24 was selected for fabrication of the 1.27 mm sheet and Heats MP-22 and MP-23 were judged to be satisfactory for the 6.35 mm thick frame material. Heat MP-25 was held as reserve material in ingot form.

C. Ingot Conversion and Sheet Fabrication

Eight ingots were radiographically inspected and found to be free of voids. Each ingot was machined to 4.83 cm (1.90 inch) diameter to fit into 5.1 cm (2.0 inch) OD molybdenum containers for extrusion. Fluorescent penetrant inspection of the machined billets MP-22, -23, -24 revealed shallow indications on the bottom of each ingot. These areas were removed by machining and the chamfer for the nose was machined on the opposite (top) end of the billets. Reinspection showed that the defects had been eliminated. The machined ingots are shown in Figure 2 as prepared for assembly with their molybdenum containers.

The ingots and the molybdenum containers were chemically cleaned and outgassed separately by heating to 1033°K (1400°F) in vacuum (2.7×10^{-4} N/m^2/2×10^{-6} torr). The ingots were sealed in the containers by EB welding the molybdenum end caps in place. The end-caps were designed with a flange (Figure 2) so that the weld could be made on the circumference of the molybdenum container rather than on the end. This design reduces stresses in the welded joint and insures a leak-tight seal of the containers.

Figure 2. Induction Melted and Machined Cr-Y-Hf-Th Alloy Ingots Prior to Being Sealed in Mo Jackets for Extrusion. (Neg. P70-8-18)

Hot extrusion of the jacketed ingots was performed using a 11.1 MN (1250 ton) horizontal extrusion press equipped with a 5.4 cm (2.125 inch) ID container. The dies were sprayed with ZrO_2 and had a 1.27 cm x 2.54 cm (0.5 inch x 1.0 inch) rectangular cross section giving a reduction ratio of approximately 7:1. The billets were inductively heated in argon and held at 1533°K (2300°F) for 0.9-1.8 ks (15-30 minutes) prior to extrusion. The hot billet was coated with powdered glass (Corning 8871) and the die was lubricated with Necroline and graphite.

Extrusion parameters were established with billets MP-16, -17, -20 and -21 prior to extruding billets MP-22, -23 and -24 for subsequent rolling to sheet. Billet MP-20 was extruded to sheet-bar at a ram speed setting of 7.6 cm (3 inches)/sec. Although the surface appearance of MP-20 was good, the press stalled before completion of the extrusion. Hence, the ram speed was increased for billet MP-16 with the result that the molybdenum jacket was torn and the chromium alloy core had a rough surface. The ram speed settings and extrusion forces for all extrusions are given in Table 6. The optimum ram speed setting appeared to be 12.7 cm (5 inches)/sec which resulted in a good surface condition for MP-21.

The sheet bar extrusions of MP-16, -17 and -21 were stripped of the molybdenum jacket by leaching in nitric acid. The chromium alloy bars were radiographed and no cracks or voids were detected. Fluorescent penetrant inspection revealed fine surface cracks in MP-21 but no surface defects were detected in the extruded bars of MP-16 and MP-17.

TABLE 6

EXTRUSION DATA FOR Cr-Y-Hf-Th ALLOY INGOT REDUCED 7:1 TO RECTANGULAR SHEET BAR

Billet No.	Ram Speed Setting cm/s (in./s)	Extrusion Force, N (Tons)		Surface Condition
		Upset	Average	
MP-20	7.6 (3)	2.22 (250)	1.96 (220)	Very good; press stalled
MP-16	17.8 (7)	2.00 (225)	1.78 (200)	Poor, Molybdenum Jacket Torn
MP-17	15.3 (6)	2.00 (225)	1.86 (210)	Fair to good
MP-21	12.7 (5)	2.14 (240)	1.78 (200)	Very good

NOTES:

1. All billets preheated in argon at 1533°K (2300°F) for 900-1800 s (15-30 minutes).
2. Glass lubricant was Dow-Corning #8871.
3. Container diameter, 5.4 cm (2.125 in.).
   Die size, 1.27 cm x 2.54 cm (0.5 in. x 1.0 in.).

Billets MP-22, -23, -24 were extruded to 1.27 cm x 2.54 cm sheet bar using the parameters established for the previous four billets (Table 6). The extrusion force averaged 1.52 MN(170 tons). The surface condition of the three sheet bar extrusions was good on one side and was torn on the opposite side indicating inadequate lubrication or a defect in the extrusion die.

The molybdenum jackets were leached from the extrusions in dilute nitric acid revealing a rough surface on sections of MP-23 and MP-24 as shown in Figure 3. The sheet bars were inspected using fluorescent penetrant techniques which revealed surface cracks in the nose and tail of MP-23 and MP-24; MP-22 was free of cracks. The defect areas were removed and the sheet bars were cut into lengths of 10 cm (4 inches) for cross rolling to sheet. The surface of all sheet bar sections were hand ground to remove all surface defects and were inspected by fluorescent penetrant techniques to insure freedom of surface cracks. The sections were individually jacketed in flattened mild steel tubing with an interface of molybdenum foil to prevent iron diffusion into the chromium alloy. The ends of the tube jacket were closed by press-forging.

The steel and molybdenum jacketed billets were heated in a hydrogen atmosphere furnace at 1323°K (1922°F) for a 600 s (10 minute) soak initially and a 180 s (3 minute) reheat between rolling passes. Reductions of 10 percent per pass were taken until the chromium alloy was reduced to 6.35 mm (0.25 inch) thick for MP-22 and MP-23 and to 1.27 mm (0.050 inch) thick for MP-24. Following the final pass, the sheets were given a stress-relief treatment at 1323°K (1922°F) for 1.2 ks (20 minutes). The total reduction by rolling averaged 86 percent for the 1.27 mm sheet stock and approximately 30 percent for the 6.35 mm frame stock.

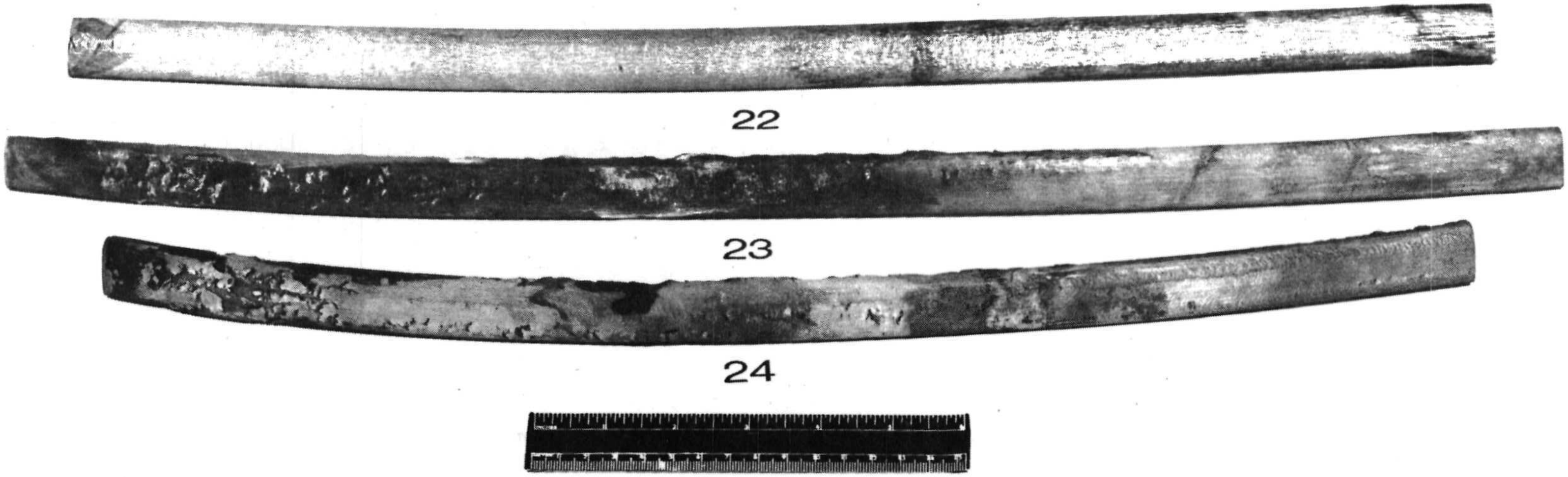


Figure 3. Extruded Sheet Bars of Cr-Y-Hf-Th Alloy After Removal of Molybdenum Jackets. (Neg. P70-11-2A)

The steel jackets and molybdenum foil interface were removed by leaching in a 50% nitric acid solution. Sufficient cladding material of 1.27 mm thickness was hot rolled from sections of MP-24 extrusion to prepare bend test specimens and cladding plates for the required composites.

D. Bend Testing

Specimens of MP-24 were mounted and polished for examination of the microstructure in the as-extruded condition, as-hot rolled to 1.27 mm (0.050 inch) sheet and after annealing for 3.6 ks (1 hour) at 1373°K (2192°F) in hydrogen; transverse and longitudinal sections of each sample were prepared. Microhardness measurements for each condition are given in Table 7 and photomicrographs of the longitudinal sections in each of the three conditions are presented in Figures 4, 5, 6, and 7. It is evident that a uniform and fine grain structure was achieved.

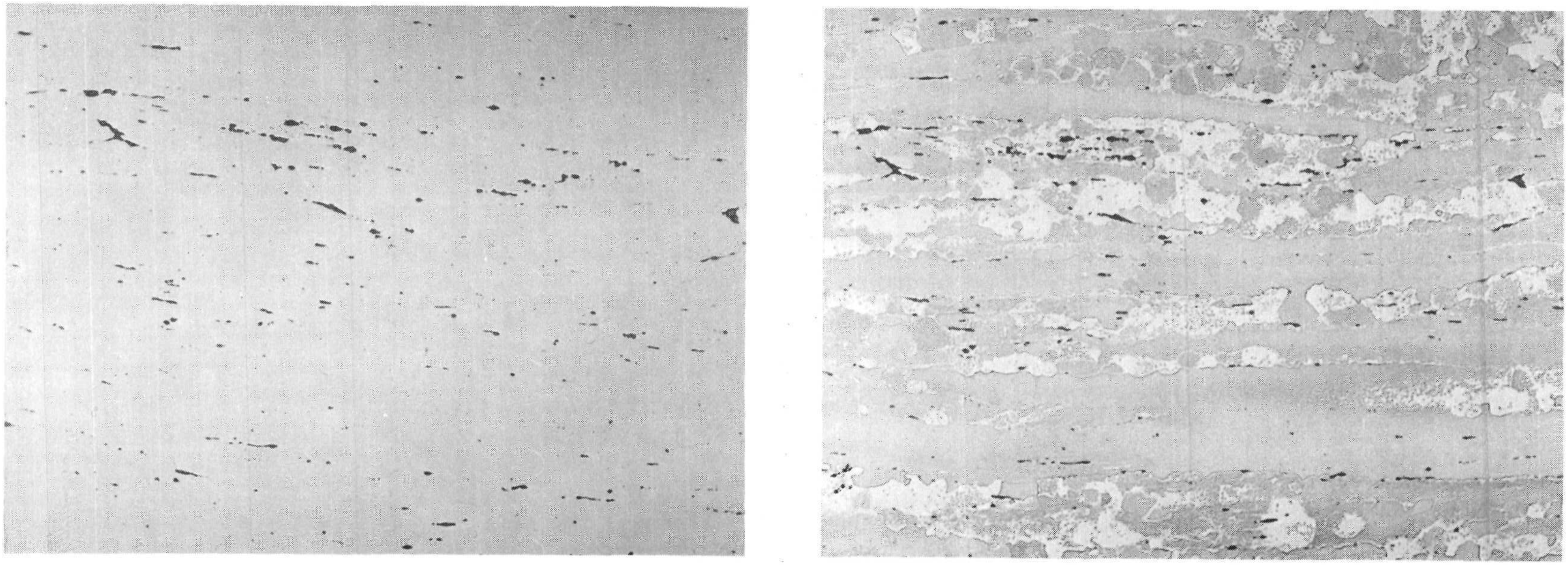
Specimen Preparation

The bend DBTT of the Cr-0.17Y-0.05Hf-0.05Th (w/o) alloy (MP-24) was determined using ten specimens cut from the 1.27 mm (0.050 inch) sheet. To avoid cracking, the chromium alloy sheet was adhesively bonded to a graphite block and cutting was done in a longitudinal direction using an overhead wheel of silicon carbide on a precision wafering machine. The specimens were cut oversize and the edges were ground back to remove any cracks generated in cutting. The surfaces were hand polished through 600 grit paper and all specimens were annealed in hydrogen at 1373°K (2192°F) for one hour. Inspection by fluorescent penetrant techniques was performed to insure freedom from surface cracks and all specimens were electropolished to remove surface scratches. Electropolishing was done in a solution of 39% sulfuric acid, 37% alcohol and 24% water at room temperature, using platinum electrodes

TABLE 7

MICROHARDNESS MEASUREMENTS FOR LONGITUDINAL SECTIONS OF Cr-Y-Hf-Th ALLOY MP-24 IN THREE CONDITIONS

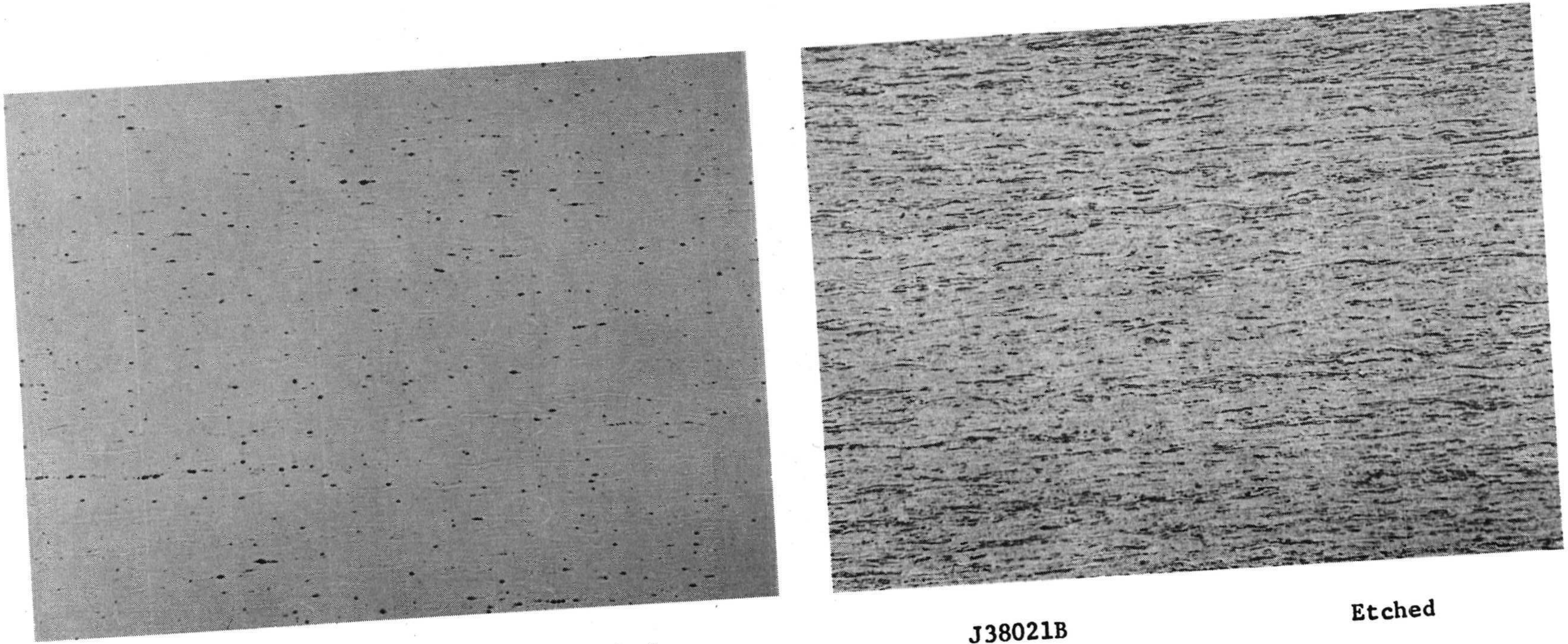
	Hardness, HV_{100}	
Condition	Center	Edge
1. As-extruded at 1533°K (2300°F)	141	153
	147	145
	141	147
2. After warm rolling at 1323°K (1922°F) to 1.27 mm sheet (86% Reduction) and a stress-relief at 1323°K	203	200
	200	176
	189	200
3. Warm Rolled Sheet Annealed at 1373°K (2192°F) for 3600 s (1 hour)	124	134
	133	127
	141	131



J38011A As-polished

J38011B Etched

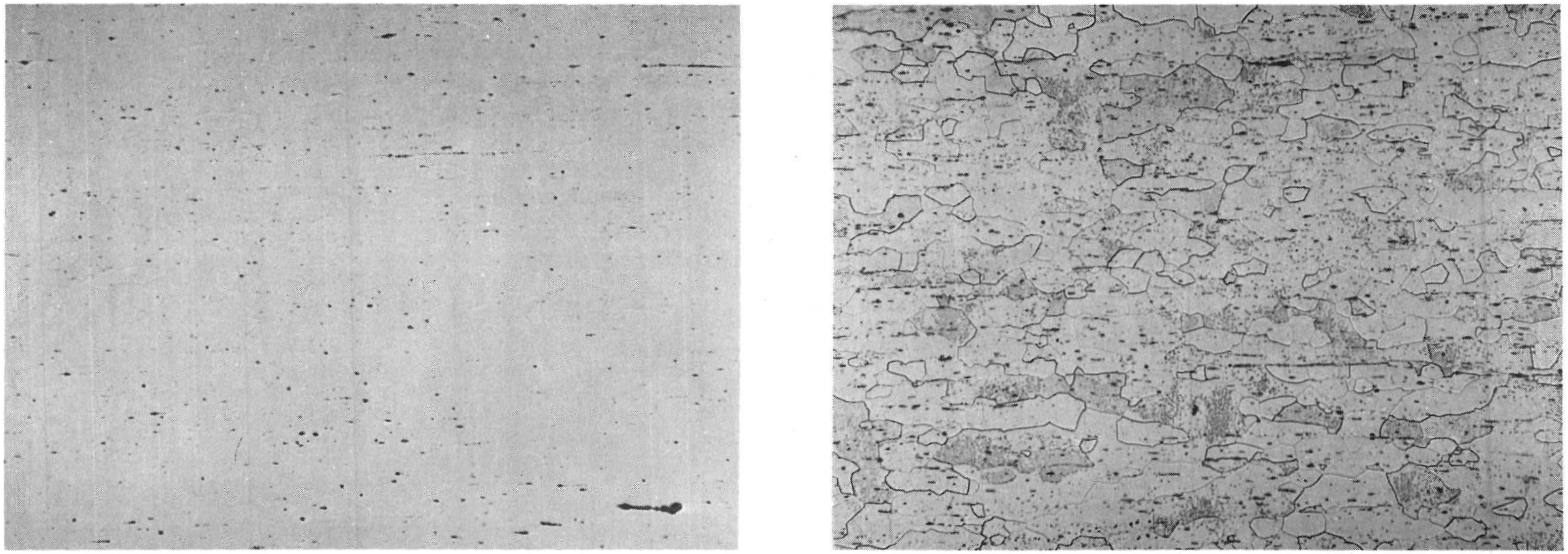
Figure 4. Microstructure of Cr-Y-Hf-Th Alloy MP-24 After Extrusion at 1533°K.
Longitudinal Section, Magnification 100X.
Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2).



J38021A As-polished

J38021B Etched

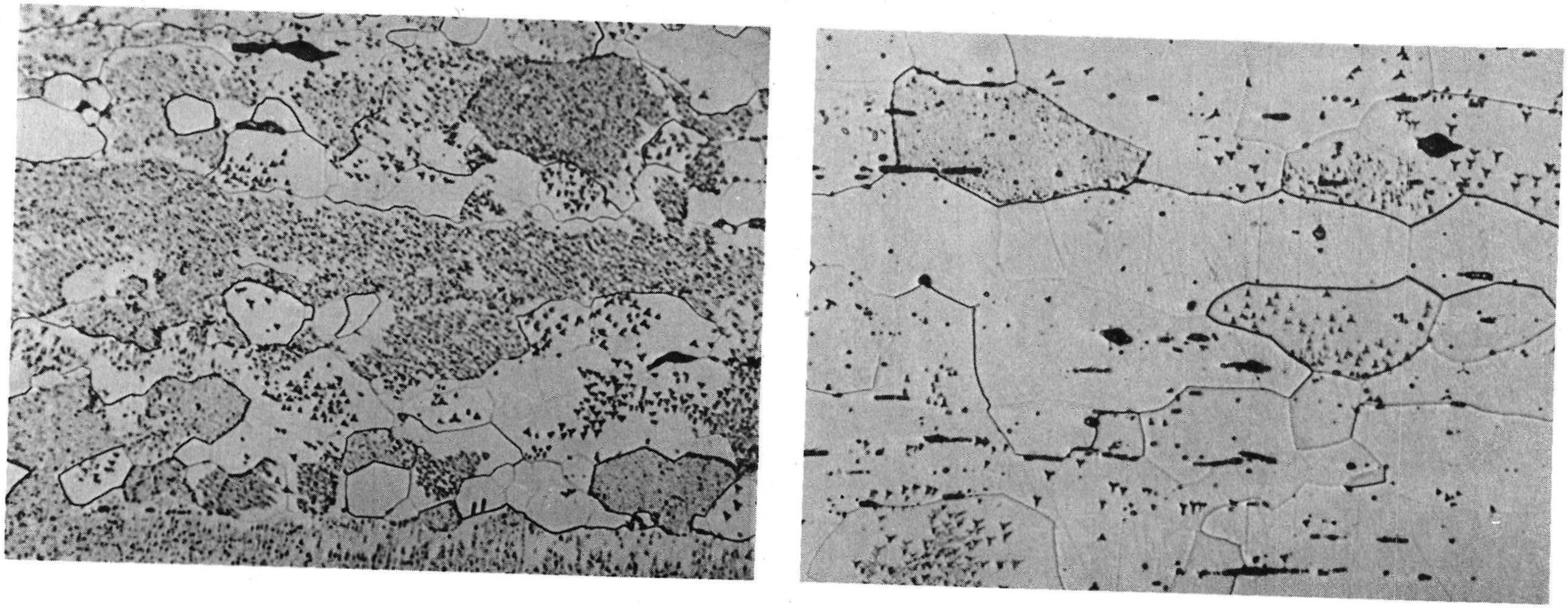
Figure 5. Microstructure of Cr-Y-Hf-Th Alloy MP-24 After Warm Rolling at 1323°K (1922°F) to 1.27 mm (0.050 inch) Thickness (86% Reduction) and a Stress-Relief at 1323°K. Longitudinal Section, Magnification 100X. Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O.



J38031A As-polished

J38031C Etched

Figure 6. Microstructure of Cr-Y-Hf-Th Alloy MP-24 After Warm Rolling (86% Reduction) at $1323^{\circ}K$ ($1922^{\circ}F$) and Annealing at $1473^{\circ}K$ for 1 Hour in Hydrogen. Grain Size is ASTM 5-7 Predominately 6.
Longitudinal Section, Magnification 100X.
Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O.



J38011C Etched

J38031D Etched

Figure 7. Microstructure of Cr-Y-Hf-Th alloy MP-24 after extrusion (left) at 1533°K (2300°F) and after warm rolling (86% reduction) at 1323°K (1922°F) and annealing at 1473°K (2192°F) in hydrogen (right).

Longitudinal section, Magnification 500X.

Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O.

and fixtures. The treatment of each specimen, 7.6 mm x 22.9 mm (0.3 in x 0.9 in.) at 0.8 amp/5.5 volts for 480 s (8 minutes) was sufficient to remove 0.0127 mm (0.0005 inch) per side and eliminate all scratches from the polished surfaces. The thickness of the finished specimens averaged 1.19 mm (0.047 inch).

Bend tests were performed in accordance with Materials Advisory Board Report MAB 216-M, "Evaluation Test Methods for Refractory Metal Sheet Materials," with the following exceptions: the specimen width was 7.6 mm (0.3 inch), the length was 22.9 mm (0.9 inch) and the test span was 15.24 mm (0.6 inch).

DBTT Bend Testing

The bend tests were conducted in a Baldwin Testing Machine equipped to record load and deflection. A punch with a 5.16 mm (0.2 inch) radius was used to make approximately a 4T bend in the chromium alloy specimens. The test specimens and fixtures were heated in an oil bath with temperatures being controlled by a thermocouple in contact with the specimen. In addition, a calibrated mercury thermometer was placed in the oil in contact with the specimen to measure test temperature within $1^{\circ}K$. In accordance with MAB 216-M a crosshead speed of 0.42 mm per s (1 inch per minute) was used.

The data for the series of bend tests are given in Table 8. The load and deflection were read from the recorder of the Baldwin Testing Machine. The bend angle was measured after each test using a calibrated protractor; in addition, the angle was calculated by graphical means using the measured deflection value.

TABLE 8

BEND TEST[(a)] DATA FOR DETERMINATION OF DBTT FOR Cr-Y-Hf-Th CLADDING ALLOY MP-24

Specimen	Thickness		Temperature		Max. Load		Deflection		Bend Angle		Results
	mm	in.	°K	°F	N	lb	mm	in.	°Meas.	°Cal.	
Cr-1	1.22	0.0481	366	200	80.0	18	0.20	0.008	-	4	Broke-2 pcs.
Cr-2	1.18	0.0464	366	200	80.0	18	0.13	0.005	< 10	3	Cracked
Cr-1	1.24	0.0489	422	300	244.4	55	6.20	0.244	95	96	Sml. edge crack
Cr-4	1.15	0.0453	368	204	231.0	52	6.17	0.243	94	95	Ductile
Cr-5	1.21	0.0475	394	250	213.4	48	6.14	0.242	94	94	Ductile
Cr-6	1.27	0.0501	377	225	271.0	61	6.02	0.237	93.5	90	Ductile
Cr-7	1.15	0.0451	374	213	195.5	44	6.10	0.240	93	92	Ductile
Cr-8	1.15	0.0454	370	206	191.0	43	5.38	0.212	89	82	Cracked
Cr-9	1.19	0.0470	366	200	95.6	21.5[b]	0.05	0.002	< 10	1	Internal crack
Cr-10	1.1	0.0433	422	300	160.0	36	6.91	0.272	113	104	Ductile

[(a)] Specimen size 7.6 mm x 22.8 mm (0.3 in. x 0.9 in.)
Fixture Span 15.2 mm (0.60 in.)
Cross-head speed 0.42 mm/s (1.0 in./min) per MAB 216-M
Bend radius 4T (5.16 mm)

[(b)] Cross-head speed 0.21 mm/s (0.5 in./min)

The DBTT for the Cr-Y-Hf-Th alloy (MP-24) as determined from a graphical plot of the bend angle versus test temperature is shown in Figure 8. From this plot, the DBTT for a full bend of 90 degrees is approximately 372°K (210°F) and for a 45-degree bend the transition temperature is only slightly lower, approximately 368°K (203°F). Nil ductility occurred at 366°K (199°F). The program objective specified that the bend DBTT for the cladding alloy for the composites be less than 422°K (300°F). The bend test results show that this condition was met by alloy MP-24 and that it was satisfactory for use in the fabrication of composites.

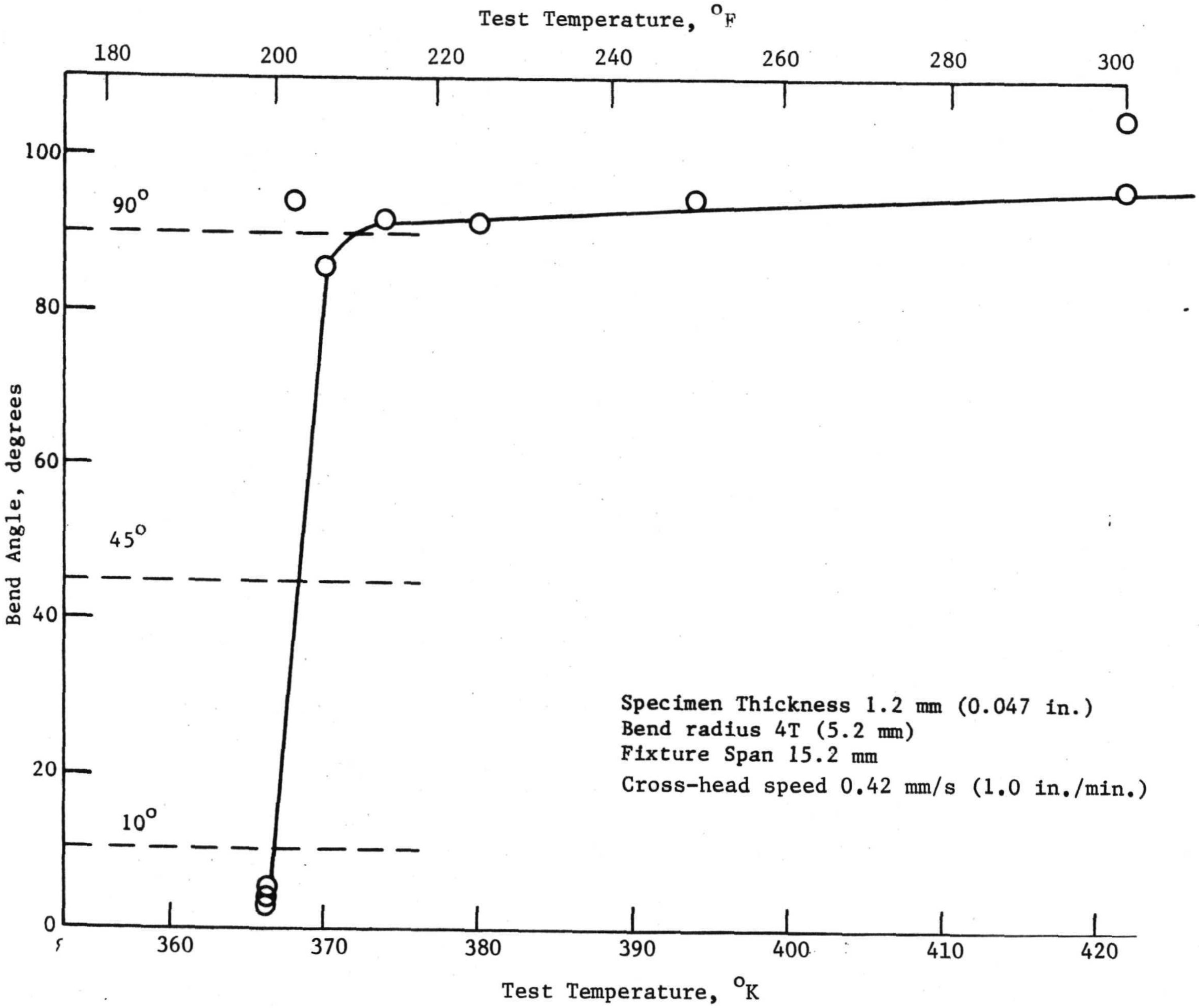


Figure 8. Bend Angle as a Function of Temperature for Cr-Y-Hf-Th Cladding Alloy MP-24 Showing DBTT as 372°K (210°F) for 90 Degree Bend.

IV. COMPOSITE SHEET PROCESS DEVELOPMENT

A. Composite Preparation/Diffusion Bonding

Four 0.64 cm x 2.54 cm x 10.2 cm (0.25 in. x 1.0 in. x 4 in.) plates of a high strength core alloy Cr-4Mo-0.6Ta-0.6Y-0.4C [Cr-7Mo-2Ta-0.1Y-0.09C (w/o)] were furnished by NASA. Detailed processing information for the production of the 0.64 cm thick plate is reported by Slaughter, et al(13) and is summarized in Appendix A. The as-received plate was in the annealed condition 1373°K (2192°F).

Three pieces of the high strength Cr-Mo-Ta-C-Y alloy plate were cut into 9 pieces of core plates, 0.63 cm x 2.54 cm x 2.54 cm (0.25 in. x 1.0 in. x 1.0 in.). The cuts were made on a precision wafering machine with a silicon carbide wheel, exercising care to avoid chipping of corners and edges. All faces of the core plates were polished through 200 grit paper and inspected for cracks by fluorescent penetrant techniques.

Frame pieces were cut from the 0.635 cm thick plates rolled from extrusions of MP-22 and MP-23. A total of 36 pieces were cut using stress-free techniques with the precision wafering machines. These frame pieces were hand ground and polished to match the thickness and size of the core plates. Cladding plates were cut from the 0.127 cm (0.050 in.) thick sheet of MP-24, using the precision wafering machine and with the chromium alloy sheet adhesively bonded to a graphite block. A total of 18 plates were cut to the size 3.8 cm x 3.8 cm (1.5 in. x 1.5 in.). The surface was cleaned by liquid honing and all plates were annealed in hydrogen at 1093°K (1508°F) for 3.6 ks (1 hour). Inspection for cracks was performed by fluorescent penetrant techniques. The surface to be bonded to the core plate was hand polished through 200 grit paper and the edges were hand ground to fit the frame assembly of each composite.

Initial bonding of the Cr-Y-Hf-Th cladding alloy sheet to the Cr-Mo-Ta-Y-C alloy core was accomplished by a hot gas pressure bonding operation. This necessitated that the chromium composite assembly be jacketed in an evacuated container. The containers were fabricated from molybdenum by box-like construction similar to the composite assemblies to form a close fitting enclosure with minimum void volume that could be electron beam welded leak-tight for the gas pressure bonding operations. The molybdenum cover sheets were 0.51 mm (0.020 in.) thick. A typical composite assembly and container assembly are shown in Figure 9.

The molybdenum container assembly was clamped around the chromium composite assembly and three sides of the molybdenum container were joined by GTA welding in a vacuum purged, argon-filled chamber. The final closure was made in vacuum by EB welding after outgassing of the assembly for 7.2 ks (2 hours).

The first five assemblies prepared in this manner were found to contain leaks and efforts to repair the leaks in the molybdenum welds by EB welding were not successful. It is believed that chromium was being vaporized at the low pressures by penetration of the electron beam and that the chromium in the weld prevented a successful closure of the molybdenum container.

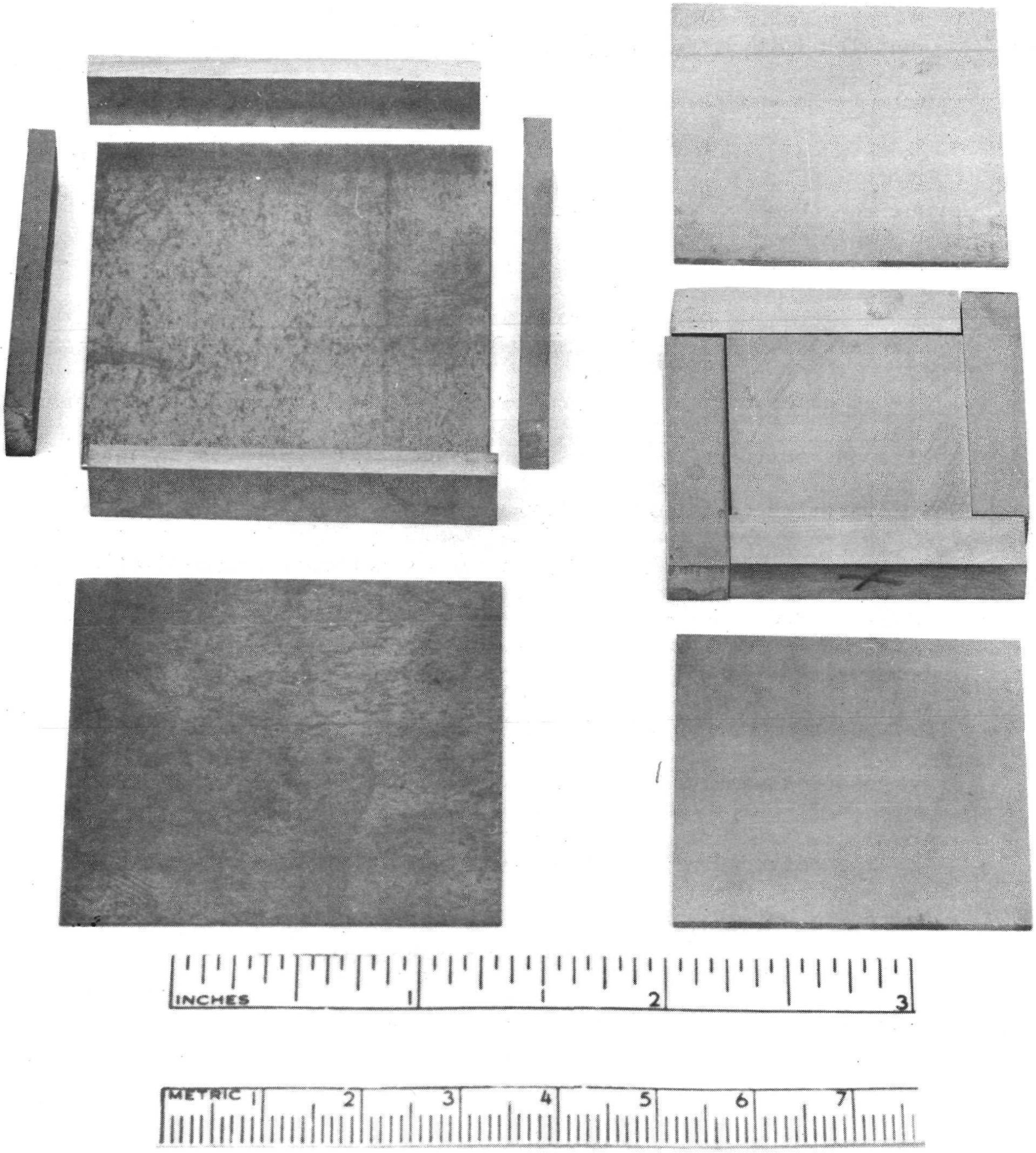


Figure 9 - Chromium composite assembly (right) with high strength chromium alloy core plate, frame pieces and cladding sheets of MP-24. For hot gas pressure bonding the composite will be sealed in the molybdenum container (left). (Neg. P71-1-3)

It was necessary to use an overcladding of mild steel around the molybdenum container. Final closure was made in vacuum by EB welding. The five composites were successfully sealed in mild steel in this manner while the remaining four composite assemblies were set aside to await results of the first hot gas pressure bonding run.

The steel-clad composites were loaded in the autoclave with three W vs. W-25Re thermocouples alongside the billets to monitor the temperature. The composites were heated to 1048°K (1427°F) before pressurizing the vessel. As the pressure was increased, the top plug overheated and the O-ring seal failed when the pressure reached 53 MN/m^2 (7700 psi) causing the autoclave run to be aborted. During this run the composites were at temperatures greater than 1403°K (2066°F) for 7.2 ks (2 hours) and for 1.8 ks (30 minutes) of this period the composite temperature was 1523°K (2282°F).* The chromium composites were undamaged except for a gas leak that was detected in one of the composites. The leak was in a fold of the steel overcladding and was readily repaired by GTA welding.

The five composites were rerun in the autoclave, holding at 1523°K (2282°F) for 7.2 ks (2 hours) at a helium pressure of 57.5 MN/m^2 (8200 psi). The composites were cooled overnight under pressure. Upon removal from the autoclave, the five composites showed considerable deformation with the outline of the frame components clearly visible. Inspection for leaks by helium mass spectrometer techniques revealed small gas leaks in the steel cladding of all composites; it was assumed

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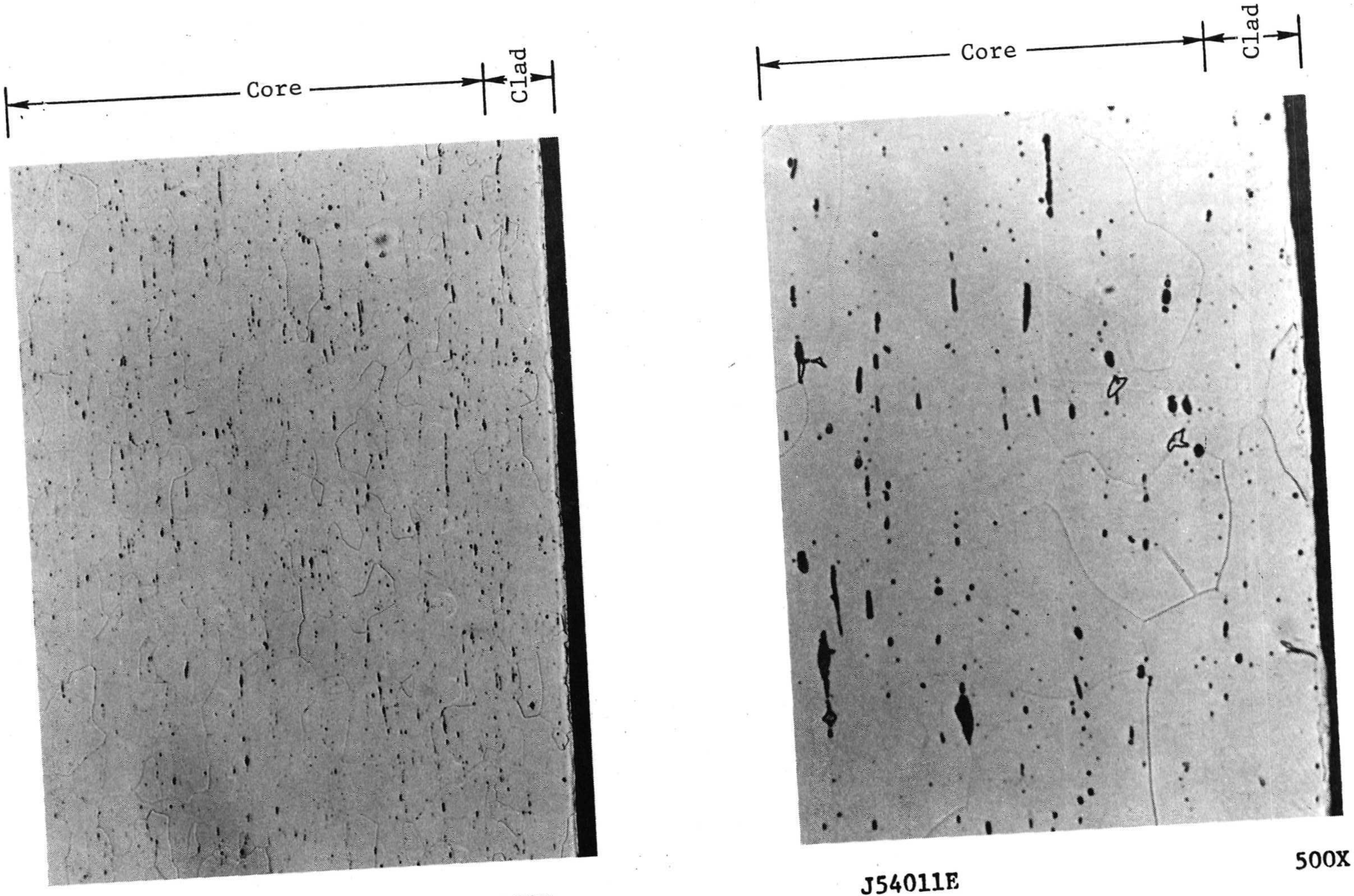
*The temperature for the hot gas pressure bonding operation was selected to coincide with final rolling temperature of the Cr-Mo-Ta-Y-C core alloy processed under Contract NAS 3-9417.

the leaks occurred during cooling in the autoclave since the observed deformation would not have been possible if gas leaks had occurred prior to pressurizing the vessel.

Inspection for bonding by ultrasonic techniques revealed indications of unbonded interfaces in all five composites but it could not be determined if the lack of bonding occurred at the steel-to-molybdenum, molybdenum-to-chromium or chromium-to-chromium interface. Therefore, one composite was stripped of the steel and molybdenum overcladding by leaching in nitric acid. This exposed the chromium composite consisting of 1.20-mm-thick cladding plates of MP-24 (Cr-Y-Hf-Th) over a 6.30-mm-thick core of Cr-M-Ta-C-Y alloy enclosed in the Cr-Y-Hf-Th alloy framing material. Ultrasonic inspection of the bare chromium composite revealed no indications of unbonded areas. Using the bare composite as a standard for the ultrasonic inspection of the four other composites, it was established that the unbonded areas in the composites occurred between the steel and the molybdenum. Since this presented no problem in subsequent hot rolling of the composites, the four composites were accepted for rolling to sheet. The fifth composite (No. 9) was stripped and sectioned for metallographic examination of the core-to-cladding bond and for microhardness measurements at the interfaces. Micrographs of the Cr-Y-Hf-Th alloy clad, the Cr-Mo-Ta-C-Y alloy core and the clad/core interface are shown in Figures 10 through 12. As can be seen in Figure 12, an excellent bond was achieved under the conditions selected for the hot gas pressure bonding operation (1523°K/57.5 MN/m^2). A microhardness traverse across the clad/core material interface was made and the data presented in Table 9. The microhardness at the interface is intermediate between the hardness of the core and clad as would be

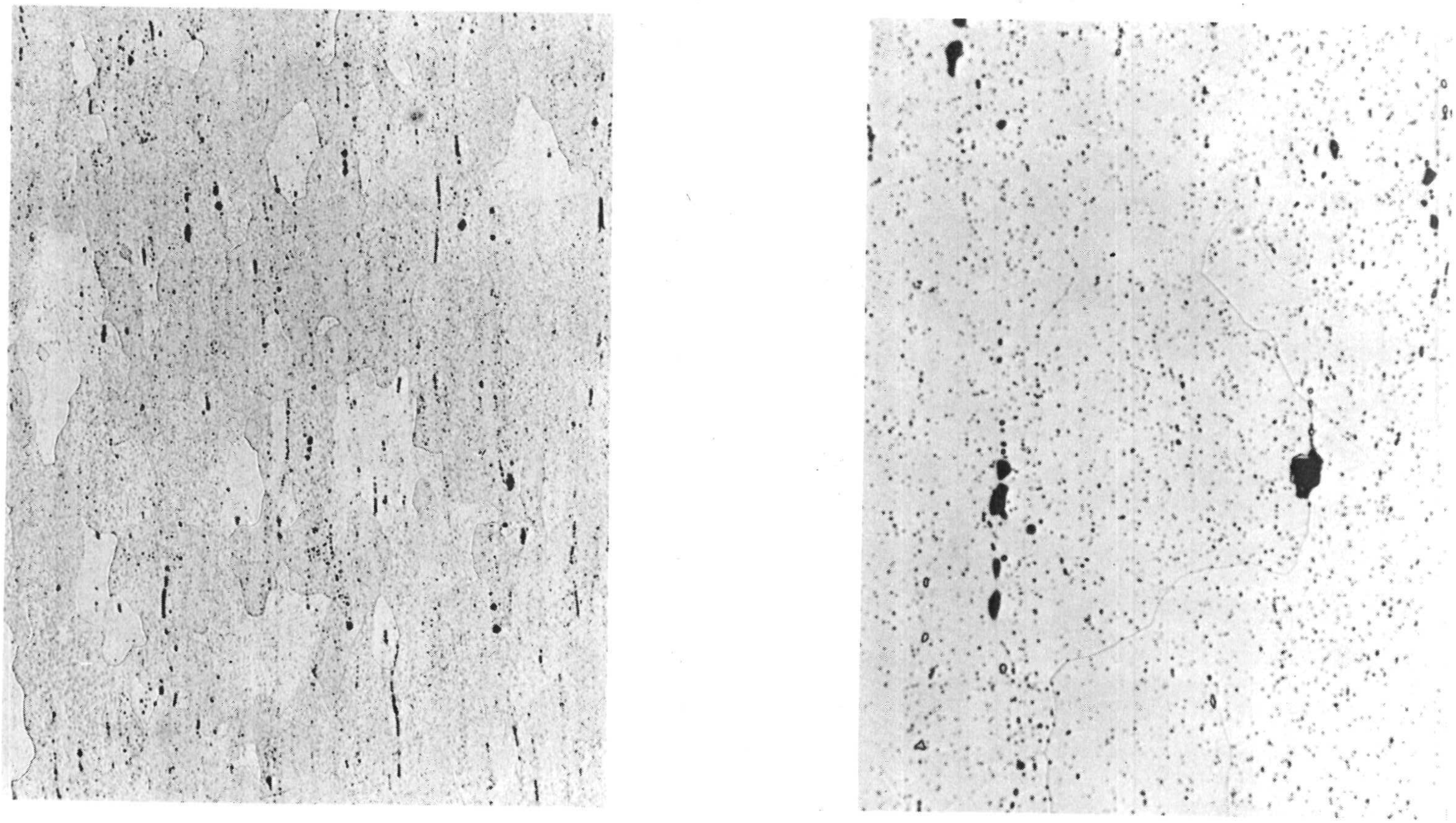


J54011B 100X

J54011E 500X

Figure 10. Microstructure of Cr-Y-Hf-Th alloy clad (Composite No. 9) after hot gas pressure bonding for 7.2 ks(2 hours) at 1523°K (2282°F) and 57.5 MN/m^2 (8200 psi).

Etchant: Electrolytic, 22% H_2SO_4; 12% H_2O_2; 66% H_2O

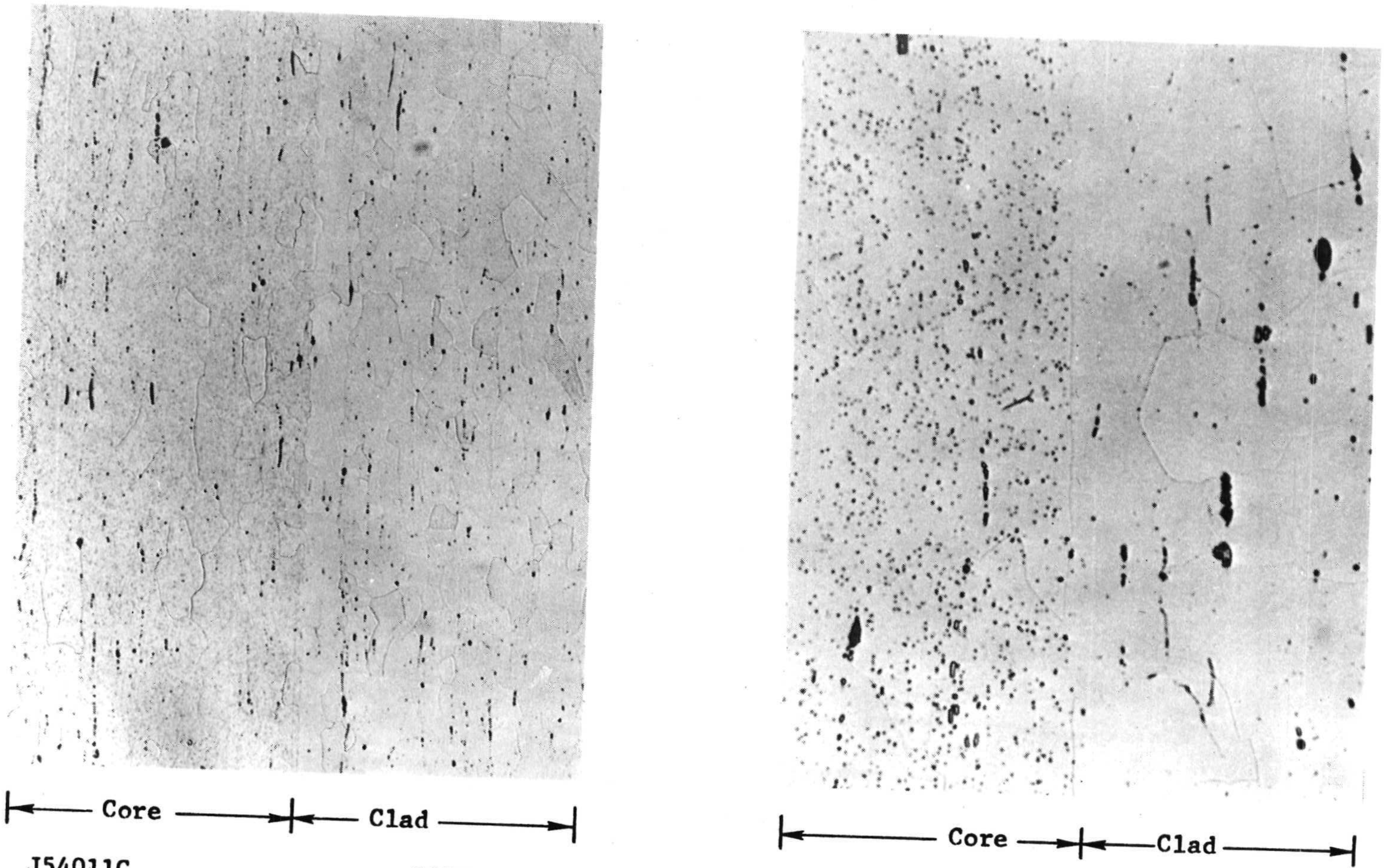


J54011D 100X

J54011G 500X

Figure 11. Microstructure of Cr-Mo-Ta-C-Y alloy core (Composite No. 9) after hot gas pressure bonding for 7.2 ks (2 hours) at 1523°K (2282°F) and 57.5 MN/m^2 (8200 psi).

Etchant: Electrolytic, 22% H_2SO_4; 12% H_2O_2; 66% H_2O



J54011C 100X

J54011F 500X

Figure 12. Microstructure of Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-C-Y alloy core interface (Composite No. 9) after hot gas pressure bonding for 7.2 ks (2 hours) at 1523°K (2282°F) and 57.5 MN/m^2 (8200 psi).

Etchant: Electrolytic, 22% H_2SO_4; 12% H_2O_2; 66% H_2O

TABLE 9

MICROHARDNESS MEASUREMENTS OF HOT GAS PRESSURE BONDED[a]

Cr-Y-Hf-Th CLAD Cr-Mo-Ta-C-Y CORE

Location	Distance from Interface, mm(in.)	Hardness, HV_{100}
Cr-Y-Hf-Th Clad	0.64 (0.025)	131
	0.20 (0.008)	131
	0.15 (0.006)	128
	0.10 (0.004)	134
	0.05 (0.002)	125
Clad/Core Interface	-	209
Cr-Mo-Ta-C-Y Core	0.05 (0.002)	310
	0.10 (0.004)	286
	0.15 (0.006)	317
	0.20 (0.008)	321
	0.64 (0.025)	312

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[a] Bonded at 1523°K (2282°F), 7.2 ks (2 hours), 57.5 MN/m^2 (8200 psi).

expected from alloy depletion of the core material alloying elements tantalum and molybdenum.

Because of the excellent results of the hot gas pressure bonding at 1523°K (2282°F), it was decided to bond the remaining four composites at the same temperature, 1523°K. The molybdenum jacket assembly was clamped around the chromium composite assembly and the molybdenum was tack welded by GTA welding in a vacuum purged, argon-filled chamber. Finally, an overcladding of mild steel was fabricated around the molybdenum container. Final closure was made in vacuum by EB welding. Leak testing using a helium mass spectrometer showed the composites to be leak tight.

The steel-clad composites were loaded in the autoclave with three W vs. W-25Re thermocouples alongside the billets to monitor the temperature. The composites were heated to 1048°K (1427°F) before pressurizing the vessel with helium. After the pressure was stabilized at 57.5 MN/m^2 (8200 psi), the temperature was raised to 1523°K (2282°F) and held for 7.2 ks (2 hours). The composites were cooled overnight under pressure. Upon removal from the autoclave, two of the four composites (Nos. 5 and 7) showed considerable deformation with the outline of the frame components clearly visible; the remaining two composites had not bonded due to apparent rupture of the steel overclad. Using a previously bonded composite (No. 9) which had been stripped as a standard, the two composites which appeared to be bonded were ultrasonically inspected and the bond was found to be sound.

B. Composite Sheet Fabrication Development

Based on the rolling schedules that were employed to produce 1.58 mm (0.062 in.) sheet of Cr-Mo-Ta-C-Y alloy(13) two trial rolling schedules were selected for the rolling of Cr-Y-Hf-Th clad composites. The rolling schedules selected were:

Composite	Rolling Schedule
7	Roll 75% reduction to 2.13 mm (0.084 in.) @ 1523°K(2282°F)
	Rx 1623°K (2462°F)
	Roll 25% to 1.60 mm (0.063 in.) @ 1473°K (2192°F)
	Anneal 1473°K (2192°F)
	Roll 44% to 0.89 mm (0.035 in.) @ 1473°K (2192°F)
5	Roll 75% reduction 2.13 mm (0.084 in.) @ 1523°K(2282°F)
	Rx 1623°K (2462°F)
	Roll 25% to 1.60 mm (0.063 in.) @ 1523°K (2282°F)
	Anneal 1523°K (2282°F)
	Roll 44% to 0.89 mm (0.035 in.) @ 1523°K (2282°F)

Composite No. 5 was rolled to an estimated 2.13 mm (0.084 in.) thickness of the chromium composite and an overall thickness of 3.05 mm (0.120 in.) in 14 passes (10% reduction per pass). Radiographic examination at this point revealed no cracks in the core material. The sheet was placed in an air furnace and the temperature raised from RT to 1073°K (1472°F). Then the sheet was transferred to the hydrogen annealing furnace which was set at 1623°K (2462°F) and held for 3.6 ks (1 hour). After the 3.6 ks (1 hour) annealing treatment, the sheet was transferred back to the air furnace which was set at 1073°K (1472°F) and allowed to furnace cool to RT. All subsequent heating and cooling of the composite sheets from or to RT for rolling or annealing were carried out in this manner.

Final rolling of composite sheet No. 5 was carried out at 1523°K (2282°F) in 12 passes with an intermediate anneal at 1523°K between the fourth and fifth passes. Cracking was observed in the center of the sheet on the seventh pass. The sheet was cut in two pieces after the seventh pass, the cracked portions removed, reclad with a double thickness of 0.076 mm (0.003 in.) Mo and rolled to final size at 1523°K.

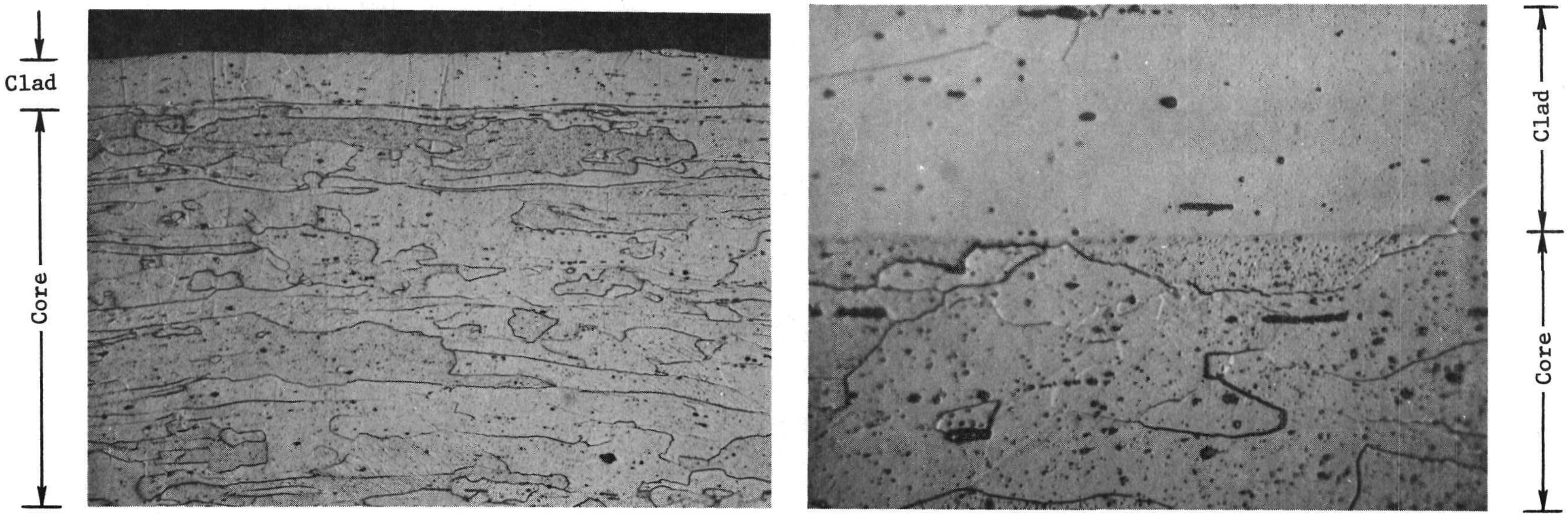
Composite No. 7 was processed to an estimated 2.13 mm (0.084 in.) thickness of the chromium composite and given an intermediate anneal at 1623°K (2462°F) in the same manner as Composite No. 5. Final rolling to size was carried out at 1473°K (2192°F) and 11 passes were required to reach the required thickness. An intermediate 3.6 ks (1 hour) anneal at 1473°K was inserted in the schedule between the fourth and fifth passes.

Both sheets were given a final 1.8 ks (1/2 hour) anneal at the final rolling temperature: 1523°K (2282°F) for composite No. 5 and 1473°K (2192°F) for composite No. 7. After stripping off the steel and molybdenum clad in an acid solution (50% HNO_3), the thickness of the sheet was 0.96 mm (0.038 in.) for composite sheet No. 5 and 0.89 mm (0.035 in.) for composite sheet No. 7. Radiographic examination of the sheets revealed numerous cracks in the core of composite No. 5 and no cracks in composite No. 7. From this basis, it was decided to roll the remaining composite sheets according to the rolling schedule utilized for composite No. 7 (1473°K [2192°F] final rolling temperature).

C. Composite Sheet Evaluation

Specimens of the 0.89 mm (0.035 in.) thick Cr-Y-Hf-Th clad Cr-Mo-Ta-Y-C alloy rolled and annealed [1473°K (2192°F)] sheet (Composite No. 7) were mounted and polished for examination of the microstructure. The rolling schedule used for the processing of composite No. 7 was described in the previous section and as stated is the same schedule that was utilized for the processing of the composites for evaluation in Phase III- Air Exposure and Evaluation of Composite Sheet. Photomicrographs of the clad and clad/core interface are presented in Figure 13. The Cr-Y-Hf-Th alloy cladding is on the order of 0.076 mm (0.003 in.) thick and is completely recrystallized with many grain boundaries normal to the surface. The Cr-Ta-Mo-Y-C alloy core is partially recrystallized with elongated grains. Microhardness measurements made in the center of the cladding and core indicate hardness levels of 144 HV_{100} and 302 HV_{100} respectively.

The bend DBTT of the rolled and annealed composite sheet (No. 7) was determined using seven specimens cut from the 0.89 mm thick composite sheet. To avoid cracking, the chromium alloy sheet was adhesively bonded to a graphite block and cutting was done in a longitudinal direction using an overhead wheel of silicon carbide on a precision wafering machine. The specimens were cut oversize and the edges were ground back to remove any cracks generated in cutting. The surfaces were hand polished through 600 grit paper and all specimens were annealed in hydrogen at $1366^{o}K$ ($2000^{o}F$) for 3.6 ks (1 hour). Inspection by fluorescent penetrant techniques was performed to insure freedom from surface cracks and all specimens were electropolished to remove surface scratches. Electropolishing was done in a solution of 39% sulfuric acid, 37% ethyl alcohol and 24% water at room temperature, using platinum electrodes and fixtures. Treatments of each specimen at 0.8 amp/5.5 volts for 480 s (8 minutes) was sufficient to remove 0.0127 mm (0.0005 in.) per side and eliminate all scratches from the polished surfaces. The nominal thickness of the finished specimens averaged 0.86 mm (0.034 in.).



K16013A 100X

K16013B 500X

Figure 13. Microstructure of Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-Y-C alloy composite sheet (No. 7). The 0.89 mm (0.035 in.) thick sheet was warm rolled and annealed at 1473°K (2192°F).

Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2,66% H_2O

Bend tests were performed in accordance with Materials Advisory Board Report MAB 216-M, "Evaluation Test Methods for Refractory Metal Sheet Materials," with the following exceptions: the specimen width was 7.62 mm (0.3 in.), the length was 38.1 mm (1.5 in.) and the test span was 19.1 mm (0.75 in.).

The bend tests were conducted in a Baldwin Testing Machine equipped to record load and deflection. A punch with a 3.55 mm (0.14 in.) radius was used to make approximately a 4T bend in the chromium alloy specimens. The test specimens and fixtures were heated in an evacuated and helium backfilled retort to prevent oxidation of the specimen during the test; the temperatures were controlled by a thermocouple in contact with the specimen. In accordance with MAB 216-M, a crosshead speed of 0.42 mm/s (1.0 in. per minute) was used.

The data for the series of bend tests are given in Table 10. The load and deflection were read from the recorder of the Baldwin Testing Machine. The bend angle was measured after each test using a calibrated protractor; in addition, the angle was calculated by graphical means using the measured deflection value.

The DBTT for the Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-C-Y alloy core as determined from a graphical plot of the bend angle versus test temperature is shown in Figure 14. From this plot, the DBTT for a full bend of 90 degrees is <713°K (824°F), >700°K (800°F). The DBTT for a 45° bend and 10° bend (nil ductility) was not determined because of insufficient number of specimens. However, the one specimen that was tested at 590°K (603°F) sustained a bend angle of 46° (calculated) prior to fracture. It should be pointed out that the initial test that was made with an as-cut specimen

TABLE 10

BEND TEST[a] DATA FOR DETERMINATION OF DBTT FOR COMPOSITE CHROMIUM ALLOY SHEET NO. 7

	Thickness		Width		Temperature		Max. Load		Deflection		Bend Angle	
Specimen	mm	in.	mm	in.	°K	°F	N	lb	mm	in.	°Meas	°Calc
7-1	0.970	0.0382	8.85	0.348	700	801	262.0	59.0	8.87	0.349[b]	90	85
7-2	0.873	0.0343	7.87	0.310	590	603	168.0	37.8	3.81	0.150	Broke	46
7-3	0.844	0.0332	7.54	0.297	700	800	164.5	37.0	5.08	0.200	Broke	58
7-4	0.866	0.0341	8.05	0.317	727	850	197.8	44.5	8.86	0.345	90[c]	84
7-5	0.864	0.0340	7.80	0.307	713	824	194.6	43.8	9.23	0.363	90[c]	88
7-6	0.856	0.0337	7.73	0.304	700	800	166.2	37.4	4.65	0.183	Broke	54
7-7	0.851	0.0335	7.75	0.305	716	829	177.8	40.0	10.24	0.403	93	92

---

[a] All tests conducted in helium atmosphere.
Specimen size 7.62 mm x 38.1 mm (0.3 in. x 1.5 in.).
Fixture Span 19.0 mm (0.75 in.).
Crosshead speed 0.42 mm/s (1.0 in./min) per MAB 216-M.
Bend Radius 4T = 7.11 mm (0.280 in.).

[b] Crosshead speed 0.21 mm/s (0.5 in./min); specimen as rough cut, unpolished and unannealed.

[c] Specimen broken during removal

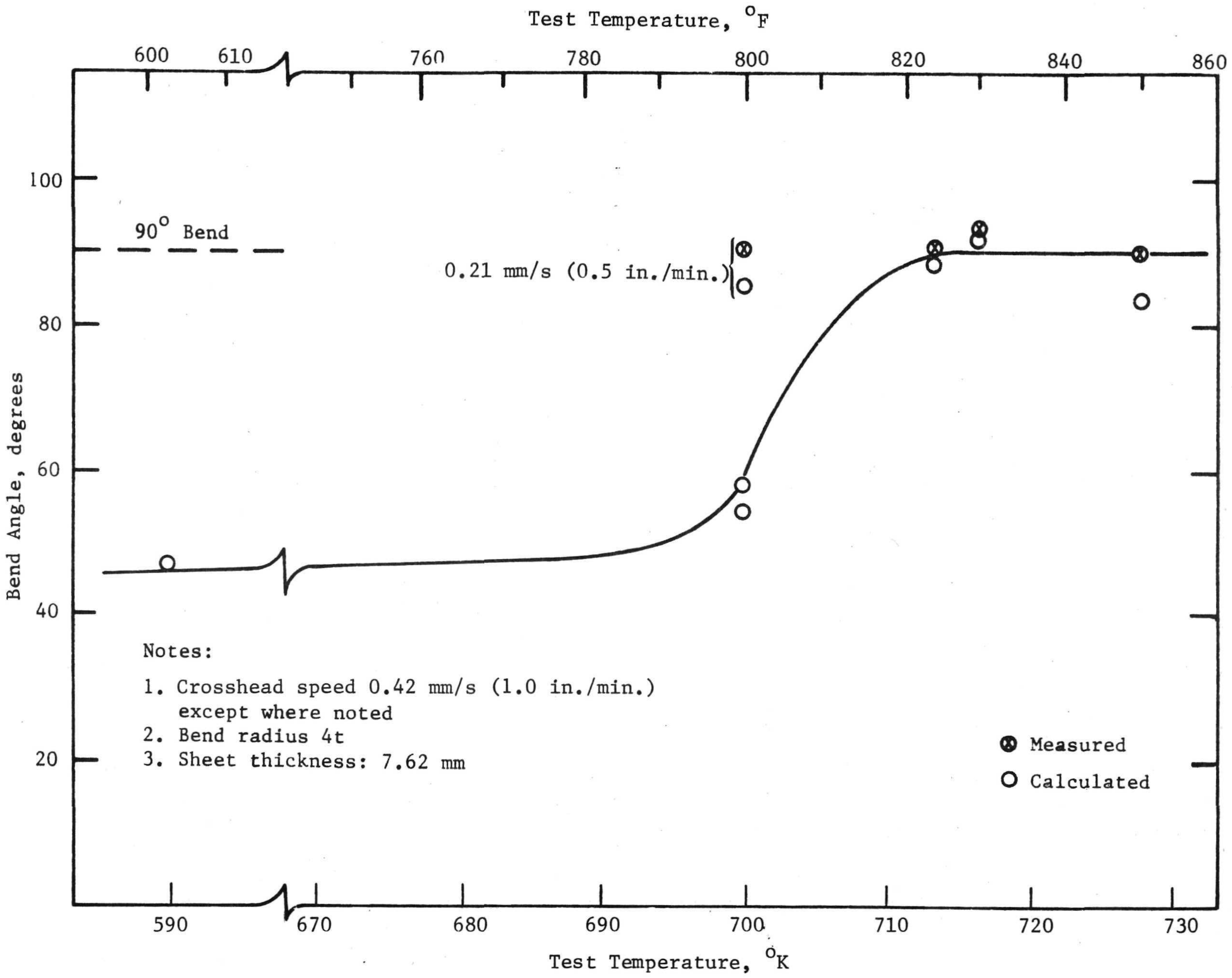
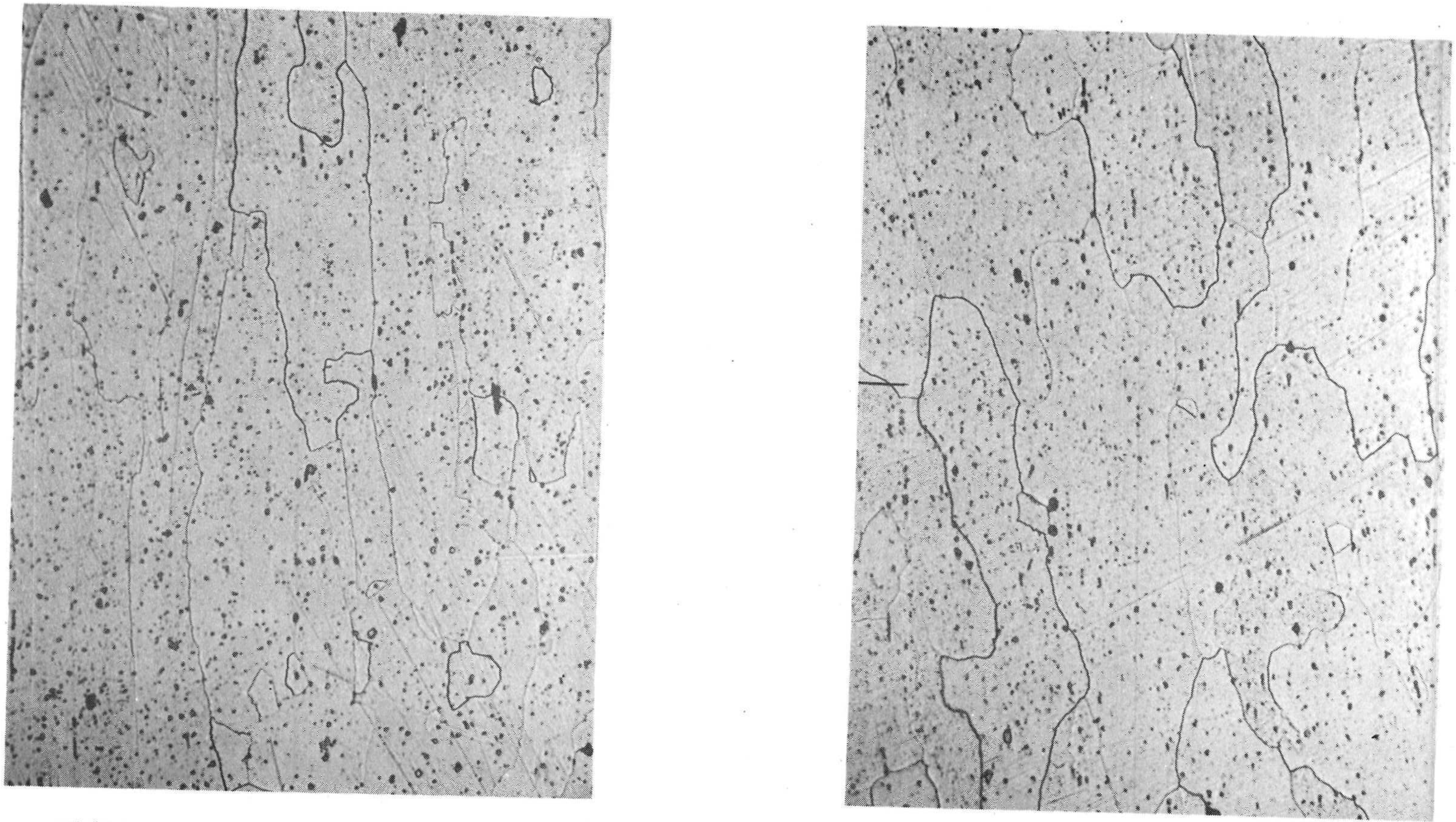


Figure 14. Bend Angle as a Function of Temperature for Composite Chromium Alloy Sheet No. 7 Showing DBTT as <713°K (824°F) and >703°K (806°F) for 90 Degree Bend.

and without any special surface preparation but with a slower ram speed 0.21 mm/s (0.5 in/min.) resulted in a full 90° bend at 700°K (800°F). Also examination of the microstructure of the Cr-Mo-Ta-Y-C core alloy in the specimens which were ground, polished and annealed at 1366°K (2000°F) after cutting revealed a more equiaxed grain structure in comparison to the structure of the core alloy in the as-processed specimens, Figure 15. This combination of the lower strain rate and elongated grain structure would result in a lower bend DBTT.



K16011A A

K16031A B

Figure 15. (A) Microstructure of as-rolled and annealed 1473°K (2192°F) Cr-Mo-Ta-Y-C alloy core (Composite No. 7).

(B) Same as (A) plus 3.6 ks (1 hour) anneal at 1366°K (2000°F).

Longitudinal Section; magnification 250X.

Etchant: 22% H_2SO_4, 12% H_2O_2, 66% H_2O

V. AIR EXPOSURE AND EVALUATION OF COMPOSITE SHEET

A. Composite Sheet Fabrication

The four Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-Y-C alloy composites (Nos. 1, 2, 3 and 6) which previously had been diffusion bonded at 1523°K (2282°F) by hot gas pressure bonding techniques (see page 48) were rolled to 0.89 mm (0.035 in.) thick sheet according to the rolling schedule utilized for composite No. 7(1473°K [2192°F] final rolling temperature) in Phase II(See page 50). Prior to rolling, the integrity of the bond was established by ultrasonic inspection and was found to be good.

The four composites were rolled to an estimated 2.13 mm (0.084 in.) thickness, 3.05 mm (0.120 in.) overall, in 12 passes (~10% reduction per pass). Radiographic examination at this point revealed no cracks in the core material. The sheet was placed in an air furnace and the furnace temperature was raised from RT to 1073°K (1472°F). Then the sheet was transferred to the hydrogen annealing furnace which was set at 1623°K (2462°F) and held for 1.8 ks (30 minutes). After the annealing treatment, the sheet was transferred back to the air furnace which was set at 1073°K and allowed to furnace cool to RT. All heating and cooling of the composite sheets from or to RT for rolling or annealing were carried out in this manner.

Final rolling of the composite sheets were carried out at 1373°K (2192°F) in 10 passes with an intermediate anneal at 1373°K between the fourth and fifth passes. The sheets were given a final 1.8 ks (30 minute) anneal at 1373°K. Radiographic examination of the sheets revealed no cracks in sheets Nos. 1, 2 and 6, while sheet No. 3 had a small edge crack near the midpoint. The steel and molybdenum overcladding was stripped in an acid solution (50% HNO_3) and the composite sheets prepared for the air exposures.

B. Air Exposures

Allocation of the composite sheets for the air exposure was made according to the following schedule:

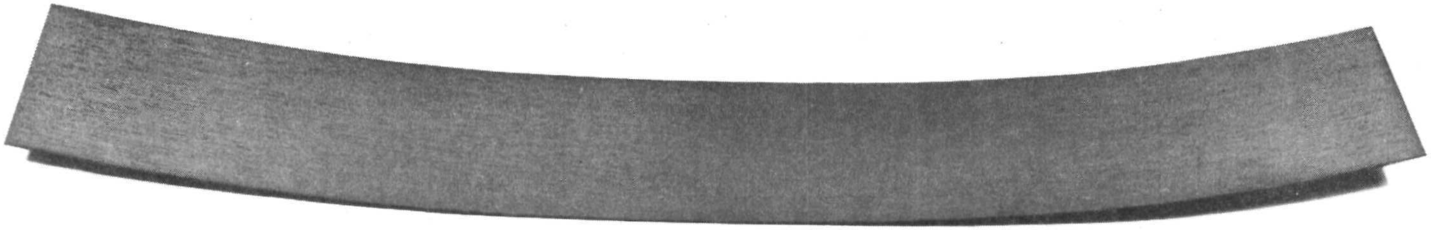
Composite No.	Temp., °K (°F)	Time, ks (hours)
3	1422 (2100)	720 (200)
2	1422 (2100)	1,800 (500)
1	1589 (2400)	72 (20)
6	1589 (2400)	360 (100)

The composite sheets were placed on edge in an Al_2O_3 boat and inserted in a SiC Glo-Bar heated muffle furnace in which slowly moving air at a measured rate of 3.93×10^{-5} m^3/s (∿5 cfh) could be passed over the sheets. Composite sheet No. 2 was inserted into the furnace first and slowly heated to 1422°K (2100°F). At the end of 360 ks (100 hours), the furnace was allowed to slowly cool to room temperature at which time the sheet was visually inspected and found to be in good condition. Composite sheet No. 2 was reinserted in the cold furnace along with composite sheet No. 3 and again the furnace was slowly heated to 1422°K (2100°F). Composite sheet No. 3 was removed after 720 ks (200 hours) and composite sheet No. 2 after a total exposure of 1.8 Mg (500 hours). After completion of the 1422°K (2100°F) exposures, composite No. 1 was inserted in the furnace and slowly heated to 1589°K (2400°F). At the end of 14.4 ks (4 hours) the furnace was allowed to slowly cool to room temperature in order to permit visual inspection of the surfaces of the sheet. The surfaces were found to be in good condition and the sheet was reinserted in the furnace along with composite sheet No. 6 and again the furnace was slowly heated to 1589°K. Composite sheet No. 1 was removed after a total exposure of 72 ks (20 hours) and composite No. 6 after a total exposure of 360 ks (100 hours).

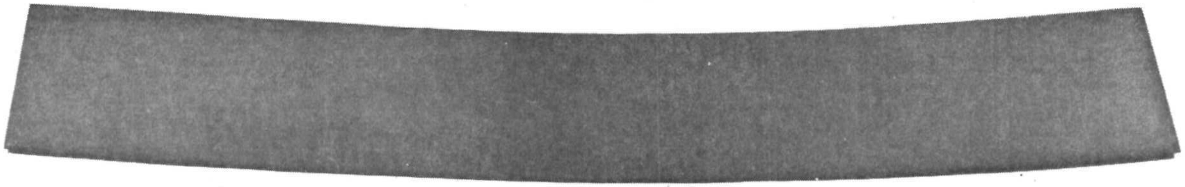
C. Evaluation of Composite Sheet Exposed to Air at 1422°K (2100°F) and 1589°K (2400°F)

A photograph showing the appearance of the four composite sheets after the air exposure is shown in Figure 16. Visually, the appearance of the scale on the surface of the composite sheet was excellent. Little or no spalling was observed on any of the sheets with the exception of the edges of composite No. 6 which was exposed for 360 ks (100 hours) at 1589°K (2400°F).

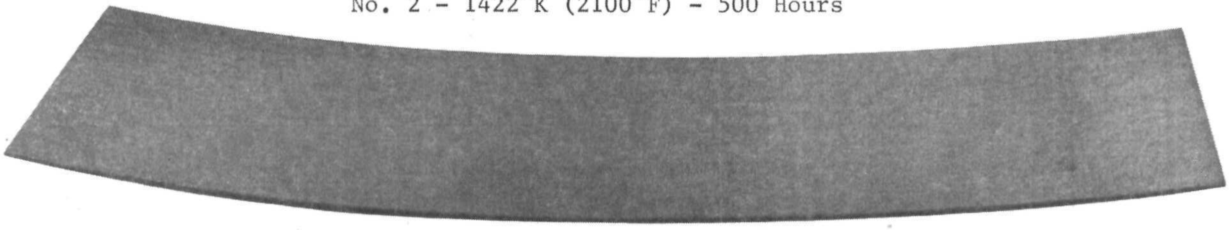
Samples were cut from each of the exposed composite sheets and examined metallographically for changes in the microstructure of the Cr-Y-Hf-Th cladding alloy and the Cr-Mo-Ta-Y-C core alloy. Photomicrographs are shown in Figures 17-20. Gross intergranular attack was observed to a depth of up to 0.0127 mm (0.0005 in.) in the sheet exposed to air at 1422°K (2100°F) and up to 0.0254 mm (0.001 in.) in the sheet exposed at 1589°K (2400°F). What appears to be nitrogen penetration and formation of a nitride was isolated within the grain boundaries of the Cr-Y-Hf-Th alloy cladding and was observed to a depth of 0.051 to 0.076 mm (0.002-0.003 in.) in the sheet exposed for 720 ks (200 hours) at 1422°K (2100°F). Complete intergranular penetration was observed in the other composite sheets. The influence of the thermal exposure on the structure of the Cr-Mo-Ta-Y-C core appears to be negligible for the 1422°K (2100°F) treatment. Some grain coarsening and coalescence of the carbide phase



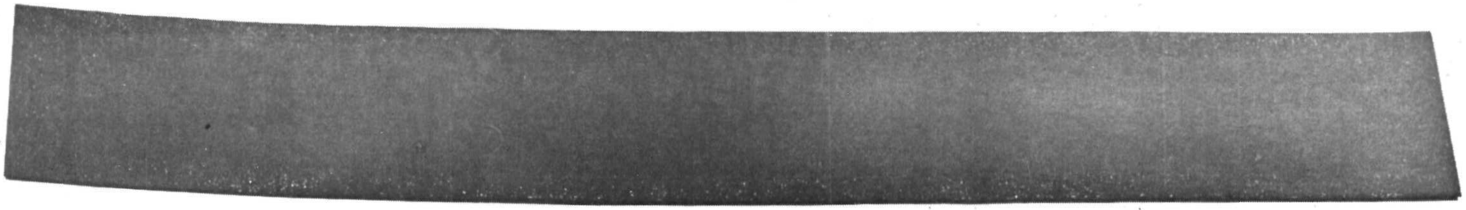
No. 3 - 1422°K (2100°F) - 200 Hours



No. 2 - 1422°K (2100°F) - 500 Hours



No. 1 - 1589°K (2400°F) - 20 Hours



No. 6 - 1589°K (2400°F) - 100 Hours

Figure 16. Surface Appearance of Composite Sheets 1, 2, 3 and 6 After Exposure to Air at 1422°K (2100°F) and 1589°K (2400°F). (Neg. P71-7-3A)

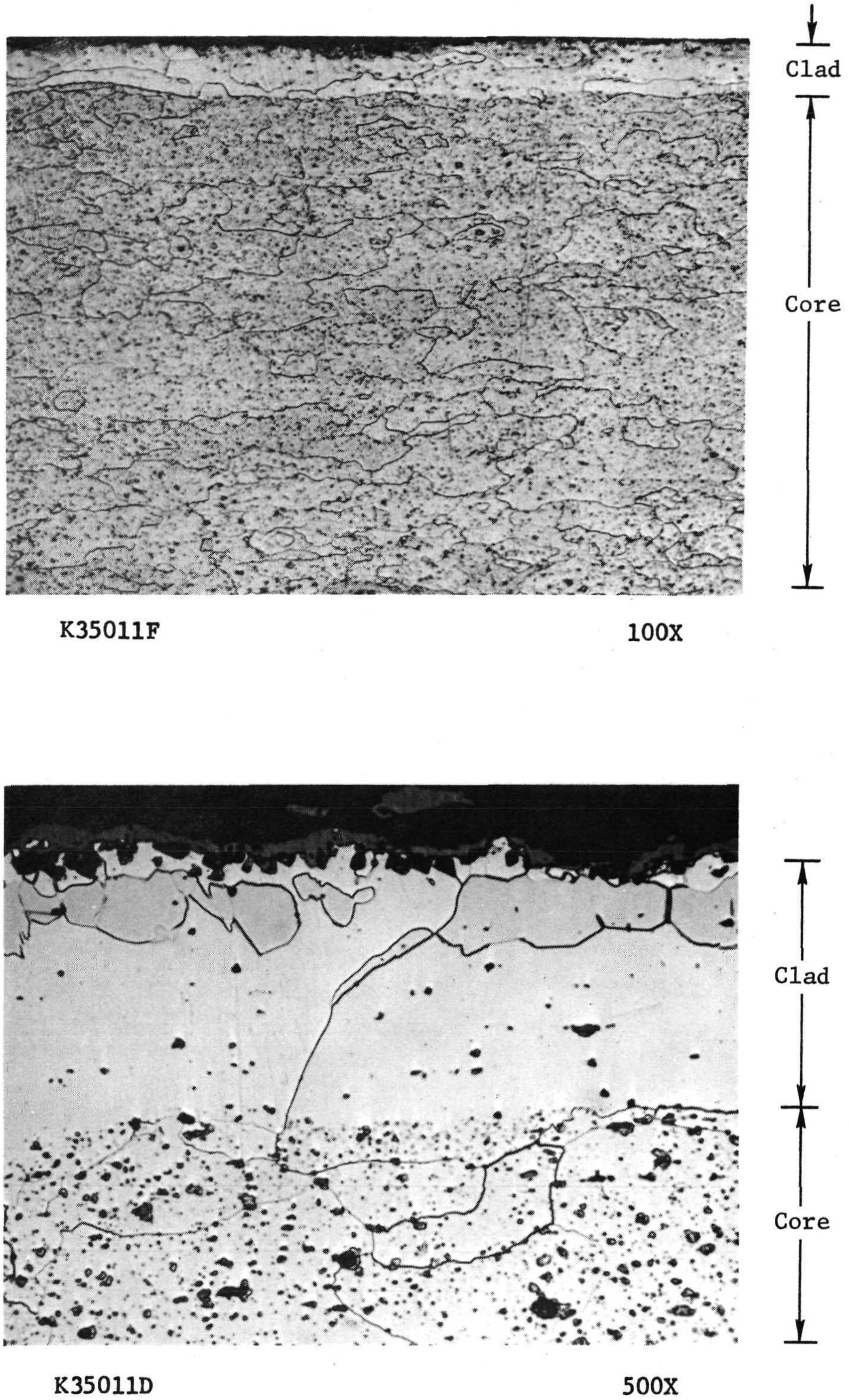


Figure 17. Microstructure of Cr-Y-Hf-Th Alloy Clad Cr-Mo-Ta-Y-C Alloy sheet after exposure to air for 720 ks (200 hours) at 1422°K (2100°F).

Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O.

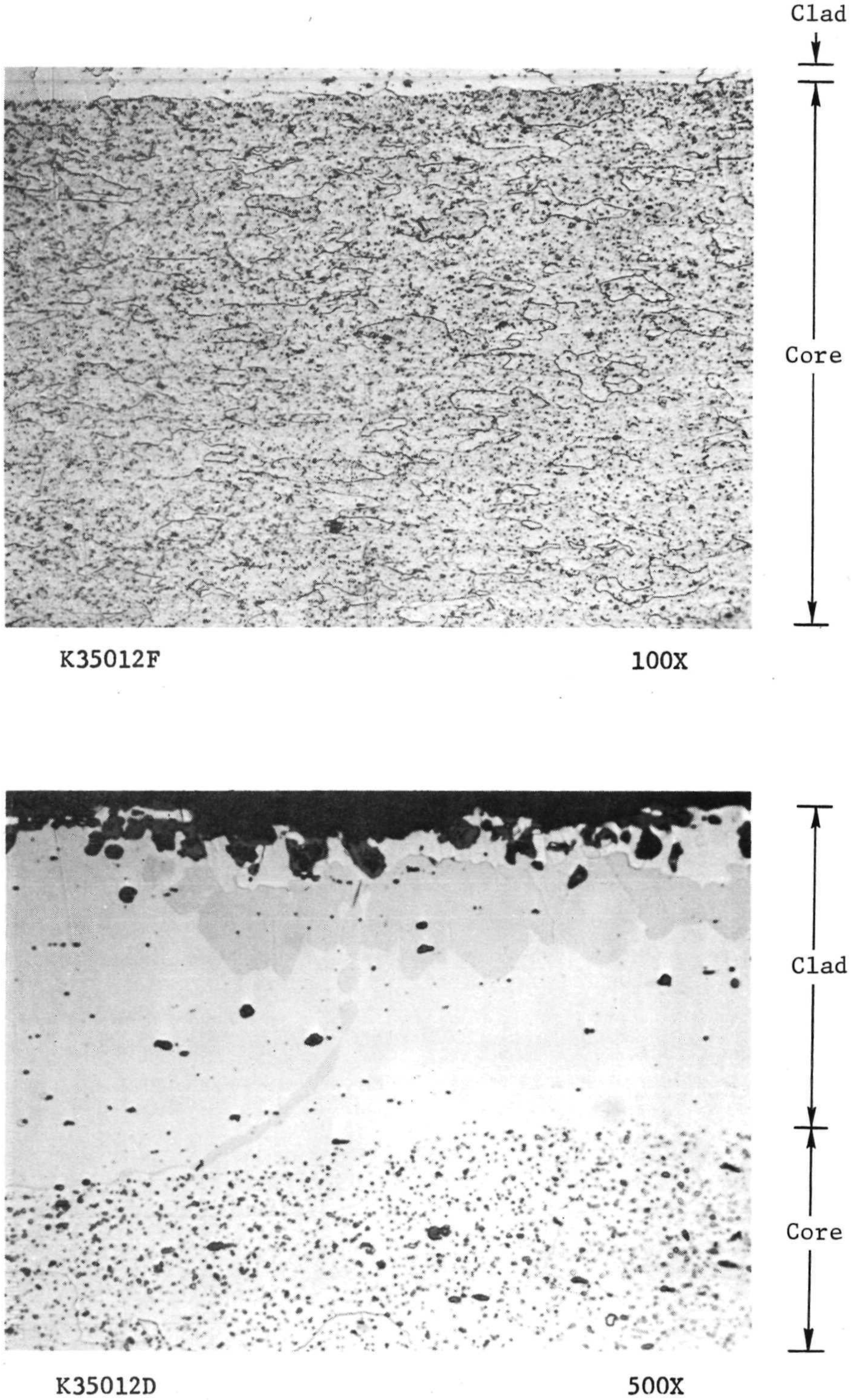
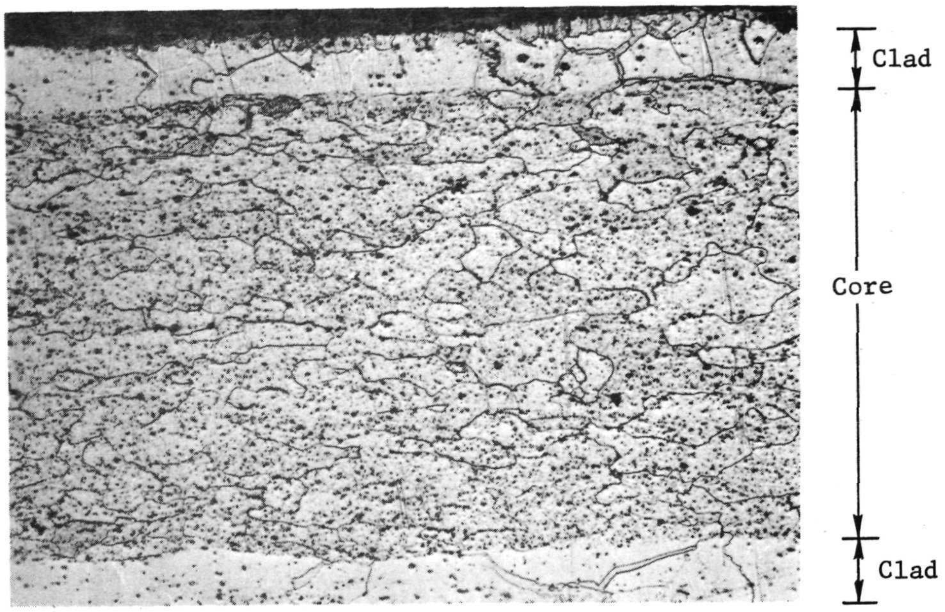
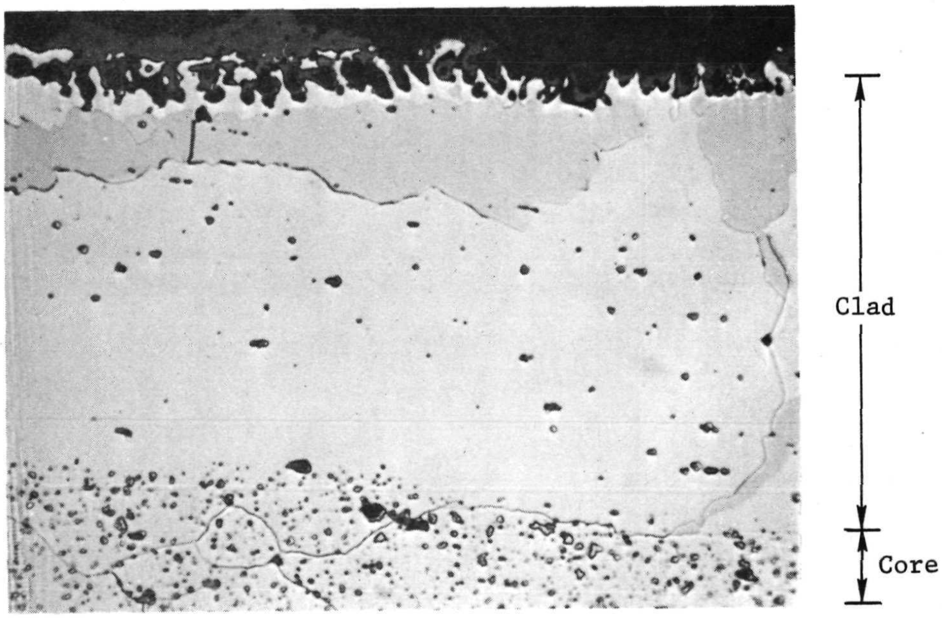


Figure 18. Microstructure of Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-Y-C alloy sheet after exposure to air for 1800 ks (500 hour) at 1422°K (2100°F).

Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O



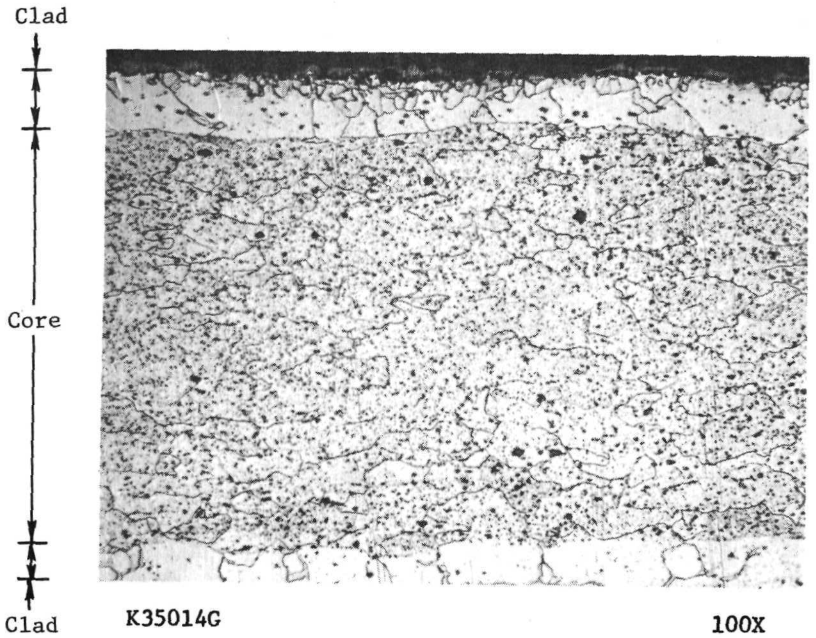
K35013F 100X



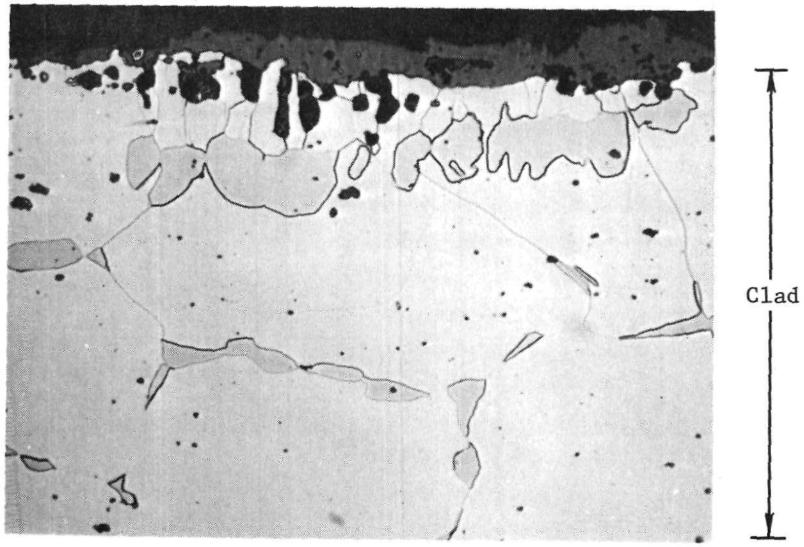
K35013D 500X

Figure 19: Microstructure of Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-Y-C alloy sheet after exposure to air for 72 ks (20 hours) at 1589°K (2400°F).

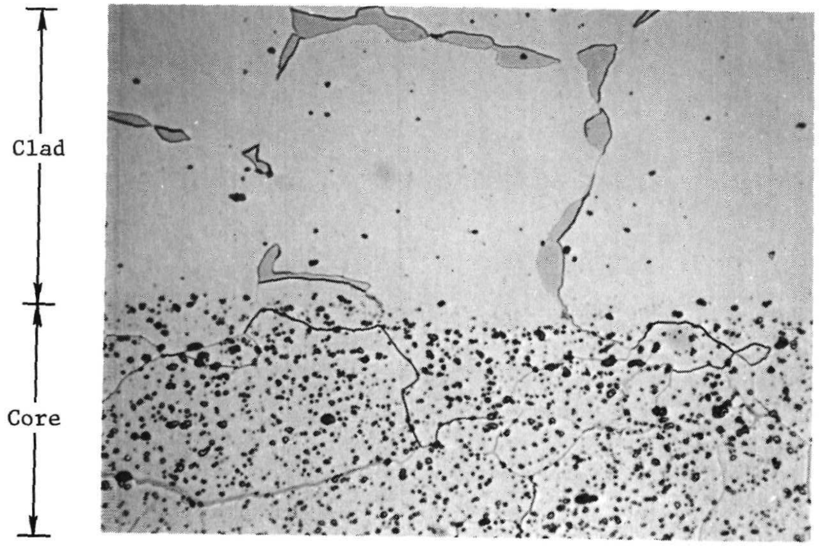
Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O



K35014G 100X



K35014D 500X



K35014E 500X

Figure 20. Microstructure of Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-Y-C alloy sheet after exposure to air for 360 ks (100 hours) at 1589°K (2400°F).

Etchant: Electrolytic, 22% H_2SO_4, 12% H_2O_2, 66% H_2O

has occurred after the exposures at 1589°K (2400°F).

Microhardness measurements of the composite sheets exposed to air were made and the data are reported in Table 11.

The bend ductile-to-brittle transition temperature (DBTT) was determined for the composite sheets 1, 2, 3 and 6 after the exposure to air at 1422°K (2100°F) and 1589°K (2400°F). The preparation of the specimens and test procedure were the same as employed for the determination of the bend DBTT of the as-rolled and annealed composite sheet No. 7 (page 51) except that the oxidized surfaces of the specimens were not disturbed by polishing and the elevated temperature tests were performed in air.

The data for the series of bend tests are given in Table 12. The DBTT for the oxidized Cr-Y-Hf-Th alloy clad Cr-Mo-Ta-C-Y alloy as determined from a graphical plot of the bend angle versus test temperature is shown in Figure 21. From these plots, the DBTT for a full bend of 90 degrees and a bend of 45° for the four composite sheets were determined and are listed in Table 13. It is apparent that there is no detrimental effect on the bend DBTT as a result of air exposure at a temperature of 1422°K (2100°F) for times up to 720 ks (200 hours). However, longer time exposures at 1422°K and exposures at 1589°K (2400°F) adversely affect the bend DBTT.

TABLE 11

MICROHARDNESS OF COMPOSITE SHEETS AFTER AIR EXPOSURE AT 1422°K (2100°F) and 1581°K (2400°F)

		Hardness, HV_{100}			
		Composite No. 3	Composite No. 2	Composite No. 1	Composite No. 6
Location	Distance from Interface, mm(in.)	1422°K (2100°F), 200 Hours	1422°K (2100°F), 500 Hours	1589°K (2400°F), 20 Hours	1589°K (2400°F), 100 Hours
Cr-Y-Hf-Th Clad	Center	176	159	152	153
Cr-Mo-Ta-Y-C Core	0.076 (0.003)	286	387	321	280
	0.153 (0.006)	286	300	334	327
	0.229 (0.009)	280	317	290	286
	0.305 (0.012)	280	295	317	300

TABLE 12

BEND TEST[a] DATA FOR DETERMINATION OF DBTT OF COMPOSITE CHROMIUM ALLOY SHEETS AFTER OXIDATION TESTING[a]

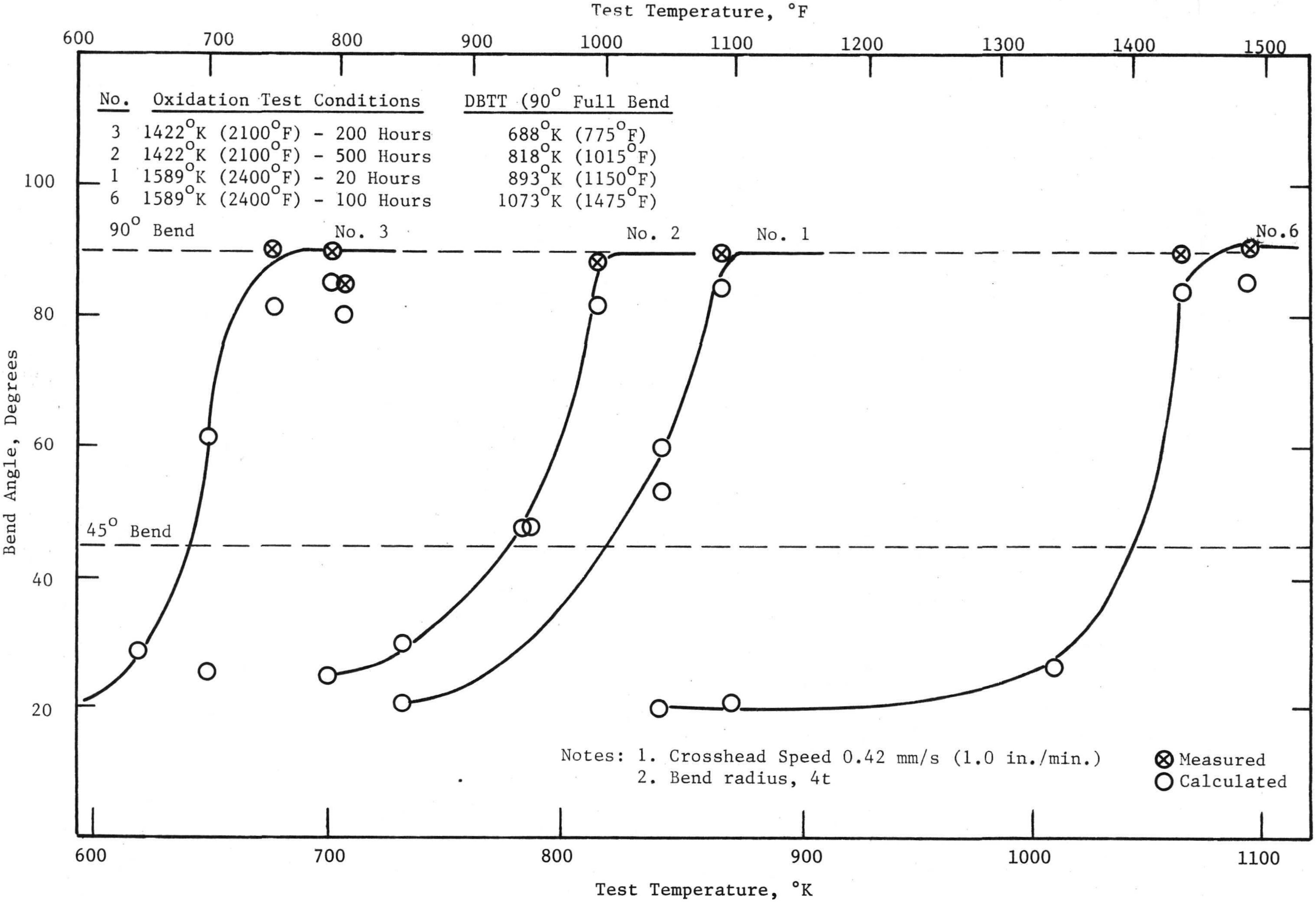
Composite No./ Specimen	Thickness[b]		Test Temperature		Max. Load		Deflection		Bend Angle	
	mm	in.	°K	°F	N	lb	mm	in.	Meas.	Calc.
Oxidized 200 hrs. at 1422°K (2100°F)										
3-1	0.904	0.0356	700	800	153.3	34.5	8.89	0.350	90°	85°
3-2	0.873	0.0344	700	801	128.9	29.0	8.51	0.335	86.5	80
3-3	0.866	0.0341	672	750	128.9	29.0	8.53	0.336	90	82
3-4	0.902	0.0355	616	649	114.6	25.8	2.62	0.103	Broke	29
3-5[c]	0.848	0.0334	644	700	123.5	27.8	6.22	0.245	Broke	61
3-6	0.892	0.0351	644	700	115.6	26.0	2.41	0.095	Broke	26
Oxidized 500 hrs. at 1422°K (2100°F)										
2-1	0.993	0.0391	698	797	177.7	40.0	2.29	0.090	Broke	25
2-2	1.016	0.0400	727	850	186.6	42.0	2.67	0.105	Broke	30
2-3	0.980	0.0386	782	948	202.5	45.6	4.57	0.180	Broke	48
2-4[c]	0.983	0.0387	811	1000	195.5	44.0	8.53	0.336	88	82
2-5[c]	0.991	0.0390	783	950	198.2	44.6	4.57	0.180	Broke	48
Oxidized 20 hrs. at 1589°K (2400°F)										
1-1	0.922	0.0363	702	804	159.1	35.8	3.56	0.140	Broke	38
1-2	0.927	0.0365	727	850	137.7	31.0	2.03	0.080	Broke	21
1-3	0.940	0.0370	864	1097	176.8	39.8	8.79	0.346	90	84
1-4	0.914	0.0360	839	1050	168.8	38.0	5.13	0.202	Broke	54
1-5[c]	0.919	0.0362	839	1050	168.8	38.0	6.10	0.240	Broke	60
Oxidized 100 hrs. at 1589°K (2400°F)										
6-1	0.930	0.0366	701	802	164.4	37.0	2.59	0.102	Broke	29
6-3	0.965	0.0380	837	1046	153.3	34.5	1.78	0.070	Broke	20
6-4	0.953	0.0375	868	1103	162.6	36.6	1.98	0.078	Broke	21
6-5[c]	0.942	0.0371	1005	1350	144.5	32.5	2.41	0.095	Broke	26
6-6[c]	0.973	0.0383	1061	1450	162.2	36.5	8.79	0.346	90	84
6-7[c]	0.968	0.0381	1061	1450	60.4	13.6	0.66	0.026	Broke	<10
6-8[c]	1.031	0.0406	1089	1500	167.0	37.6	9.07	0.357	91	86

---

[a] Specimen Size 7.6 mm x 38.1 mm (0.3 in. x 1.5 in.); Fixture Span: 19.1 mm (0.75 in.); Crosshead speed 0.42 mm/s (1.0 in./mm) (per MAB-216-M); Bend Radius 4T (3.6 mm).

[b] Includes thickness of oxide layer.

[c] Edges handpolished before testing.



Notes: 1. Crosshead Speed 0.42 mm/s (1.0 in./min.)
2. Bend radius, 4t

Figure 21. Bend Angle as a Function of Temperature for Oxidized Composite Chromium Alloy Sheet.

TABLE 13

BEND[a] DBTT Cr-Y-Hf-Th ALLOY CLAD Cr-Mo-Ta-Y-C ALLOY SHEET AFTER EXPOSURE TO AIR AT 1422°K (2100°F) AND 1589°K (2400°F)

	Air Exposure Conditions		DBTT, °K (°F)	
Composite No.	Temp., °K (°F)	Time, ks (Hours)	90°Bend	45° Bend
3	1422 (2100)	720 (200)	688 (775)	636 (685)
2	1422 (2100)	1800 (500)	818 (1015)	772 (930)
1	1589 (2400)	72 (20)	893 (1150)	816 (1010)
6	1589 (2400)	360 (100)	1073 (1475)	1042 (1415)

---

[a] Specimen Size 7.6 mm x 38.1 mm (0.3 in. x 1.5 in.); Fixture Span: 19.1 mm (0.75 in.); Crosshead speed 0.42 mm/s (1.0 in./mm) (per MAB-216-M); Bend Radius 4T (3.6 mm).

VI. CONCLUSIONS

The effectiveness of a dilute nitridation and oxidation resistant Cr-Y-Hf-Th alloy as a protective cladding alloy for a high strength chromium alloy was examined. Conclusions that were generated from this work are:

(1) The dilute Cr-Y-Hf-Th alloy clad surface had little or no effect on the bend DBTT of the Cr-Mo-Ta-Y-C core. It is possible that the irregularities of the clad-core interface may have resulted in a slight increase in the bend DBTT of the Cr-Mo-Ta-Y-C alloy. The DBTT for a full 90° bend of the composite sheet was determined to be 700°-713°K (800°-824°F).

(2) The dilute Cr-Y-Hf-Th alloy was effective in inhibiting nitrogen embrittlement of the Cr-Mo-Ta-Y-C alloy upon exposure to air for up to 720 ks (200 hours) at 1422°K (2100°F). Only partial intergranular penetration [0.051-0.076 mm (0.002-0.003 in.)] of the clad occurred and no increase in the bend DBTT of the composite sheet was observed.

(3) Longer time [1,800 ks (500 hours)] exposures to air at 1422°K (2100°F) or short time [72-360 ks (20-100 hours)] exposure at 1589°K (2400°F) resulted in complete intergranular penetration of the Cr-Y-Hf-Th clad and significant increases in the bend DBTT of the composite sheets. The bend DBTT for the composite sheet exposed to air for 1,800 ks (500 hours) at 1422°K (2100°F) increased to 818°K (1015°F); the bend DBTT for the composite sheets exposed to air for 72 ks (20 hours) and 360 ks (100 hours) at 1589°K (2400°F) increased to 893°K (1150°F) and 1073°K (1475°F) respectively.

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APPENDIX A. - DETAILED PROCESS DESCRIPTION FOR FABRICATION OF CHROMIUM ALLOY PLATE (13)

(Cr-4Mo-0.6Ta-0.06Y-0.4C)

Operation No.	Operation	Description
1	Alloy Consolidation	The chromium alloy was consolidated by the vacuum induction melting process. Approx. 45.4 kg (100 pounds) of alloy was melted in a Y_2O_3 stabilized ZrO_2 crucible using H_2 reduced electrolytic chromium flake.
2	Ingot Conversion	The 45.4 kg ingot was machined and hot extruded to bar at 1753°K (2696°F).
3	Annealing of extrusion	The chromium alloy extrusion was preheated to 1651°F (1173°K). This operation, as well as all other heating operations, was done in a hydrogen atmosphere. The extrusion was then transferred to the annealing furnace and annealed for one hour at 2552°F (1673°K). The extrusion was next returned to the furnace at 1652°F (1173°K) and allowed to cool to that temperature. Finally, the extrusion was cooled to ambient temperature in a hydrogen atmosphere in about 30 minutes.
4	Jacketing of extrusion	The extrusion was band sawed and machined into sections approx. 2.38 in. (6 cm) long. The exposed chromium alloy was wet ground with 180 grit abrasive paper and electroetched. The exposed alloy was inspected for cracks by visual or fluorescent penetrant inspection. The jacket was then assembled around the extrusion and welded.
5	Rolling	The pack, i.e., the assembly of the extrusion and its jacket, was evacuated for 15 minutes by a mechanical vacuum pump and the evacuation tube was sealed. The pack was preheated for 30 minutes at 1652°F (1173°K) and then was heated for 20 minutes before the first rolling pass and for lesser lengths of time before later rolling passes. The minimum heating time was five minutes. The pack was warm rolled the first two rolling passes at 2372°F

APPENDIX A (CONTINUED)

Operation No.	Operation	Description
		(1573°K) to a reduction of 18% per pass at a mill speed of 40 ft/min. (0.203 m/s). The diameter of the rolls was 12 in.(30.5 cm).After the first two rolling passes, the pack was transferred to a furnace at 1652°F (1173°K) so that the temperature of the rolling mill furnace could be changed. The pack was then rolled two passes at 18% reduction each at 2282°F (1523°K). The pack was annealed at 1562°F (1173°K) for 30 minutes, furnace cooled to approx. 1292°F (973°K) and, finally, cooled to ambient temperature buried in insulating material. A rolling lot consisted of from one to nine packs assembled at one time. No more than five packs could be accommodated in the two furnaces used for: a. Preheating and storing at 1652°F (1173°K) b. Heating for rolling and for annealing Thus, rolling and annealing was accomplished on groups of no more than five packs. Packs were heated for rolling individually, while the rest of the group was stored at 1652°F (1173°K). No more than two packs were annealed simultaneously at 2462°F (1623°K).
6	Replenishment of jacketing	The pack was grit blasted and ground to remove scale. An additional sheet of steel, 0.10 in. (0.25 cm) thick, was welded to the top and to the bottom of the pack.
7	Rolling	After being preheated for 20 minutes at 1652°F (1173°K), the pack was rolled to gauge in three passes of about 19% reduction each at 2282°F (1523°K) at a mill speed of 40 ft/min. (0.203 m/s). The pack was stored briefly at 1652°F (1173°K) while a furnace temperature change was being made.

APPENDIX A (CONTINUED)

Operation No.	Operation	Description
8	Annealing	The pack was annealed one hour at 2192°F(1473°K), then held in a furnace at 1652°F (1173°K) for 30 min., furnace cooled to approx. 1292°F(973°K) and, finally, cooled to ambient temperature buried in insulating material.
9	Removal of jacket	Excess steel was sawed from the ends and sides of the pack. The pack was pickled in a mixture of nitric, sulphuric and hydrofluoric acids to remove the steel and molybdenum jacket.
10	Electroetching	The chromium alloy plate was etched anodically in a saturated solution of oxalic acid in water to remov a layer of the alloy, 0.002 in. (0.005 cm) thick, from each surface.
11	Abrasive sawing	The chromium alloy plate was abrasive sawed to the required size, 1.4 in. x 1 in. x 4 in. (0.61 cm x 2.54 cm x 10.16 cm) by plunge cutting in two or three passes.
12	Electroetching	The alloy was electroetched for about 15 seconds.
13	Inspection	The pieces were inspected at low magnification for surface defects.

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