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80 668 Second Progress Report Covering the Pariod September 1 to October 31, 1949 on Research and Development on Titanium Alloys (None) Simmons, O. W.; Greenidge, C. T.; Craighead, C. M; and others Battelle Memorial Institute, Columbus, Ö. 2nd (Same) for AMC, Wright-Patterson Air Force Base, Dayton, O. photos. Oct 49 Unclass. U.S. English tables, diagra, graphs 120 (Same)

Progress is reported in development of titunium alloys. Phase relations in titunium - 0 to 1% germanium and titanium - 0 to 10% nickel alloys were investigated. Nickel was found to limit markedly the alpha-phase field and to lower the beta scolvus line. The range of compositions investigated in the binary titanium-silver systems was extended to 5% silver, and titanium-beryllium alloys containing 0.1 to 1% beryllium were investigated. Additions of 1 and 2% columbium or tantalum to Process A metal increased the tensile strength and lowered the ductality of Process A titanium. Ternary alloys of managese and carbon, manganese and vanadium, and molybdenum and tungsten, prepared by adding the pare metals during arc melting, had quite erratic tensile properties when tested after fabrication to aheet. Tests were completed on evaluation of " hot-pressed" titanium carbide and graphite crucibles.

Copies obtainable from CADO_

Titanium alloys

Materials (8) Mis. Non-Ferrous Metals and Alloys (12)

USAF Contr. No. AF 33(038)-3736

BATTELLE MEMORIAL INSTITUTE

INDUSTRIAL AND SCIENTIFIC RESEARCE

COLUMBUS 1, OHIO

November 11, 1949

AF 909 SO Wright-Patterson Air Force Base Service Area Building 258 Dayton, Ohio

Attention MCREXM3 Contract No. AF 33(038)-3736

Gentlemen:

Enclosed are thirty (30) copies and one (1) reproducible of the Second Progress Report prepared under Contract No. AF 33(038)-3736.

This report contains an account of the following:

- 1. A description of the alloy development work done during the bimonthly period September 1 to October 31, 1949.
- 2. The progress made during the same period on the development of refractories for holding molten titanium.
- 3. Further work on the vacuum-fusion technique for determining oxygen in titanium.

Very truly yours,

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L. W. Eastwood

LWE:mr Enc.(31)

SECOND PROGRESS REPORT

COVERING THE PERIOD SEPTEMBER 1 TO OCTOBER 31, 1949

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RESEARCH AND DEVELOPMENT ON TITANIUM ALLOYS Contract No. 33(038)-3736

to

"RIGHT-PATTERSON AIR FORCE BASE DAYTON, OHIO

BATTELLE MEMORIAL INSTITUTE

October 31,1949

BATTELLE MEMORIAL INSTITUTE

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SECOND PROGRESS REPORT

COVERING THE PERIOD SEPTEMBER 1 TO OCTOBER 31, 1949

on

RESEARCH AND DEVELOPMENT ON TITANIUM ALLOYS Contract No. AF 33(038)-3736

te

WRIGHT-PATTERSON AIR FORCE BASE DAYTON, OHIO

from

BATTELLE MEMORIAL INSTITUTE

October 31, 1949

SUMMARY

The phase relations in titanium -0 to 1 per cent germanium and titanium - 0 to 10 per cent nickel alloys were investigated. The addition of germanium to Process A metal was found to raise the beta and lower the alpha solvus lines. No germanium-rich phase was observed.

Nickel was found to limit markedly the alpha-phase field and to lower the beta solvus line. A nickel-rich phase appeared in the microstructure of the alloy containing about 7.5 per cent nickel when the specimen was quenched from 1450°F. The eutectoid composition in the binary titanium-nickel system is placed between 6 and 7 per cent nickel.

In the binary titanium-silver system, the range of compositions investigated was extended to 5.0 per cent silver, and titanium-beryllium alloys containing 0.1 to 1.0 per cent beryllium were investigated. None of these alloys had tensile properties of interest. Binary titanium-zirconium alloys containing additions of 1 to 10 per cent zirconium were also studied. No alloys of interest were noted.

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Additions of 1 and 2 per cent columbium or tantalum to Process A metal were found to increase the tensile strength and lower the ductility of Process A titanium. The range of these alloying additions will be ex-

-61-

Ternary alloys of manganese and carbon, manganese and vanadium, and molybdenum and tungsten, prepared by adding the pure metals during arc melting, were found to have quite erratic tensile properties when tested after fabrication to sheet. This condition is attributed to incomplete melting and nonuniform distribution of the alloying metals during arc melting. The use of master alloys, or hardeners, for introducing the alloying additions was investigated. This practice appears, in the limited tests conducted, to give greater homogeneity in the ingot. This melting technique will be used more extensively in the future.

Steps have been taken to re-evaluate the more promising highstrength alloys. This work is directed toward selecting an alloy composition on which extensive engineering data will be obtained. Limited data indicate that the tensile properties can be reproduced on most of these alloy compositions.

Tests were completed on the evaluation of "hot-pressed" titanium carbide and graphite crucibles lined with tantalum carbide and tungsten boride as refractories for molten titanium. Melts were prepared in crucibles made of zirconium oxide (stabilized with CaO), calcium oxide, calcium oxide fluxed with TiO₂, and aluminum oxide. The stabilized zirconium exide crucible was the first refractory tested which had areas not wet by the molten titanium. Therefore, additional melts in this type of crucible are planned to evaluate this refractory further. None of the other refractory materials tested appear to be useful. Standard specimens of iodide titanium, containing known amounts of oxygen added as TiO₂, were prepared and submitted to Dr. G. Derge, of the Carnegie Institute of Technology, for vacuum-fusion analysis. The results reported from this laboratory indicate that the vacuum-fusion technique, as it presently exists, yields fairly reliable results. Relatively minor inconsistencies in the analytical results were obtained. At the present time, it is not known whether this is inherent in the analytical technique, or merely reflects slight nonuniformity in the composition of the sample ingets.

-62-

INTRODUCTION

During the bimonthly period covered by this report, September 1 to October 31, 1949, the experimental program on titanium has continued. The phases of the work described in this report are as follows:

1. Arc Melting Titanium-Base Alloys.

2. Evaluation of Experimental Titanium-Base Alloys.

3. Investigation of Refractories for Melting Titanium.

4. Analytical Methods for Titanium-Base Alloys.

ARC MELTING TITANIUM-BASE ALLOYS

(O. V. Simmons and C. T. Greenidge)

Approximately thirty-five 0.5-pound ingots were made and submitted for fabrication during the period September 1 to October 15, 1949. The intended compositions of the ingots, on which the testing has been completed, are listed under the section on "Evaluation of Experimental Titanium-Base Alloys".

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Some difficulty was experienced when high-strength alloys containing manganese were tested. It was found that manganese had not satisfactorily gone into solution to produce a homogeneous ingot. Although manganese is a relatively low-melting-point material, its density is sufficient to cause it to sink below the surface of the molten bath where it cannot be subjected to the direct heat of the arc. This necessitates solution of a solid phase in the liquid bath rather than the mixing of two liquid phases.

-63-

To counteract this nonhomogeneity, it was decided that the manganese should be added in the form of a master alloy. A 35 per cent manganese alloy was prepared by arc melting electrolytic manganese and Process A titanium. This alloy was readily crushed and then screened to 20- to 40-mesh size and used in preparing three ingots to evaluate this procedure.

This technique appeared to produce more uniform ingots, and, subsequently, the following master alloys were prepared:

- 1. 58 per cent chromium eutectic alloy (crushed readily).
- 2. 35 per cent manganese alloy (crushed readily).
- 3. 35 per cent iron alloy (crushed readily).
- 4. 34 per cent nickel eutectic alloy (crushed readily).
- 5. 35 per cent vanadium alloy (very difficult to crush).

Since the constitution diagram of the manganesc-titanium system was not known, several small ingots were prepared for microscopic examination. These ingots contained 35, 40, 50, 60, and 70 per cent manganese, respectively. An alloy containing about 53 per cent manganese was selected as having essentially a eutectic structure.

Small test ingots of vanadium and of iron have also been prepared for microscopic examination. The eutectic compositions in these systems will be selected for the master alloys, and the necessary melting stock will be prepared.

-61-

Two 2-pound ingois of the 5 per cent chromium, 2 per cent iron, and 0.25 per cent calon alloy were prepared for forging. These ingots were melted in the arc furnace, using pure metal additions and a carbon electrode. The carbon addition was made by incidental pickup from the electrode.

A master alloy containing about 2.5 per cent nitrogen was prepared in the following manner: Titanium powder about 40 to 60 mesh was treated in the Sievert's apparatus to produce a nitrogen content of about 2.5 per cent. This material was very thoroughly mixed after removal from the apparatus to eliminate any nonuniformity which might exist. The powder was then compressed into bars 1/2 inch wide by 1/4 inch thick, which were sintered at 1900°F. for 2-1/2 hours at -0.1-micron pressure. The resultant bars were cut into pellets about 4-mesh size to provide stock for adding to the melts. The pelleting procedure severed a double function. As originally treated, the titanium powder was very difficult to add to the bath. The small size and low density of the powder permitted it to be carried out of the melting chamber by the hot furnace gases. However, the pellets dropped directly to the bath, where they floated until melted by the direct impingement of the arc. This procedure insured a uniform ingot composition through the mixing of two liquid phases.

EVALUATION OF EXPERIMENTAL TITANIUM-BASE ALLOYS

-65-

(C. M. Craighead, F. Fawn, and L. W. Eastwood)

The alloy evaluation work during the present bimonthly period has been directed toward the investigation of:

1. Binary titanium-germanium alloys.

2. Binary titanium-nickel alloys.

3. Binary titanium-silver alloys.

4. Binary titanium-beryllium alloys.

5. Binary titanium-zirconium alloys.

6. Binary titanium-columbium alloys.

7. Binary titanium-tantalum alloys.

8. Ternary alloys of titanium.

9. Evaluation of selected alloys.

Binary Alloys of Titanium

In the previous bimonthly report, data on the mechanical properties and the response to heat treatment and aging of titanium-germanium and titanium-nickel alloys were listed in Tables 1 and 2. The microscopic examination of specimens of these alloys, quenched from temperatures of 1450, 1550, 1600, 1650, 1700, and 1750°F., has now been completed. Table 16 shows the phase relations observed in the titanium-germanium alloys. These data are shown graphically in Figure 19.

"ith the addition of germanium, up to about 1 per cent, no germanium-rich phase was evident in the structure of the quenched specimens. However, as indicated in Figure 19, the addition of germanium to Process A titanium raised the beta and lowered the alpha solvus lines.

Heat No.	Intended Composition, %	As Hot Rolled 1450 F.	Heat Treated 1450 F .	Heat Treated 1550 F .	Heat Treated 1600 F .	Heat Treated 1650 T .	Heat Treated 1700 F .	Heat Treated 1750 F .
WH180	Unalloyed	a.	C.	a	80 - 20 α + β	15 - 85 α + β	β	β
WH 17 0	0 .1 Ge	a	a	85 - 15 α + β	60 - 40 α + β	5 - 95 α + β	β	β
WH169	0.5 Ge	α	α	95 - 5 α + β	70 - 30 α + β	10 - 90 α + β	β	β
UH168	1.0 Ge	a	α	90 - 10 α + β	70 - 30 α + β	30 - 70 α + β	β	β

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 TABLE 16.
 PHASES PRESENT IN BINARY TITANIUI-GERMANIUM ALLOYS

 AT TEMPERATURES INDICATED - PROCESS A METAL BASE

-66-



FIGURE 19. TENTATIVE DIAGRAM SHOWING TRANSFORM-ATION RANGE OF TITANIUM-O TO I.O PER CENT GERMANIUM ALLOYS MADE FROM PROCESS A METAL Similar data on the structure of binary titanium-nickel alloys, containing up to 10 per cent nickel, are listed in Table 17 and Figure 20. Figures 21 to 24, inclusive, show typical structures observed in these alloys.

As indicated by Figure 20, nickel markedly limits the alpha field and lowers the beta solvus line. At 1450°F., the limit of the alpha-phase field is less than 1.75 per cent nickel and, as indicated in the diagram, probably less than 1.0 per cent nickel.

As illustrated in Figure 24, a nickel-rich phase (\$) appears at 7.5 per cent nickel when quenched from 1450°F., but is absent, Figure 23, in the specimen quenched from 1550°F. From the available data, the euteotoid composition is placed between 6 and 7 per cent nickel in Figure 20.

As indicated previously, the data do not justify further investigation of binary titanium-germanium or titanium-nickel alloys.

Data for binary alloys of titanium with silver, beryllium, zirconium, columbium, and tantalum are listed in Tables 18 and 19.

Titanium-Silver Alloys

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In the Summary Report, Part III, alloys containing 1 and 2 per cent silver were considered. The range of silver content was subsequently increased to 2.5, 3.5, and 5.0 per cent.

-68-

TABLE 17. PHASES PRESENT IN BINARY TITANIUL-NICKEL ALLOYS AT TEMPERATURES INDICATED - PROCESS A METAL BASE

A.

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		Composit	tion. 5	70		Rolled	Rolled	Heat	Heat	Heat.	Hoot	Haot	Hoot
lleat No.	Intended		Actue C	1		at 1450 TF.	Aged 4 Hrs750°F.	Treated 1450°F.	Treated 1550 T.	Treated 1600 T.	Treated 1650°F.	Treated 1700 T.	Treated 1750 °F.
WH247	1.75 Ni	1	ſ	I	t	а + Ю	ະ ເ	70 - 30 a + B	50 - 50 α + β	20 - β0 α + β	e.	ත	ൻ
MH246	2.5 NI	.1	ţ.	ŧ	i	я + В	a + 13	60 - 40 α + β	40 - 60 α + β	ů.	ದ್	Ø.	Q.
VIB245	3.5 NL	1	t	ł	1	50 - 50 a + β	50 - 50 α + β	40 - 60 a + B	15 - 85 α + β	ß	Ċ,	 	ب م
UH244	5•0 M	4	· 1	t	ŧ	6 +	. ଟ ମ	15 - 35 a + 8	Ċ.	cî,	á,	<u>6</u>	œ,
UH241	7.5 NI	ŧ	ł	ł	L	* * +	β + γ*	β + Tr. Υ	۲ *	G,	G,	e.	œ.
VIII237	IO.O NI	ì	T	ſ	t	לי אייני	છે + પ્ર	*. + ત	β + γ*	9 + 7 *	6 + ۲	ą	đ
**T0.1	10°0 MT	11•4 MÌ	0•07	0.029	0•59	в + т *	ł	9 + 3	β + γ*	β. + γ *	* , ≁ * વ	8 + γ	₽ + %

** Data taken from Summary Report - Part III, page 196.

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CENT NICKEL ALLOYS MADE FROM PROCESS A METAL.

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ATING AND AGING DATA FOR BINARY ALLOIS OF WITH SILVER, BERTLLIUN, ZIRCONIUM, COLUMBIUM, UM PREPARED FROM PROCESS A METAL -73-

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	Heat Treated 1750 F. (10)		×1/2 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,256 2,567 2,566 2						>1/r*		>1/4* >1/4* 3/16
	Heat Treated 1700°F		>1/14 3/16 1/16 1/16	t			× × × × × × × × × × × × × × × × × × ×	;	>1/4*		>1/1* 3/16 3/16
estment(2)	Heat Treated 1650°F. (8)		3776		3716 3716 3716 3716 3716 74 *				1/16 3/16 3/16		1/16 3/16 3/16
icated Tr	Heat Treated 1600°F.	· .	1/16 3/32 3/16				111 111 111 111 111 111 111 111 111 11		1/16 3/16 3/16		1/16 3/16 1/16
After Ind	Heat Treated 1550°7. (6)		3/16 3/16 3/16		23/16 37/16 27/16 27/16 27/16		2777 2777 2777 2777 2777 277 266 277 277		3/16 3/16 3/16		3/16 3/16 3/16
d Radius	Heat Treated 1450°F.	• -	3/16 3/16 3/16		21/16 21/16 21/16 21/16 21/16		3/16 3/16 3/16 3/16		3/16 1/16 3/16		3/16 3/16 3/16
Minimum Ber	As Hot Rolled Aged 4 Hrs 750°F.		3/16 3/16 3/16 3/16		3/16 3/16 3/16 3/16 16		3/16 3/16 3/16 3/16		3/16 3/16 3/16	Ţ	3/16 3/16 3/16
	As Hot Rolled (3)		3/16 3/16 3/16		2116 2716 3716 3716 23716 216		3/16 3/16 3/16 3/16 >1/4		3/16 3/16		3/16 3/16 3/16
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1)	Heat Treated 1700°F. (9)	um - Silve	209 227 216	a - Beryll	220 249 288 293 293	<u>a - Zirco</u>	209 207 208 208 208 274	H - Colum	209 216 236	m - Tanta	209 201 221
treatment(Heat Treated 1650°F. (8)	Titaniv	194 206 191	T1 tani w	188 221 221 241 241	Titaniu	194 212 225 225 225 225 266	T1 taniu	194 182 232	Titani u	194 200 225
dicated 7	Heat Heat Treated 1600°F. (7)		202 201 281 281		180 200 210 266 249		196 202 203 219 242		196 180 233		194 194 194
lfter Tv	Heat Treated 1550°F. (6)		196 188 187		179 191 237 214 214 214		252 131 188 198 252 131 188 198 252 131 188 198		196 172 247		196 179 206
To a start	Heat Heat Treated 1450°F. (5)		191 176 182 168		179 206 183 183 185		191 171 177 216 216 216		191 160 221		161 171 191
	As Hot As Hot Rolled Aged 4 Hrs 750°F.		216 221 201 218		223 225 225 223 223 223 223 223 223 223		212 212 223 222 235 235		216 205 219	·	216 201 201
	As Hot Rolled (3)		201 2018 2018		201 215 215 215 215 230 230		203 218 219 219 219 219 219 219 219 219 219 219		203 197 236		203 201

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LE 19 HEAT-TREAT TITANIUN Y OR TANTAIN	 -	Intended Composition, 5	Unalloyed 2.5 Ag 3.5 Ag 5.0 Ag	Unelloyed 0.10 He 0.15 He 0.25 He 0.50 He 1.0 He	Unalloyed 1.0 2r 2.5 2r 3.5 2r 5.0 2r 10.0 2r	Unalloyed 1.0 Cb 2.0 Cb	Unalloyed 1.0 Ta 2.0 Ta 2.0 Ta	, _ · · ·
TAB		Heat No.	WER248 WER248 WER255 WER2555	WH262 WH265 WH265 WH265 WH263 WH263	MH248 WH253 WH253 WH2551 WH2550 WH249	#H248 #H270 #H269	WH248 WH248 WH273 WH272 Footnc	
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WIZ55 5.0 kg 82,700 20.0 20.1 1/16 WIZ56 5.0 kg 91,900 20.5 3/16 3/16 WIZ65 0.10 Be 91,900 20.5 3/16 3/16 WIZ65 0.10 Be 95,600 14.0 215 3/16 WIZ65 0.10 Be 95,600 14.0 215 3/16 WIZ65 0.20 Be 100,000 14.0 215 3/16 WIZ65 0.20 Be 95,600 12.0 210 3/16 WIZ65 0.20 Be 95,600 12.0 210 3/16 WIZ64 1.0 Dz 22 22 210 3/16 21/4* WIZ65 2.5 Zr 92,500 18.6 210 3/16 WIZ64 10.0 Zr 95,600 18.6 210 3/16 WIZ65 2.5 Zr 9716 210 3/16 21/4* WIZ65 10.0 Zr 2.0 Zr 21/4* 21/4* 21/4*	WH2248	Unalloyed 2.5 Ag	95,600 93,200	21.0	203	3/16 3/16	94,700 91,200	20.5 20.0	216	3/16
Titantum Titantum Berrilium WT262 Unalloyed 85,700 21.5 201 3/16 WT265 0.10 Be 91,1000 18.5 201 3/16 WT265 0.10 Be 91,1000 18.5 201 3/16 WT265 0.10 Be 91,1000 18.5 215 3/16 WT264 0.50 Be 97,900 12.0 200 3/16 WT264 0.50 Be 97,900 12.0 200 3/16 WT264 0.50 Be 97,900 12.0 200 3/16 WT255 2.5 Zr 98,300 12.0 203 3/16 WT255 2.5 Zr 98,300 18.5 200 3/16 WT255 2.0 Zr 97,600 18.5 210 3/16 WT255 2.0 Zr 97,600 16.5 249 3/16 WT255 10.0 Zr 10.0 Zr 203 3/16 11.4 WT255 2.0 Zr 97,6	WH256	3.4 ∂ 5.0 ⊉8 88	82 , 700 91 , 900	20.5	201 210	1/16 3/16	83, 500 92, 900	20.0 19.0	201	1/16 3/16
WI262 Unalloyed 85,700 21.5 201 3/16 WI265 010 Be 9,500 11.5 215 3/16 WI265 015 Be 95,900 11.0 201 3/16 WI265 015 Be 95,900 11.0 201 3/16 WI265 055 Be 95,900 12.0 203 3/16 WI265 057 Be 95,900 12.0 203 3/16 WI265 1.0 Be 95,600 12.0 203 3/16 WI261 3.5 Zr 95,500 18.5 203 3/16 WI251 3.5 Zr 98,300 18.5 203 3/16 WI252 2.0 Zr 18.6 2.0 Zr 2.1/4 H WI252 2.0 Zr 2.0 Zr 2.0 Zr 2.1/4 H W					Titanium -	Beryllium		·		, , ,
WIEGG O.15 Be YL,100 IS.0 ZI5 YL WIEGG O.15 Be YL,100 IS.0 ZI5 YL WIEGG O.50 Be YL,200 IS.0 ZI5 ZI6 ZI6 <thz6< t<="" td=""><td>WH262 WH267</td><td>Unalloyed</td><td>85,700</td><td>21.5</td><td>201</td><td>3/16</td><td>95,900</td><td>20.0</td><td>203</td><td>3/16</td></thz6<>	WH262 WH267	Unalloyed	85,700	21.5	201	3/16	95 , 900	20.0	203	3/16
MH265 0.25 Be 100,000 14.0 215 3/16 MH263 0.50 Be 95,900 14.0 215 3/16 MH263 1.0 Be 97,300 12.0 230 3/16 MH263 1.0 Be 97,300 12.0 230 3/16 MH263 1.0 Er 95,600 21.0 230 3/16 MH263 2.5 Zr 95,500 18.0 21.0 230 3/16 MH263 2.5 Zr 95,600 21.0 203 3/16 H1 MH269 5.0 Zr 92,500 18.0 21.0 203 3/16 MH269 5.0 Zr 92,500 18.0 21.0 203 3/16 MH269 5.0 Zr 92,500 18.6 249 3/16 H1 MH270 1.0 Cr 92,500 18.6 249 3/16 H1 MH270 1.0 Cr 92,500 16.5 249 3/16 H1 MH270	WH266	0.15 Be	008'96	18•0 16•5	206 215	1/16 3/16	90,000	18.0	198	5Ý1
WI263 1.0 Be 77,300 12.0 230 3/16 WI223 1.0 Be 97,300 12.0 230 3/16 WI253 1.0 Er 95,600 21.0 230 3/16 WI253 1.0 Er 95,600 21.0 230 3/16 WI253 2.5 Zr 95,600 21.0 203 3/16 WI255 2.5 Zr 95,600 21.0 203 3/16 WI255 2.5 Zr 95,600 18.5 210 3/16 WI259 10.0 Zr 118,800 15.0 249 3/16 WI249 Unalloyed 95,600 21.0 203 3/16 WI226 Unalloyed 95,600 21.0 203 3/16 WI226 1.0 Cb 95,600 21.0 203 3/16 WI226 1.0 Cb 95,600 21.0 203 3/16 WI226 1.0 Cb 95,600 21.0 203 3/16 WI226 2.0 Cb 1.0 Cb 95,500 16.5 2.0 2.0 2.0	WH265 WH264	0.25 Be 0.40 Pe	100,000	14.0	215	3/16	88,100	14.5	213	9/10 3/16
WH248 Unalloyed 95,600 21.0 203 3/16 WH252 2.5 Zr 92,500 18.5 202 1/8 WH253 1.0 Zr 92,500 18.5 202 1/8 WH253 2.5 Zr 92,500 18.5 202 1/8 WH253 3.5 Zr 95,400 18.5 21.0 203 3/16 WH253 3.5 Zr 95,400 18.5 21.0 203 3/16 WH259 10.0 Zr 11.0,800 15.5 249 3/16 11 WH248 Whalloyed 95,600 15.0 203 3/16 11 WH248 Unalloyed 95,600 16.5 203 3/16 11 WH270 1.0 0 Cb 108,900 16.5 203 3/16 11 WH270 2.0 0 Cb 108,900 16.5 203 3/16 11 WH270 2.0 0 Cb 108,900 16.5 203 3/16 11	WH263		97,300	50°0	230	3/10 >1/4*	94,400 90,100	14.5 9.5	221	3/16 3/16
WH248 Unalloyed 95,600 21.0 203 3/16 WH253 2.5 Zr 92,500 18.5 202 1/8 WH253 2.5 Zr 92,500 18.5 202 1/8 WH253 3.5 Zr 98,300 18.5 202 3/16 WH259 5.0 Zr 96,400 18.5 210 3/16 WH259 10.0 Zr 104,300 16.5 249 3/16 WH249 Unalloyed 95,600 21.0 203 3/16 WH248 Unalloyed 95,600 21.0 203 3/16 WH270 1.0 Cb 200 19.5 236 3/16 WH279 2.0 Cb 2.0 Cb 29.5 236 3/16					<u>Titanium -</u>	Zirconium				-
MEZ53 1.0 $\mathbb{Z}r$ 92,500 18.5 202 1/8 ME251 2.5 $\mathbb{Z}r$ 98,300 18.5 202 1/8 ME251 3.5 $\mathbb{Z}r$ 98,300 18.5 210 3/16 1 ME250 5.0 $\mathbb{Z}r$ 10.0 $\mathbb{Z}r$ 104,300 18.5 210 3/16 1 ME260 5.0 $\mathbb{Z}r$ 10.6,5 2.49 3/16 1 1 ME260 10.0 $\mathbb{Z}r$ 118,800 15.0 249 3/16 1 ME248 Unalloyad 95,600 21.0 203 3/16 1 ME248 Unalloyad 95,600 21.0 203 3/16 1 ME248 Unalloyad 95,600 21.0 203 3/16 1 ME269 2.0 $\mathbb{C}b$ 108,900 16.5 2.36 3/16 1 ME248 Unalloyad 95,600 21.0 203 3/16 1 ME269 2.0 $\mathbb{C}b$ 2.	WH248	Unalloyed	95,600	21.0	203	3/16	94.700	20.5	216	3/16
MI253 3.5 Zr $96,400$ 18.5 210 $3/16$ 11 MI259 5.0 Zr 10.0 Zr $10.1/4$ * 11 WH249 Unalloyed $95,600$ 15.0 249 $3/16$ $11/4$ * 11 WH248 Unalloyed $95,600$ 15.0 249 $3/16$ $11/4$ * 11 WH248 Unalloyed $95,600$ 15.0 20.3 $3/16$ $11/4$ * 11 WH248 Unalloyed $95,600$ 16.5 23.0 $3/16$ $3/16$ $3/16$ WH248 Unalloyed $95,600$ 16.5 236 $3/16$ $3/26$ $3/16$ MH273 1.0 10 20 21.0 203 $3/16$ MH273 1.0 20 21.0 203 $3/16$ 21.0 203 $3/16$ 21.0 203	WH253		92,500 06,300	18.5	202	1/8	64,100	21.0	212	1/8
WH250 5.0 Zr 104,300 16.5 249 3/16 11 WH249 10.0 Zr 118,800 15.0 249 3/16 11 WH248 Unalloyed 95,600 15.0 249 3/16 11 WH248 Unalloyed 95,600 21.0 203 3/16 11 WH248 Unalloyed 95,600 21.0 203 3/16 11 WH248 Unalloyed 95,600 16.5 23.0 3/16 11 WH248 Unalloyed 92,700 19.5 236 3/16 3/16 WH269 2.0 Gb 108,900 16.5 236 3/16 3/16 WH269 2.0 Gb 108,900 21.0 203 3/16 21.0 236 3/16 WH273 1.0 Ta 89,900 21.0 203 3/16 21.0 203 3/16	152H	3.5 25	96,400	18.5 18.5	210	3/16	102,400	17.5	223	3/16
WH248 Unalloyed 95,600 21.0 203 3/16 WH248 Unalloyed 95,600 21.0 203 3/16 WH269 2.0 Cb 1.0 Cb 92,700 19,5 197 3/16 WH269 2.0 Cb 1.0 Cb 92,700 19,5 297 3/16 WH269 2.0 Cb 108,900 16,5 236 3/16 WH269 2.0 Cb 108,900 16,5 236 3/16 WH2248 Unalloyed 95,600 21.0 203 3/16 WH2248 Unalloyed 95,600 21.0 203 3/16	WH250 WH249	5.0 Zr 10.0 Zr	104, 300	16.5	672 576	3/16	117, 300 114, 300	13.5	222 235	3/16 3/16
WH248 Unalloyed 95,600 21.0 203 3/16 WH270 1.0 Cb 92,700 19,5 197 3/16 WH269 2.0 Cb 108,900 16.5 236 3/16 WH269 2.0 Cb 108,900 16.5 236 3/16 WH269 2.0 Cb 108,900 16.5 236 3/16 WH269 2.0 Cb 108,900 21.0 203 3/16 WH273 1.0 Ta 89,900 21.0 203 3/16 WH273 1.0 Ta 89,900 21.0 203 3/16					Titanium -	Columbium	ļ) 6 1		
WH270 1.0 Cb 92,700 19,5 197 3/16 WH269 2.0 Cb 108,900 16.5 236 3/16 WH269 2.0 Cb 108,900 16.5 236 3/16 WH269 2.0 Cb 108,900 21.0 236 3/16 WH248 Unalloyed 95,600 21.0 203 3/16 WH273 1.0 Ta 89,900 21.0 201 3/16	WH248	Unalloyed	95,600	21.0	.203	3/16	002 - 200	3 00	710	, , , , , , , , , , , , , , , , , , ,
Titenium Tantelum WH2/8 Unalloyed 95,600 21.0 203 3/16 WH273 1.0 Ta 89,900 21.0 201 3/16	WH270 WH269	1.0 Gb 2.0 Gb	92,700 108,900	19.5	197 236	3/16	97,500	18.0	205 205	3/16 3/16
WH248 Unalloyed 95,600 21.0 203 3/16 WH273 1.0 Ta 89,900 21.0 201 3/16					Titanium - '	Tantel un				i.
	WH248 WH273	Unalloyed	95,600 30,000	21.0	203	3/16	64*700	20+5	216	3/16
TUCK C. C. U 18 99,100 18.0 220 3/16 10	WB272	2.0 Ta	09,100	0.81	220	3/16 3/16	90, 300 104, 700	20•0 17•0	201	3/16

Footnotes on following page.

Footnotes for Table 18:

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- (1) As hot rolled at 1450°F.
- (2) Hot-rolled sheet aged 4 hours at 750°F. in air and air cooled.
- (3) Average of two longitudinal l4-gauge specimens, 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section.
- (4) 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and to the rolling direction.
- (5) Minimum bend radius without cracking on a single longitudinal specimen 3 inches long by 0.5 inch wide. Tested with surface skin present.

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* Some ductility.

** Very little ductility.

Footnotes for Table 19:

- 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and to the rolling direction.
- (2) Minimum bend radius without cracking on a single longitudinal specimen 3 inches long by 0.5 inch wide. Tested with the surface skin present.
- (?) As hot rolled at 1450°F.

(4) Hot-rolled sheet aged 4 hours at 750°F. in air and air cooled.
(5) Heated 1/2 hour in air at 1450°F. and quenched in cold water.
(6) Heated 1/2 hour in air at 1550°F. and quenched in cold water.
(7) Heated 1/2 hour in air at 1600°F. and quenched in cold water.
(8) Heated 1/2 hour in air at 1650°F. and quenched in cold water.
(9) Heated 1/2 hour in air at 1700°F. and quenched in cold water.

(10) Heated 1/2 hour in air at 1750°F. and quenched in cold water.

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Mechanical Properties. The mechanical properties of the three titanium-silver alloys, mentioned previously, in both the as-hot-rolled condition and after aging the as-hot-rolled temper for 4 hours at 750°F. are listed in Table 18. Figure 25 illustrates graphically the effect of silver on the tensile properties and hardness of the as-hot-rolled temper.

As shown by the data in Table 18, titanium-silver alloys in the as-hot-rolled condition do not respond to an aging treatment of 4 hours at 750°F. From the present data, and the results previously listed in the summary report, it is concluded that silver, in amounts up to 5 per cent, is a relatively ineffective alloying addition to titanium.

Structure and Transformation Range. The structures observed in titanium-silver alloys quenched from temperatures of 1450, 1550, 1600, 1650, 1700, and 1750°F. are listed in Table 20. The phase relations are shown graphically in Figure 26. In this figure, the data previously listed in the summary report for alloys containing 0.87 and 2.12 per cent silver are included. It is evident that silver raises the beta and lowers the alpha solvus lines.

Heat Treatment. The Vickers hardnesses and bend characteristics of the 2.5, 3.5, and 5.0 per cent silver alloys, after quenching from temperatures of 1450, 1550, 1600, 1650, 1700, and 1750°F., are listed in Table 19. At the higher quenching temperatures, slight increases in hardness were obtained, but there is little evidence to indicate that titaniumsilver alloys respond to solution heat treatment. Aging the as-hot-rolled alloys for 4 hours at 750°F. did not increase the hardness.

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FIGURE 25 EFFECT OF SILVER ON THE PROPERTIES OF AS-HOT-RCLLED PROCESS A TITANIUM SHEET

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TABLE 20. PHASES PRESENT IN BINARY TITANIUL-SILVER ALLOYS AT TEMPERATURES INDICATED - PROCESS A LETAL BASE

1.1

		Composit	Non. 5			As Hot Rolled	As Hot Rolled	Heat	Heat	Heat	Meat	Heat	Heat	
Heat			Actua			at	Aged	Treated	Treated	Treated	Treated	Treated	Treated	
No.	Intended		IJ	ц	=	1450%	4 Hrs750 %.	-To 0471	-1-06CT	• T-009T	-1000T	-H-00/J	-T-05/T	
11077*	<u> Unalloyed</u>		0•0	0. 026	0.10	੍ਰ ਖ	I	ರ	95 a + 7 50	35 - 15 α + β	G.	ପ	<u>.</u>	•
*76°0M	1.0 Ag	0.87 Ag	0• 03	0• 085	0.13	ರ	- 1	ġ	ರ	90 + 3 β + β	വ്വം	œ,	ମ୍ଭ	
*96DM	2•0 Åg	2.12 Ag	0.02	0• 035	0• 03	B	ł	ម	95 - 5 a + 3 b	30 - 20 α + β	10 - 90 a + B	5 - 95 α + β	<u>ت</u>	-76-
WH254	2•5 Ag	4	i	1	ì	U	ರ	t	90 – 10 α + β	70 – 30 a + B	60 - 1 a + B	e.	φ.	
VH255	3*5 Ag	ł	I	ł	ť	đ	B	¢,	95 - 5 a + 5 a	60 - 40 a + B	30 = 70 a + B	ୟ	л,	
MH256	5.0 Ag	Ť.	L	ſ	t	ğ	t	ರ	g + 117. g +	60 = 40 α + β	40 = 60 α = β	œ.	39 	

* Data taken from Summary Report - Part III, page 91.



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FIGURE 26. TENTATIVE DIAGRAM SHOWING TRANS-FORMATION RANGE OF TITANIUM-OTO 5 PER CENT SILVER ALLOYS MADE FROM PROCESS A METAL

0-13670

Titanium-Beryllium Alloys

Previously, in the summary report, titanium-beryllium alloys containing 1.0 and 2.0 per cent added beryllium were considered. At 2.0 per cent beryllium, a beryllium-rich phase was noted in specimens quenched from temperatures ranging from 1450 to 1750°F. Alloys with beryllium additions of 0.1, 0.15, 0.25, 0.5, and 1.0 per cent were prepared.

-78-

Mechanical Properties. The tensile properties, hardness, and bend characteristics of the beryllium alloys, in both the as-hot-rolled condition and after aging the alloys in the as-hot-rolled temper for 4 hours at 750°F., are listed in Table 18. Figure 27 graphically illustrates the effect of beryllium on the tensile properties and hardness of as-hot-rolled titanium sheet. In this figure, the data previously listed in the summary report have been included. Beryllium produces no marked increase in the strength or hardness, but progressively decreases the ductility. As will be noted from the data in Table 18, the titanium-beryllium alloys in the ashot-rolled temper did not respond to an aging treatment of 4 hours at 750°F.

<u>Structure and Transformation Range.</u> Specimens of the titaniumberyllium alloys quenched from temperatures ranging from 1450 to 1750°F. were examined microscopically. Beryllium appeared to limit the alpha-phase field and raise the beta solvus line, but the limit of solubility of the beryllium-rich phase could not be established with any degree of accuracy. Further study of these alloys will be required before a tentative phase diagram can be constructed.



0-13671

Heat Treatment. The Vickors hardness values and bend characteristics of the titanium-beryllium alloys when quenched from temperatures of 1450, 1550, 1600, 1650, 1700, and 1750°F. are listed in Table 19. At the higher quenching temperatures, some increase in hardness was obtained, but there is no evidence that these alloys respond appreciably to heat treatment. As shown by the data in Tables 18 and 19, these titanium-beryllium alloys in the as-hot-rolled temper do not age harden:

-80-

Titanium-Zirconium Alloys

Additions of 1.0, 2.5, 3.5, 5.0, and 10.0 per cent zirconium were made to Process A titanium. These alloys are considered in the following section.

Mechanical Properties. The tensile strength, elongation, hardness, and bend characteristics of the titanium-zirconium alloys in both the as-hot-rolled condition and after aging the alloys in the as-hot-rolled temper for 4 hours at 750°F. are listed in Table 18. Figure 28 graphically illustrates, for the as-hot-rolled condition, the effect of zirconium on the tensile strength, elongation, and hardness. Zirconium raises the tensile strength and hardness with relatively little drop in elongation. With 10 per cent zirconium added, the tensile strength increased about 37,000 p.s.i., with 6.0 per cent drop in elongation. As compared to chromium, manganese, molybdenum, and some other elements, zirconium is a relatively ineffective alloying addition.



0-13672

As shown by the data in Table 18, the titanium-zirconium alloys are not appreciably benefited by aging the alloys in the as-hot-rolled temper for 4 hours at 750°F.

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<u>Structure and Transformation Range.</u> The phases present in the titanium-zirconium alloys when quenched from temperatures of 1450 to 1750°F. are listed in Table 21, and are shown graphically in Figure 29. Zirconium limits the alpha-phase field and lowers the beta solvus line. No zirconiumrich phase was observed at concentrations up to 10 per cent zirconium.

Heat Treatment. The Vickers hardness values and minimum bend radii of these titanium-zirconium alloys when quenched from temperatures of 1450, 1550, 1600, 1650, 1700, and 1750°F. are listed in Table 19. Some increase in hardness will be noted after quenching the alloys from the higher temperatures. However, the change in hardness is not comparable to that observed in some of the other binary systems. Aging the as-hot-rolled titanium-zirconium alloys for 4 hours at 750°F. did not produce any increase in hardness. It is, therefore, concluded that titanium-zirconium alloys in the range of composition investigated do not show any significant response to heat treatment or aging.

FHASES PRESENT IN BINARY TITANIUM-ZIRCONTUM ALLOYS AT TEMPERATURES INDICATED - PROCESS A METAL BASE

TABLE 21.

						-				
Heat No.	Intended Composition, %	As IIct Rolled at 1450 T.	As Hot Rolled Aged 4 Hrs750 °F.	. Heat Treated 1450 Tr.	Heat Treated 1550 m.	Heat Treated 1600 °r.	Heat Treated 1650 ° Tr	Heat Treated 1700 °F.	Heat Treated 1750 Tr	
VII1248	Unalloyed	ರ	ರ	ය + Tr. යි	90 - 10 a + B	30 – 20 a + B	70 – 30 a + p	œ.	đ	
WH253	1.0 Zr	ିଅ	ರ	e	ť	30 - 20 a + β	20 - 30 α + β	Ċ.	Q.	
VH252	2.5 Žr	B	ರ	ರ	α + Tr. β	30 = 20 α + β	20 = 30 α + β	a.	-ر 0-	-83
WH251	3•5 Zr	ಶ	ರ	ਰਾਂ ਹੈ 11. 11. 11. 11. 11. 11. 11. 11. 11. 11	90 – 10 α + β	50 - 50 α + β	30 - 70 α + β	Q,	β	
WH250	- 5.0 Zr	α + β	a + B	ອ ສ	ອ ເຊິ່	50 - 50 α + β		ଜୁ	ب ال	
VH249	10.0 2r	හ + ප	ਦ ਦ	70 - 30 α + β	70 - 30 α + β	ପ	Q ,	ġ.	a.	

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0-13673

Titanium-Columbium Alloys

Additions of 1.0 and 2.0 per cent columbium were made to Process A titanium. The properties of these alloys are considered in the following section.

-85-

Mechanical Properties. The tensile properties, hardnesses, and minimum bend radii of the two titanium-columbium alloys, in both the ashot-rolled condition and after aging the alloys in the as-hot-rolled temper for 4 hours at 750°F., are listed in Table 18. Figure 30 graphically illustrates the effect of columbium on the as-hot-rolled properties. The tensile strength and hardness increase with increasing columbium content, while the elongation decreases. These two columbium-containing alloys did not respond when the as-hot-rolled sheet was aged 4 hours at 750°F.

Considering the above properties, the range of columbium content will be extended to 10 per cent.

Titanium-Tantalum Alloys

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Alloys containing 1.0 and 2.0 per cent tantalum were investigated. The properties of these alloys are considered in the following - section.

Mechanical Properties. The tensile properties, hardnesses, and minimum bend radii of the two titanium-tantalum alloys are listed in Table 18. These data are for the alloys in the as-hot-rolled condition and after aging the alloys in the as-hot-rolled temper for h hours at 750°F. Figure 31




A CONTRACTOR AND

FIGURE 31. EFFECT OF TANTALUM ON THE PROP-ERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET graphically illustrates the effect of 1.0 and 2.0 per cent tantalum on the tensile properties and hardness of Process A titanium in the as-hot-rolled condition. The addition of 2.0 per cent tantalum increased the tensile strength of Process A titanium sheet about 20,000 p.s.i. with a reduction in clongation of about 3.0 per cent.

Additional data on binary titanium-columbium and titanium-tantalum alloys, when the range of alloy additions has been extended to about 10 per cent, will be considered in a future report.

Ternary Alloys of Titanium

The following three series of alloys were prepared and fabricated to sheet:

1. 3.5, 4.0, 4.5, and 5.0% Mn with 0.25% C.

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2. 1.75, 2.5, 3.5, and 5.0% Mn with 1.0 and 2.0% V.

3. 2.5, 3.5, and 5.0% Mo with 1.0, 2.0, and 3.0% ".

The tensile properties were obtained on these alloys in both the as-hot-rolled condition and after aging the alloys in the as-hot-rolled temper 4 hours at 750°F. In many cases, variations in strength and clongation of considerable magnitude were noted between the duplicate specimens from the same alloy sheet. Retesting selected alloys showed similar results, and it was concluded that nonuniform distribution of the alloying elements during melting was responsible for these variations. These alloys will be made over, using a technique in melting designed to give more uniform distribution of the alloying additions.

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As a step in obtaining a more uniform distribution of alloying elements, the possibility of employing alloy hardeners rather than the pure metals in arc melting was investigated. A brittle manganese alloy containing about 35 per cent manganese was prepared by arc melting. This alloy was crushed to pass 20 mesh and used to prepare the following 0.5-pound ingots:

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Heat No.	Composition, %
V/H312	5 Mn
V/H310	5Mn, 0.25C
V/H313	5Mn, 0.1N

These ingots and an unalloyed heat were fabricated to sheet, and six longitudinal tensile specimens were taken at various locations throughout the length of the sheet. Two specimens each were taken from opposite sides and ends of the sheet and two adjacent specimens were from the center. The mechanical properties and hardness of the various specimens are listed in Table 22.

As noted in Table 22, some of the specimens of the manganese alloys showed a small amount of unmelted alloy in the fracture. Although the properties of such specimens have not been included in the average values, the small amount of inhomogeneity observed in the fracture has not had an appreciable effect on the tensile strength. Elongation values of these specimens, however, tend to be low.

Considering the small cross-sectional area of the test specimen, about 0.016 square inch, these properties appear relatively uniform and compare favorably with values previously reported for similar compositions, as will be noted from the following:

COMPARISON OF THE PROPERTIES OF TITANIUM ALLOYS

-90-

(As-Hot-Rolled Temper)

Alloys Prepared Using Metal Additions During Arc Melting Alloys Prepared Using Alloy Intended Hardeners in Arc Melting or Actual Tensile Elong., Intended Tensile Elong., Heat Composi-Strength. % in Heat Composi-Strength, % in No. tion, % 1 Inch p.s.i. VHN No. tion, % 1 Inch p.s.i. VHN "(C)₁₁(1) 4.37Mn 157,900 7.5 303 "H78(1) 5.0Mn 162,800 6.5 315 "H312 5Mn 169,100 5.8 366 rc58(2) 4.80Mn, 5.5 176,500 342 'WH310 5.0Mn, 178,730 6.2 378 0.210 0.250 ":H93(2) 5.0Mn. 193,400 2.5 444 0.250 "H162(3) 5.0Mn, 173,500 8.5 101 ".H313 5.0Mn, 183,340 6.0 409 0.1N 0.1N (1)Data from Summary Report, page 182. (2)Data from Summary Report, page 238a.

(3) Data from Progress Report, Table 5, page 21.

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TABLE 22.	TEST OF	UNIFOR	MITY OF	THE	TENSILE	PROPERTI	ES AND
	HARDNES	S ÓF ŤÌ	TA NIUM	ALLOY	SHEET.	INCOTS	MELTED
	USING A	35 PER	CENT M	ANGAN	ESE ALLO	OY HARDEN	İER

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Heat	Specimen No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(1)	Elong., % in 1 Inch(1)	VHN (2)
".H301	X-1	Unalloyed	Front, side	87,300	17.5	216
11	X-2	11-	Front, side	92,920	18.0	205
11	Y-1	11	Center	92, 310	17.0	212
11	Y-2	11	Center	91,600	19.0	210
11	Z-1	.11	Rear, side	90,000	18.0	209
11	Z-2	*1	Rear, side	88,000	19.5	207
11		ŧ	Avg.	90, 360	18.2	210
"H312	X-1	5Mn	Front, side	166,620	6.5	348
11	X-2		Front, side	167,220	8.5.	343
H ~	Y-1	tt.	Center	168,790*	4.5*	368
tt	Y-2	11	Center	167,720	4.0	372
ff	Z-1	H.	Rear, side	174,830	4.0	378
17	Z-2	tt.	Rear, side	175,860*	4.5*	386
11		11	Avg.	169,100	5.8	366
"H310	X-l	5Mn, 0.25C	Front, side	185,430	4.5	377
tt	X-2	11 11	Front, side	162,160	6.5	358
-11	Y-1	11 11	Center	197,120	. 5.0	378
n	Y-2	11 · 11	Center	181,090	8.0	381
tí	Z-1	li ti	Rear, side	190,070*	2.5*	398
11	Z-2	n 11	Rear, side	167,830	7.0	375
n		11 1 1	Avg.	178,730	6.2	378
".H313	X-1	5.0Mn,0.1N	Front, side	201,01 0*	4.0*	425
મ	X-2	เมิน	Front, side	192,310*	4 .0 *	407
11	Y-1	st tł	Center	194,900*	4.0*	420
ÌT	Y-2	u u	Center	189,870	7.0	401
11	2 -1	tt et	Rear, side	181,610	7.0	418
11	Z-2	11 II	Rear, side	178,530	4.0	386
11		11 (1	Avg.	183,340	6.0	409

* Unmelted alloy evident in fracture - results not included in average.
(1) Longitudinal specimen - as-hot-rolled temper.
(2)" Center hardness transverse specimen. Average of 5 readings.

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This technique of using intermediate alloy hardners for the introduction of the alloy additions rather than the use of pure metals during arc melting appears very promising and will be investigated more fully.

Evaluation of the Alloys of Greatest Interest

In the Future "Fork section of the previous report, it was indicated that the more promising alloys that have been prepared to date would be reinvestigated. The object of this work is to select an alloy composition that will be prepared in large-size ingots and on which extensive engineering data will be obtained.

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To this end, a group of alloys having tensile strengths in the range of 180,000 to 200,000 p.s.i. was selected for further study. A small amount of the original sheet was still available from these heats, and longitudinal tensile specimens in duplicate were prepared and tested using SR4-type A7 strain gauges to obtain the 0.2 per cent yield strengths. In a few cases, as will be noted subsequently, the range of the electrical strain recorder was not adequate to permit a full extension of 0.2 per cent. In these cases, the 0.2 per cent yield strength is either not reported, or it is indicated that the yield strength was extrapolated by slightly extending the stress-strain curve.

The original tensile properties, hardness, and bend characteristics reported for these alloys and the redetermined tensile properties and the chemical analyses of the various heats are listed in Table 23. In general, the agreement between the two tests is quite good. The three titanium-manganese-vanadium alloys listed at the end of the table, however, show wide variations between the two sets of properties. These three alloys,

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TABLE 23. EVALUATION OF SELECTED TITANUM-BASE ALLOYS IN THE AS-HOT-ROLLED CONDITION

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				Origi	nal Properti	88		R	sdetermined Pro	operties	
Heat No.	Spec. No.	Com Intended	position, S. Actual	Tensile Strength, p.s.i.(8)	Elong. f in l Inch (NHA (6)	Bend Bend tadius, inch(10)	Tensils Strength, p.s.i.(8)	Yield Strength, p.s.i. at (0.2% Offset)	Elong., \$ in 1 Inch	Minimum Bend Radius, Inch(10)
FG27(1)	L Avg.	3.50r,0.250,2.0re	3.930r,1.42Fe	191, 300 190, 000 190, 700	6.0 6.0	373	3/16	176,750 1 <i>6</i> 9,300 173,000	148,100 153,500# 150,800	N O O IA	3/16
W031(1)	Avg.	5.00r,0.25c,2.0Fe	4.660r,2.15Fe	183,500 212,500 198,000	6•0 4•0	to	***//*<	184 , 600 186, 700 190, 650	169,300 167,700 168,500	000 000	**//*
WG40(I)	L 2 Avg.	5.00r,0.25c,2.0Mn	4.630r, 2.02Mn	196,300 192,500 194,400	0000 0000	otic	3/16	188,700 183,000 185,850	177,400# 173,200# 175,300	000 	*1/1*
(2)6110M	AV g.	3.5cr,1.0No,0,2N	C. Olda G. 178N, 3. 81Cr. 0. 91MC	190,000 190,000 190,000	7.0 7.0	379	3/16	*** 193,100 193,100	*** 175, 350 175, 350	*00	*1//T<
<u>भूत</u> ्रो। 8(3)	Avg.	5.00r,0.25C,0.1N	0.168c,0.086N,4.31Cr	181,300 185,000 183,200	6°000	66	3/16	185,800 181,700 183,750	171,300 155,550# 163,400	~4v voo	3/16 3/16
үн 93(4)	1 2 Åyg,	5 .0M n , 0.250	0.2126,0.021N,4.57Mn	195 , 300 193, 300 194, 300	2.000 2.000	ŢŢ	3/16	183,350 180,000 181,700	168,150 164,300 166,200	1900 000	*1/1×
(5)22(5)	Avg.	5.00r, 0.1N	0.022C,0.106N,5.77Cr	189,400 183,800 186,600	2 0 0 0 0 0 0	36	3/16	170,400 166,450 168,400	158 , 600 152, 250 155 , 400	000	3/16 3/16
HG1 69 (5)	5	5.00r,0.2N	0.0200,0,161N,5.870r	200,000 170,000 185,000	0.0.0 0.000	56	3/16	181,950 186,700 184,300	173,400 186,100 179,750	00 <i>1</i> 6 0 1	*1/1<
(9) IE THM	AVG.	5.0Mn,1.0W	0.0360,0.026N,4.21Mn,0.92#	181,300 200,000 190,700	4 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	137	3/16	181,000 **** 181,000	161,300 **** 161,300	7.0	3/16 >1/l#
(t)62HM	Avg.	5.0Gr,2.0V,0.250	0.174c,0.021N,6.12Cr,1.27V	190,700 188,600 189,700		178	3/16	188,900 189,400 189,150	171,100 168,950 170,000	0 0 M M N N	>1/l** 3/16
WH55(2)	Avg.	5.0¢r,1.0V,0.2N	0.0285,0.163N,5.62Cr,0.69V	214, 300 201, 300 207, 800	6.0 5.0 0 5.0 0 5.0 0 5.0 0 5.0 0 5.0 0 5.0 0 5.0 5.	8	×1/1×	200,700 201,700 201,200	182,7504	000	**1/1~

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TABLE 23. (CONTINUED) -93a-

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artis signa artis artis artis ar				Original	Propert:	Les		Rec	letermined Fro	oerties		
			· .	Tensile	El ong.,		Bend	Tensile	Yield Strength,	Elong	Minimum Bend	
Heat No.	Spec. No.	Comp Intended	osition, % Actual	Strength, ; p.s.1.(8)	6 in 1 Inch	NHA (6)	Radlus, Inch(10)	b.s.i.(8)	0.2% Offset)	Inch Inch	Inch(10)	
M0113(2)	4 10 L	2.5Gr.1.0M0.0.2N	0.0260,0.181N,2.890r,0.92Mo	191,000 185,900 188,500	6.0 6.0	378	3/16	172,600 175,150 173,900	163,200 150,950# 157,100	000 ••••	3/16 >1/l4	· ·
я́Н27(2)	AV8.	5.00r,2.0N1,0.2N	0.024c,0.145N,4.63cr,1.98m	189 , 300 200,000 194,700	4•0 3•0	2대	3/16	198,900 200,000 199,450	176,450# 191,050 183,750	200 100 100	*1/1×	n an
ин 283(7)	AV C. L	3•5Жп ₅ 1•07	0.032C,0.038N,2.43Mn,0.78V	*** 183 ,3 00 183 ,3 00	*** 8.0 8.0	245	1/4	138,350 165,500 151,900	104,000	12.0 6.0 9.0	3/14 >1/1+	· • • i
мн 293(7).	1 2 Avg.	5.0Mn,1.0V	0.0141c,0.030N,4.46Mn,0.90V	195,300 201,600*** 198,400	3.0 3.0***	302	*1//1*	150, 350 154, 400 152, 400	130,450 119,750 125,000	7.0 + 7.0 +	77/1* >7/1*	•
(7) 79292	Avg.	3•5Mn,2•0V	0.032C,0.032N,2.27Mn,1.20V	193 , 750 **** 193, 750	2.0 **** 2.0	235	т 7/г	141,100 169,950 155,500	111,850 -	9.0 7.0	3/16	
*** Ver *** Ver	le duct y litt elted	ulity. Me ductility. alloy evident in fr	acture.									

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Defective specimen. ****

Extrapolated value; curve did not reach 0.2% offset.

Data taken from Summary Report, Table 59. n n n n n n Table 67. n n n n n n Table 56.
From ternary titanium-manganese-vanadium series of alloys showing nonuniform Data not previously reported.

distribution of the alloying elements. ଞ୍ଚତ୍ର

Longitudinal li-gauge specimens 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section. Longt load. Hardness at the center of the cross section of the sheet 90° to the surface and the rolling direction. Minimum bend radius without cracking on a longitudinal specimen 3 inches long by 0.5 inch wide. Tested with surface

skin present.

as noted in the section on Ternary Alloys, were found to have nonuniform distribution of the alloying additions. These data and the chemical analyses further substantiate this conclusion.

Referring to Table 23, it will be noted that the 0.2 per cent offset yield strengths of the various alloys average about 20,000 p.s.i. lower than the ultimate strength.

As a further step in the evaluation of these selected alloys, 0.5-pound ingots were prepared using alloy hardeners for the addition of the various elements. Four of the alloys were also prepared, using pure metal additions in arc melting, as a further check on the new melting procedure.

These ingots were fabricated to l4-gauge sheet and six longitudinal tensile specimens were taken at various locations in the sheet. Two specimens each were taken from opposite sides and ends of the sheet and two adjacent specimens were from the center.

The tensile properties and hardness of the various specimens, from each alloy on which the testing has been completed, are listed in Table 24.

As noted in Table 24, a few of the specimens showed some evidence of a nonuniform cross section, but there was no indication of unmelted alloy in any of the fractures. Comparing the tensile properties for the various alloys as listed in Tables 23 and 24, it is evident that results quite comparable with/the earlier tests were obtained from a considerable number of these compositions.

This work of re-evaluating the better alloys will be continued.

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TABLE 24. EVALUATION OF SELECTED TITANIUM-BASE ALLOYS

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		ALLOYS	Prepared Usir	ng Alloy Har	deners(1)						
		Thtended	Kelative Tocation				T	loys Prepare	d Using Pu	ure Meta	Ls S
Heat No.	Spec. No.	Composition,	of Snecimen	Strength,	fin 1	NHA	Heat	Tensile Strength,	Elong.	NHA	
				hest.	тиси	<u>5</u>	No.	p.s.i.(2)	Inch	(3)	
vilio	T-X	Unalloyed	Front, side	75.400	22-0	1 7.8				•	
t	X-2	n	Front, side	75,200	22.0	178					
z :	T-T	, tt	Center	75,500	23.0	179		·			
æ	Y-2	æ	Center	76,000	21.0	178					
	I2	tt tt	Rear, side	75,000	22.0	176					
ż	22	Ŧ	Rear, side	74,000	22.0	172					
			AVG.	75,200	22.0	776					
	К-Т Э	-5Cr, 0.25C. 2.0Fe	e Front, side								
	X-2	u u	Front side								
	II	ti ti ti	Center								
	7-2-X	11 11 11	Center	•							
	- T-Z	u 11 11	Rear side								
	2-2	1 11 11	Réar, side) \ \							
. 6113 .	X-1 5	.00r.0.25C.2.0Fe	Front side	224.200	0-1	001	ין הבינוש) ?	.tac	
ŧ	X-2	=	Front, side	202.900		<u>ነ</u> የ				せ う う	
=		t nt 11	Center	204,000	1•0	133	#	181.200			
	7-7	8 8	Center	Broke in	grips	125	ŧ	207,900		901	
, t			Rear, side	214,300	0 . 1		Ħ	180,400	2-0	370	1
	2-2		Rear, side	212,400*	1°0%	121	=	181,100*	*0. v	371	
5.			. Avg.	211 , 600	1.0	h26	11	190, 700	0	39.5)
The second secon	Statistical and an and an an an and an an								•	1	

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with a three

TABLE 24. (CONTINUED)

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1				1							~/	20					-								
	re Metals	VIEW	(E)	3),7	956	387	383	106	325	364								LO3	376	374	397	380	366	382	
	Using Pu	HLONG.,	Inch	0-0	8	2.0	3.0	0.0	0.6	1 1 1								0°•0	0 0	*0 5	0 * C	0.8	1	5. 6	
	oys Prepared	Tensile Strength	p.s.i.(2)	190.800	167.300	168,600	200, 300	183,000	156,300	177,700								167.100	168,000	180,100*	200,000	196,500-	200,900	185,400	
	LLÁ	Heat.	No.	662H, ¹	=	E	z	z	=	2								7.H300		8	H	11	=	÷	
		NHN	(E)								1	1	393	1 1 2	399	211	<u>5</u>						•		
rs(1)		Elong., % in l	Inch						-		I	I	ر م	T+"0*	6.0	6.0	2°5								
Loy Hardene		Tensile Strength.	p.s.i.(2)								I	I	188,600	173,500*	177,500	170,900	177,600								
ared Using Al	Relative	Location of	Specimen	Front. side	Front, side	Center	Center	Rear, side	Rear, side	AVG	Front, side	Front, side	Center	Center	Rear, side	Rear, side	AVE.	Front, side	Front, side	Center	Center	Rear, side	Rear, side	AVG	
Alloys Prep	-	Lntended Composition.	et R	5.0Cr.0.25C.2.0Mn		N 11 33	H II H	4 41 44	2 . 2		3.5Cr, 1.0Mo,0.2N	11 11 11		11 11 11	11 († 11	1F 1F 1F		0Cr.,0.25C,0.1N	11 II	H H	tt tt tt	, tt , tt , tt ,	41 JI · JJ		
		Spec.	No.	L-X	X-2	I-Y	7-2	<u>7-7</u>	Z2		I-X	X-2	Ţ	7-7	7-7	2-2 2-2	-	Х-1 Т-Х	X-2	TX	2-7	2-1-2	2-2		
		Heat	No.								mJL13		2	11	¢	11		-				۱	•		

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TABLE 24. (CONTINUED)

Ŭ,	<u>e</u> l		-95b-		
in Ninto	VHN (3)		385 380 380 380 380 380 380 380 380 380 380	-	
vid na foll	Elong., Flong., Inch Inch		0000000 m-10000000		
bowerowd and	Tensile Strength, p.s.i.(2)		180,700* 182,200 189,900 188,000 183,000 181,600 184,600	ν.	
	Heat No.		т.H302 н н н		
	VHV (5)	397 373 373 373 373 373 373 373 373 373		385 391 1403 1120	
lers(1)	Elong. % in 1 Inch	N & & & O O O O O O O O O O O O O O O O		18864	
Alloy Harder	Tensile Strength, p.s.i.(2)	177,900 189,800 175,900 178,800 185,900 173,000 180,200		206,500 193,500 202,100 1188,600	
pared Using	Relative Location Specimen	Front, side Front, side Center Center Rear, side Rear, side Rear, side	Front, side Front, side Center Center Rear, side Rear, side Rear, side	Front, side Front, side Center Center Rear, side	
Alloys Pre	Intended Composition,		, ост ₂ 0. ТМ н н н н н н н н н н	OCr.,O.,2N 6 1 7 1 8	
	Spec. No	22222222 2221222 2211212 202222	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	5 77777 X X X X X X	
	Heat No.	1166 11 11 11 11 11 11		Lilter n T	

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TABLE 24. (CUMPINUED)

1	. 1	l I																						
	Metals	NHA	6																					
	sing Pure	Elong., % in l	THCH																					
	s Prepared Us	Tensile Strength,	1-1-T-S-1																					, , , ,
	ALLOYS	Heat	•01																					
	ļ	NHA	6	372	86 86	381	370	376	375	375	379	361	370	386	370	EL4	379	I	I	359	383	379	Lo1	380
s(1)		Elong.	TUCH	0*1	1.0	1.0	2°0	ð M	т о 10	₽•5	0*1	1°0	0-11	0°0	1.0	0-1	3 . 8	1	I	6 •0	6,0	о о	<u>ک</u>	ۍ ۲
oy Hardener		Tensile Strength,	p+S+1+14	187 , 600	187,500	187,600	177,800	175,000*	173,200*	181,500	185,600	1.84.800	192,900	194,300	195,600	196,100	191,600	ł	ł	177,400	178,300	180, 300	180,300	179,100
red Using All	Relative	Location of Snoting	opecrimen	Front, side	Front, side	Center	Center	Rear side	Rear, side	ÂVG.	Front, side	Front, side	Center	Center	Rear, side	Rear, side	Avg.	Front, side	Front, side	Center	Center	Rear side	Rear, side	AVC.
oys Prepa		tended osition,	R	n,1.0"	÷	ŧ	11	Ħ	ŧ		W,0.250	ii	ł	44	Ľ			V,0,2N	ŧ	11	11		1	••
LIA		Comp		NO. V	2	8	*	ų	**		.0Cr.2.0	11 11	11 11	8 B	11 II	11 II		.00r.1.0		44 14	11 11	ti tt	11 11	
	1	Spec.	*oN	TX	X2	ተታ	¥-2	2-1	2-2		5 T-X	X-2	Ч-Ч К-Ч	Y-2	Z1	2-2		X-1-2	X2	T-T	2-7	1-2	2-2	•
		Heat	*01	۰۰ JSO	#	44	43	4	11		".738	ų	#	ft	54	4.5	=	r Jh2	t	44	3.5	4	*	1 2

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TABLE 24. (CONTINUED)

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	}	i i	-95d-		. 1
Metals	NHA (E)		•· •	}	
sing Pure	Elong., % in 1 Inch			. 1	
s Prepared U	Tensile Strength, p.s.1.(2)	i	۱.		
Alloy	Heat No.		·		•
	VHN (3)	367 364 364 364 364 152 152 361 152 361 152 361		103399889977 103399889977 103399889977	
(1)	Elong., % in l Inch	2000000 5555 7455		000 200 200 200 200 200 200 200 200 200	
y Hardeners ⁽	Tensile Strength, p.s.i.(2)	178,100* 182,000 173,700 179,600 / 186,300 184,700		153,500 153,500 156,000 157,600 166,100 166,000 158,600	
ed Using Allo Relative	Location of Specimen	Front, side Front, side Center Center Rear, side Rear, side Rear, side	Front, side Front, side Center Center Rear, side Rear, side	Front, side Front, side Center Center Rear, side Rear, side Rear, side	Front, side Front, side Center Center Rear, side Rear, side
Alloys Prepare	Intended Composition, %	2.5Cr.J.OMo.O.2N 	5*00trs2*0Nir0.2N 11 11 11 11 11 11 11 11 11 11 11 11 11	3.5Mm, 1.0V 8 8 8 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9	Vong 1. OV
	Spec. No.	1212 112 212 212 212 212 212 212 212 21	ЧЧХ ЧЧХ ХЧЧХ ХЧТХ ХЧТХ ХЧТХ ХЧТХ ХЧТХ Х		С-2 С-2 С-2 К-2 К-2 К-2 К-2 К-2 К-2 К-2 К-2 К-2 К
	Heat. No.	177 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			-

TABLE 24. (CONTINUED)

		illare Prenare	4 Tietne Allow F	lardeners(1)						
		A THE TE ANT THE ANT THE	Relative	1-10 TOTTON TOT		ł	OTTO	ys Prepared	Using Pure	Metals
Heat	Spec.	Intended Composition,	Location of	Tensile Strength,	Flong.	NHA	Heat	Tensile Strength ₅ ,	Elong., % in l	NHA
No.	No.	5 8	Specimen	p.s.i.(2)	Inch	<u>e</u>	No.	p.s.i. (2)	Inch	(3)
7.M.:''	X-1	3.5Mn.2.0V	Front side	164,100	6•0	370				
	X2		Front, side	175,500	6 • 0	373				
, ,	1-1	11 II	Center	184,800	7.0	363				
11	X-2	11 13	Center	171,100	7•0	. 369				
15	Z-7	11	Rear, side	171,900	7-0	366				
it	2-2	2 2	Rear, side	171,900	7•0	387				
			ÅVG+	173,200	••0	371				
".JLS	T-X	S. Ohn. O. IN	Front, side	1.81,800*	* 0 * 9	379				
	X-2		Front, side	1.87,000	6 <u>.</u> 0	363				
	TX	8 2	Center	192,700	6 . 0	384				
	5-75 7-75	4	Center	195,800*	* ``	388				
	1-2	11 ff	Rear, side	190,000	ۍ م	364				
	2-2	2	Rear, side	185,800	6 •0	366				
			AVG.	188,900	5.6	374				
				in the second second						

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P IND OP m surucume IO THINNIOU 5 STIRUE EVIDENCE (ery

Chromium added as a 50% Cr entectic alloy, manganese as a 35% in master alloy, vanadium as a 35% M master alloy, iron as a 35%Fe master alloy, nickel as a 34% M eutectic alloy, molybdenum as a 40% Mo sintered compact, nitrogen as a 2.5%N sintered compact, and tungsten as compressed pellets of tungsten and titanium porders. *E

Longitudinal specimens of 14-gauge sheet, 3 inches long by 0.375 inch wide, with a. 0.250-inch-wide reduced section. 3

10-kg. load. Hardness at the center of the cross section of the sheet tensile specimen 90° to the surface and to the direction of rolling. Average of at least 5 readings. $\widehat{\mathbf{O}}$

State of the second INVESTIGATION OF REFRACTORIES FOR MELTING TITANIUM (P. D. Maddex and L. W. Eastwood)

The investigation of refractories for melting titanium was continued. Following the previously established practice, 10- to 15-gram melts were made under an argon atmosphere. After cooling, the melts and crucibles were sectioned and examined.

During the period covered by this report, the evaluation of the melts made in the tantalum carbide- and tungsten beride-lined carbon crucibles was completed. Additional melts were made in hot-pressed titanium carbide, hot-pressed zirconžum oxide (stabilized with CaO), calcium oxide, calcium oxide (fluxed with TiO_2), and aluminum oxide crucibles. The last 3 crucibles, made by compressing and sintering, had porosities of 1.6, 0.8, and 1.6 per cent, respectively.

The hot-pressed zirconium oxide crucible was wet by the titanium in only one place. However, the Vickers hardness increased from 160 for the melting stock to 440 for the melt. A survey of the melt showed that the metal near the point of wetting was 40 to 60 Vickers numbers harder than the rest of the melt. No hardness values below 400 were found. It is, therefore, remotely possible that the hardness increase may have resulted from contamination introduced at the point where the titanium wet the crucible.

This zirconium oxide crucible will be examined to show variations in composition or structure if any can be detected. Since this is the first refractory material which had areas not wet by the titanium, further experimental work will be carried out to explore the refractory fully.

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Examination of the melts made in calcium, oxide, calcium oxide fluxed with 2 per cent TiO_2 , and aluminum oxide crucibles shows that the liquid titanium reacts with these crucible materials. Hardness measurements will be made and they should indicate the extent of the reaction.

The results of the present experimental work are summarized in Table 25.

ANALYTICAL METHODS FOR TITANIUM-BASE ALLOYS

Studies on the Chemical Analysis of Oxygen in Titanium by the Chlorine-Carbon Tetrachloride Method

(E. J. Center and A. C. Eckert)

In the previous bimonthly report, there was a brief discussion of the chlorine-carbon tetrachloride method for determining oxygen. It was indicated that a detailed description of the apparatus and techniques used in the investigation of this proposed analytical method would be included in this report. The following section contains these data.

It was suggested that the decomposition of titanium by a mixture of carbon tetrachloride and chlorine might serve as the basis for a determination of small quantities of oxygen in the metal. Treatment with this gaseous mixture should distill the titanium as titanium tetrachloride, release any free oxygen present, and convert any combined oxygen to carbon monoxide or carbon dioxide. Passage of the products of this reaction over hot carbon should convert all oxygen to carbon monoxide, which could then be collected and analyzed by conventional methods. This proposal was based on reported methods for the determination of oxygen in organic compounds^(1,2), together with the suggestion that the carbon tetrachloride-chlorine mixture should readily decompose titanium metal and titanium oxides at moderately

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DATA ON REFRACTORIES TESTED AND RESULTS OBTAINED TABLE 25.

Crucible Material	Melting Atmosphere	"etting of Crucible by Ti (1)	Chemical Analysis, %	Vickers Hardness, (10-Kg.Load)	Metallographic Examination	Crucible Attack at Ti-Refractory Interface
Hot-pressed TiC (Repeat)	Argon	Yes	0•42 C	269(2)	Considerable carbide phase present (4)	No attack evident
TaC heavy lining on graphite crucible	Argon	Yes	ī	267(2)	Ditto	Ditto
"B lining on graph- ite crucible	Argon	Tes	ł	306(2)	See Footnote 5	Ditto
Hot-pressed ZrO2	Argon (bily in one spo	1	山山(3)	Not complete	Ditto
porosity - 1.6%	Argon	lery little wet	ting –	Not	complete	Some attack
CaO (fluxed with 2% TiO ₂) compressed; porosity - 0.8%	Argon	'et 1/2 diamete srucible	۲ کو	Not o	omplete	Some attack
AlgOg compressed; porosity - 1.6%	hrgon "	let only one si of crucible	l de	Not	somplete	Some attack

Indicated by meniscus on top of ingot. 60

Consequently, hardness values greater than this are caused by contamination when the test ingots were made. If no contamination occurred, melting the forged bar would lower the hardness slightly. Vickers hardness of the titanium ingot from which the forged-rod melting stock was made was 160. The carbide phase appears in a dendritic pattern uniformly distributed throughout the sample. Vickers hardness of the titanium ingot from which the forged-rod melting stock was made was 210.

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A second phase, very similar in appearance to melts made in carbide crucibles, was present; however, the constituent is much smaller and appears to have a grain-refining action. -Fig

<u>-08</u>

high temperatures.

This investigation shows that such a method involves certain difficulties which make it impractical. A variation of the method based on distillation of the titanium (as the tetrachloride) by treatment with chlorine followed by chemical analysis of the residue also appears to be impractical.

This investigation consisted in first finding satisfactory materials for construction of the necessary apparatus and then finding a set of conditions under which decomposition of the sample would run to a sufficient degree of completion.

Figures 32 and 33 show a schematic drawing and a photograph of the apparatus. The purifying train was constructed of Pyrex glass and Tygon tubing and the combustion tube was made of fused silica. Fused silica and ceramic Leco-Type HF-C boats were used to contain the sample. The various samples used included both Bureau of Mines titanium powder and Du Pont sponge titanium. Table 26 gives the typical analysis of the Bureau of Mines powder. Table 27 summarizes pertinent information concerning the runs made.^{*}

In order to check the general operation of the train and furnace, a number of qualitative runs were made. These runs are not included in Table 27.

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Element	Per Cent	
C N Fe Mg Cl Si H2 Al	0.02 0.02 0.14 0.46 0.04 0.02 0.12 0.01	

In Table 27, it is evident that the decomposition of the sample was never better than 99.7 per cent, except when temperatures above 800°C. were used. Although the residues of these high-temperature treatments are approximately 3 per cent of the sample treated, they represent less than two hundredths per cent of the original sample, since they are residues from titanium metal decompositions which took place at lower temperatures. This two-step procedure was used because it was found that the heat of reaction of the bulk sample was so great that it was difficult to avoid cracking the boats. In order to have a weighable quantity of material after the high-temperature treatment, it was necessary to start with a large quantity of original sample. Because of physical limitations, it was generally more convenient to perform the lower temperature decomposition of this large quantity in several smaller batches.

The results recorded in Table 27 show that, although the decomposition of the sample was sufficient at the higher temperatures, a significant amount of attack took place on both kinds of boats investigated.

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TABLE 27. SUMMARY OF RUNS

	and the second se			19		× .
Run No.	Average Temperature, °C.	Time, Hours	Sample	% Residue	Boat Con- sumption, Mg	
<u>]</u>	450	5 I	Bureau of Mines powder	1.2		
2	150	2	Ditto	0.8		
3	1,50	$\overline{J_1}$	n a	8.	(2)	
Ĩ.	525	្តី	1t -		25	
1 T	525	6-1/2	17	3.72 (7)	25	
6	505	6-1/2	19			
7	525 550		**			
{	550	1-1/2	**	0.0	0.0	
0	550	2-1/2			(2)	
9	550	7-1/2	Du Pont sponge	0,32		
10	575	1 I	Bureau of Mines powder		(2)	
11	650	7	Ditto	0.54	15	
12	650	6	11	0.75	3 (2)	•
13	675	5	1 1	0.72	60	
14	675	Ĩ₁-1/2		0.5	126.3	
15	850	3-1/2	Residue Run No. 6	2.7	26.2 (3)	
16	850	ייק – ב	Residue Rune Non 9	3.3	26.1 (3)	
10	0,0	,	11, 13	و در		
17	875	6	Residue Run No. 12	4	2.4	

No CCl_l used. Boat cracked during run. Leco HF-C boat used. (1) (2) (3)

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Since the attack on a silica boat probably involves a reaction in which oxygen is released, as carbon monoxide, it is likely that the quantity evolved from the walls of the silica combustion tube would cause an excessively high oxygen blank in an actual determination.

Correlation of results from spectrographic and X-ray diffraction analyses of residues from the lower temperature reactions indicates that these materials consist essentially of spinels. Only a trace of quartz is present. Magnesium is present, principally as magnesium oxide, although it is possible that some of it is a constituent of one of the spinels. No oxide of titanium is found, although some of the spinels possibly present contain titanium together with oxygen in the ratio of 1 or 2 oxygen atoms per titanium atom. Because of their close similarity in structural arrangement and lattice constant, individual spinels cannot be distinguished on the basis of X-ray evidence.^{*} The existence of spinels is probably the underlying factor in making development of the analytical method difficult, since they are extremely difficult to decempse. Schardson and co-workers⁽³⁾ found that in a stream of chlorine at 1000°C. for 35 hours a similar spinel lost only 16.5 per cent of its weight.

On the residue from reactions with chloring along, chunical analyses account for a large percentage of the oxygen expected to be found in the samples. However, uncertainty exists us to loss of exygen and the oxidation state of titanium associated with exygen. Therefore, without additional information, it is impossible to establish a definite factor for calculating exygen coul at from the obtained analyses.

* The following spirels could be present: willer(1; F-Ti(1; whTiC1.

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On the basis of results discussed above, it is concluded that the carbon tetrachloride-chlorine method is impractical for the determination of small amounts of oxygen in titanium metal because a significant amount of the oxygen is combined in spinels and, thus, conditions are required for decomposition that cause excessive attack on the boat and the walls of the combustion tube. The chlorine-chemical analysis method fails because of the uncertainty as to the Ti-O relationship in the original sample and in the chlorination residue.

The Analysis of Titanium for Oxygen by Vacuum-Fusion Methods

In the previous bimonthly report, the technique used at Battelle and the results obtained in the analysis of titanium for oxygen by the vacuum-fusion method were described. In the above report, it was indicated that samples of iodide titanium with known amounts of oxygen added as TiO_2 were being prepared and would be sent to Dr. G. Derge, of the Carnegie Institute of Technology, for vacuum-fusion analysis in his latoratory.

This section describes (1) the preparation of the standard samples, (2) the apparatus and technique used by Dr. Derge, and (3) the results of analysis.

Freparation of the Standard Jamples

Samples with known weights of oxygen added as TiC₂ were prepared from iodide titanium crystal bar as follows:

V. A. Aluise, et al., anal. Chem. 14, 317-1, (1917).
 (2) A. A. Linerstein and F. J. Aliji, anal. Chem. 21, 500-7 (1918).
 (3) H. V. Bichardson, et al., Frans. Folt. Conv. Lett. 11, 195-605 (1918).

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A small capsule of iodide titanium was prepared by drilling a hole in the iodide titanium, adding a weighed amount of dried C.P. TiO2 in the hole, and plugging the hole with a machined rod of iodide titanium. The prepared standard was then double melted in argon. Before melting, the system was evacuated to about 40 microns three times, flushing to about 60-cm.pressure with dry, pure (99.95%) argon between each evacuation. Then, the system was evacuated to 0.1 micron, and 10-cm. pressure of argon was admitted. The system was evacuated to 0.1 micron again and 10-cm. pressure of argon was admitted. The arc was then struck against the copper crucible away from the charge. Melting was complete in 10 to 15 seconds. after cooling, the button was turned over and the melting procedure was repeated.

Ten standard samples of iodide titanium with known additions of oxygen added as TiO₂ were prepared in this manner. After melting, the specimens were ground and the Vickers hardness determined on the top and bottom of each specimen. Table 28 contains the data pertinent to the preparation of the standard samples. Figure 34 shows the relation of the Vickers hardness and the calculated oxygen content. The correlation between hardness and oxygen content shows remarkably little variation. These samples were assigned numbers, as indicated in Table 28, and sent to Dr. Derge for analysis.

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TABLE 28. OXYGEN STANDARDS FOR ANALYSIS

Sample No.	Total ™t. of Sample Before Melting, Grams	Total [†] /t. of Sample After Double Melting, Grams	Loss or Gain in "Teight During Melting, Grams	TiO ₂ Added, Grams	Per Cent Oxygen in Sample*	VH Top	N(1) Bottom	Designation Assigned to Sample Sent to Dr. Derge
i	7 0216	7.0231	-0.0015	None	None	07 8	07.0	
$\frac{1}{2}$	7.7914	7.7952	+0.0008	None	None	89.1	101.3	· 5
3	7.9997	8.0135	+0.0138	0.0038	0.019	108	106	3
Ĩ4	8,0045	8.0026	-0.0019	0.0043	0.0215	106	106	9
5	8.0176	8.0163	-0.0013	0.0201	0.100	164	168	ì
6	8.0205	8.0202	-0.0003	0,0201	0.100	153	156	4
7	8.0516	8.0500	-0.0016	0.0516	0.257	206	214	7
8	8.0486	8.0456	-0.0030	0 .0 488	0.243	218	216	10
9	7.9989	7.9970	-0.0019	0.0999	0,500	296	281	6
10	8.0004	7.9950	-0.0054	0.1020	0.510	286	275	8

* Calculated from final weight of sample after melting. (1) Average of 5 readings.

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TANIUM SAMPLES

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The Derge Method of Analyzing for Oxygen in Titanium by Vacuum Fusion (M. W. Mallett)

Apparatus and General Technique. The modification of the vacuumfusion method used by Dr. G. Derge, at the Carnegie Institute of Technology, for the determination of oxygen in titanium may be described as follows: Figure 35 is a schematic diagram of the Carnegie vacuum-fusion apparatus. Specimens are introduced into the evacuated system by the mercury lift, A, and stored in arm, B. A tapered, ground cap permits nonmagnetic samples to be put in the storage arm with the system at atmospheric pressure. This assembly is connected to the Pyrex furnace head.

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The furnace assembly is shown in detail in Figure 36. The techniques have been described rather completely by Derge, Peifer, and Richards(4) and Derge(5). Because most of the details of the analytical train and procedure are also given in the first paper, they will be dealt with but briefly here.

Two mercury diffusion pumps, H and I, are used to remove rapidly the evolved gases from the furnace. Pumps, K and L, also aid in the evacuation but are used primarily to pack the gases into the analytical train. Later, they circulate the gas sample through the corper oxide tube, Q, and the freezing trap, M. The pressure in the collection system is read on the dibutyl phthalate manometer, G.

(4) Metals Technology, T.P. 2362, June, 1948.
(5) Journal of Fetals, Vol. 1, pp. 31-33, 1949.

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The completion of the gas evolution from the metal sample is shown by a reduced rate of pressure rise, equivalent to that for blank gases. At this time, stopcock, 2, is closed and the gas is measured in the small volume, to stopcock, 5; the medium volume, to stopcock, 6; cr the large volume, to stopcock, 7; depending upon the amount of gas extracted. After measurement, the gas is oxidized (CO to CO₂ and H₂ to H₂O) in the copper oxide tube, and the H₂O is frozen out in trap, M, by means of a dry ice-acetone mixture. After 10 minutes, the residual gases (CO₂ and N₂) are collected, in 5 minutes, in a suitable volume and measured. Liquid nitrogen is then put on the trap, M, and the CO₂ frozen out in a period of 5 to 10 minutes. The residual gas is then pumped into a collection volume and measured.

The volumes of H_2 and CO_2 are determined from the calibrated volumes of the system and the drop in pressure during selective freezing. The residual gas is nitrogen.

The Analysis of Titanium. One or more titanium samples are stored in the Pyrex storage arm along with a supply of carbon-saturated iron and lumps of tin. For a 1-gram sample of titanium, 25 grams of iron and 5 grams of tin are used. The furnace assembly is degassed by heating at 1950-2200°C. for h to 8 hours. Then a 5-minute blank at this temperature indicates the system to be sufficiently degassed, the temperature is dropped to 1800°C. and 25 grams of iron are dropped into the crucible and degassed. A 10-minute blank is taken at 1800°C. A second blank is taken at 1700°C. Five grams of tin are dropped at 1600°C. and degassed, and a third blank is taken. The blanks at 1600°C, and 1700°C, are usually similar in volume.

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The 1800°C. blank is higher. A curve of the blank gas volumes per unit time versus temperature is drawn and used in correcting the gas volumes collected from the sample.

The sample of titanium is then maneuvered into the furnace by means of an iron slug moved by a small permanent magnet. If gas evolution at 1400°C. is not greatly higher than that of the empty crucible, the temperature is gradually raised to 1800°C., hesitating at intermediate temperature where a marked increase in the rate of gas evolution is noted. The extraction is usually complete in 30 to 60 minutes. The gas is measured and analyzed as indicated above.

During analysis of the gas, the furnace is exhausted into the storage bulb, J. Upon completion of the analysis, the system is connected to the fore pump, and 5 minutes allowed for removing residual gas from the analytical train and the gas from bulb, J.

In preparation for the second sample, an additional 25 grams of iron are dropped and degassed and the entire procedure of determining blanks and dropping additional (5 grams) tin is repeated.

Most of the hydrogen is evolved immediately upon melting the sample. If the sample contains considerable oxygen, a burst of CO is noted at 1600-1630°C.

Notes and Comments. The dry ice-acetone mixture is aspirated, lowering its temperature to -105°C., before placing on the trap. However, it returns almost immediately to about -80°C. when placed around the trap.

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The copper oxide is made by oxidizing 1/4" by 1/4" squares of dopper gauze by heating in air.

The furnace tube is cleaned with acid each day. The crucible and graphite funnel are discarded. Other graphite parts and powder are re-used without treatment.

The speed of the furnace pumps and the conductance of the connecting tubing down to the melting crucible is comparable or inferior to that of the Battelle vacuum-fusion outfit.

Dr. Derge uses a split graphite container for his graphite insulating powder, whereas a beryllia thimble is used for that purpose at Battelle. Using a graphite shell may result in a slightly lower blank. A similar split graphite shell tried at Battelle showed excessive heating, apparently because the frequency (300 Kc) of the Lepel converter is much higher than that of an Ajax converter (30 Kc).

The apparatus at Carnegie Institute uses a water-cooled furnace tube, whereas an air-cooled furnace tube is used at Battelle. This variation between the equipment at the two laboratories may possibly make a difference in the gettering effect of vaporized metal. In fact, when the Battelle and Carnegie vacuum-fusion apparatus and techniques are compared in their entirety, the difference in cooling the furnace tube appears to be the only factor which might have significance.

The Results from Vacuum-Fusion Analysis. The analytical results reported by Dr. G. Derge for the ten samples are listed in Table 29, together with the calculated oxygen contents. With reference to these analyses, Dr. Derge had the following comments:

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"Samples were cut from the discs you submitted in pie-shaped segments so as to average effects of segregation as much as possible.

"Sample 1-1 was analyzed We were aware of the fact that our blank was unsatisfactory, but it seemed wisest to proceed with the analysis..... We do not believe this result has anything but qualitative value. A reasonable correction for the additional gas collected at the end of this analysis would bring this result into agreement with Sample 1-2, but we prefer to regard this as fortuitous. It will also be observed that the original calculations for Sample 1-3 show an unusually high nitrogen value, indicating that the copper oxide catalyst was exhausted during this analysis. If the nitrogen results from Samples 1-1 and 1-2 are averaged and Sample 1-3 is recalculated on this basis, the agreement with Samples 1-2 becomes satisfactory for oxygen. We believe that this is a legitimate correction of the data.

"Sample 6-2 was analyzed in the same crucible as Sample 6-1 by adding additional iron and tin and repeating the normal analytical procedure, similarly for Samples 8-1 and 8-2. In both cases, the second sample was considerably lower than the first and this practice was discontinued at this point. All other samples were analyzed in individual crucibles."

The analytical data are listed in Table 29 and graphically represented by Figure 37. It will be noted that excellent checks were obtained by duplicate analysis of the same specimen. With the exception of the sample containing 0.257 <u>added</u> oxygen, all the analytical results are within ± 0.05 per cent of the intended analysis, i.e., the added oxygen plus the residual oxygen. The relationship of hardness to the per cent added oxygen is much more precise than the relationship of hardness to the oxygen content by analysis. This is shown by comparing Figures 37 and 38.

"hile the accuracy of ±0.05 per cent oxygen leaves something to be desired, it should be noted that the intended analysis of the "standard" samples may be less accurate than anticipated. Likewise, the disparity between the intended analysis and analytical result obtained on the sample

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		Sample No. Assigned to the Specimen		a y carrange (staling y		<u>بەرد</u> مىنىپە بەرىتەرچىن	
Battelle Sample	Per Cent Oxygen	Before Sending to	Run	Vacuum-Fusion Analysis, %			Average VHN of
1900 	AQQEQ	Dr. Derge	NQ.	H	. 0 . 	<u>N</u>	Sample
l	None	2	1 2	0.0121 0.0125	0.01.35 0.0142	0.0007 0.0018	98
			Avg.	0.0123	0.0139	0.0013	·
2	None	5	1	0.0131	0.041	0,0002	95
3	0.019	3	1	0.0130	0.088	0.0028	107
4	0.0215	9	l	0.0088	0.083	0.0063	106
5	0.100	1	l 2 3 recalc.	0.0106 0.0102 0.01149	0.042* 0.094 0.056** 0.080	0.0033* 0.0041 0.045**	166
•		,	Avg.	0.0126	0.087	0.0039	
6	0.100	4	l 2 Avg.	0.0136 0.0128 0.0132	0.124 0.146 0.135	0.0005 0.0008 0.0006	155
7	0.257	7	l 2 A v g.	0.0097 0.0108 0.0103	0.094 0.136 0.125	0.0023 0.015 0.0087	210
8	0.243	10	l 2 A v g.	0.0106 0.0234 0.0170	0.304 0.307 0.306	0.01); 0.017 0.016	217
9	0.500	6	l 2 Avg.	0.0146 0.0127 0.0137	0.562 0.513*** 0.538	0.0016 0.0015 0.0016	289
10	0,510	8	1 2 3 A vg.	0.0098 0.0089 0.0159 0.0115	0•547 0•485*** 0•518 0-517	0.0007 0.0008 0.0008 0.0008	280

Unsatisfactory blank, poor value. See discussion. Result probably low. See text. *

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containing 0.257 per cent added oxygen remains unexplained. It is concluded that, even though further work is required to produce greater accuracy and reliability, the techniques developed by Dr. Derge represent a real contribution to the technology of titanium.

FUTURE WORK

The evaluation of the more promising high-strength alloys will be continued. However, before selecting an alloy composition that will be prepared in relatively large ingots, several selected alloys will be prepared as 2-pound ingots to evaluate the fabrication characteristics of these alloys when prepared as intermediate sized ingots.

Preparation and study of 0.5-pound binary, ternary, and more complex alloys will be continued.

The investigation of refractory materials for holding molten titanium will be continued. Test crucibles of tantalum silicide, tungsten silicide, and molybdenum silicide have been made by siliconizing machined crucibles of each of the metals. Hot-molded crucibles of molybdenum carbide and thorium oxide have been received from the Norton Company. A magnesium oxide crucible has been prepared by pressing and sintering fine powder. Tests will be made in all of these crucibles.

Hot-molded crucibles of tantalum carbide, both TaC and Ta₂C, titanium nitride, zirconium nitride, and boron carbide are being prepared by the Norton Company for evaluation. Titanium boride, tantalum boride, zirconium boride, tungsten boride, and molybdenum boride powder have been ordered. The Norton Company will attempt to prepare hot-molded crucibles from these materials and from tungsten and molybdenum silicides now on hand.

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Quotations are being obtained from the Norton Company on hotpressed crucibles made with a mixture of carbides such as 75 per cent silicon carbide-25 per cent boron carbide, and 75 per cent silicon carbide-25 per cent zirconium carbide, and on hot-pressed crucibles of titanium boride, zirconium boride, chromium boride, and boron nitride. The present attempt to locate a supplier of refractory materials such as titanium, zirconium, magnesium, and thorium sulphide, cerium, vanadium, and thorium nitride, and tantalum, zirconium, and titanium silicide will be continued.

The results obtained with the zirconium oxide (lime stabilized) crucible indicate that double-oxide compounds may be promising as refractories for titanium. Therefore, compounds such as thorium zirconate, calcium thorate, and zirconium pyrophosphate, and other double oxides or combinations such as TiO-CaO, ThO₂-ZrO₂, TiO₂-ZrO₂, ThO₂-CaO, and ZrO₂-CaO, will again be considered. Dense crucibles of these materials will be obtained and tested.

Parts of the "standard" titanium oxygen samples sent to Dr. Derge were returned. These will be sent to Mr. Chapin, at the Naval Research Laboratory, where they will be subject to redetermination of the oxygen content. This work will be done gratis.

The data from which this report was prepared are recorded in the following B.M.I. Notebooks:

No. 3912, pp. 14 to 30, inclusive; No. 4112, pp. 91-98, inclusive; No. 4728, pp. 6 to 54, inclusive; No. 733, pp. 53 to 95, inclusive; No. 4461, pp. 18 to 22;-32 to 34; 39 to 60; 63 to 84; and 90 to 100.

November 7, 1949

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Research and Development on Titanium Alloys	(None)
AUTHOR(S) : Simmons, O. W.; Greenidge, C. T.; Craighead, C. M; and others	ORIG. ADENCY NO.
PUBLISHED BY : (Same) for AMC, Wright-Patterson Air Force Base, Dayton, O.	PUBLISHING AGENCY NO.
Oct'49 Unclass. U.S. English 120 tables, diagrs, graphs	(Same)
ABSTRACT: Progress is reported in development of titanium alloys. Phase relations in titanium manium and titanium - 0 to 10% nickel alloys were investigated. Nickel was found it alpha-phase field and to lower the beta solvus line. The range of compositions inve titanium-silver systems was extended to 5% silvet, and titanium-beryllium alloys of beryllium were investigated. Additions of 1 and 2% columbium or tantalum to Proc the tensile strength and lowered the ductility of Process A titanium. Ternary alloy carbon, manganese and vanadium, and molybdenum and tungsten, prepared by addin during arc melting, had quite erratic tensile properties when tested after fabricativ were completed on evaluation of "hot-pressed" titanium carbide and graphite cruci	n - 0 to 1% ger- to limit markedly (sstigated in the bin containing $0, i$ to 1% ess A metal increa s of managese and ig the pure metals on to sheet. Tests ibles.
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