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Second Progress Report Covering the Period 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, O.
(Same) for AMC, Wright-Patterson Air Force Base, Dayton, O.

2nd

Oct'49 Unclass. U.S. English 120 photos, tables, diagrs, graphs (Same)

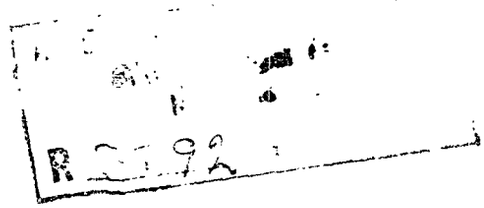
Progress is reported in development of titanium alloys. Phase relations in titanium - 0 to 1% germanium and titanium - 0 to 10% nickel alloys were investigated. Nickel was found to limit markedly the alpha-phase field and to lower the beta solvus line. The range of compositions investigated in the binary 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 ductility of Process A titanium. Ternary alloys of managese and carbon, manganese and vanadium, and molybdenum and tungsten, prepared by adding the pure metals during arc melting, had quite erratic tensile properties when tested after fabrication to sheet. 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 RESEARCH
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,

L. W. Eastwood

L. W. Eastwood

LWE:mr
Enc.(31)

SECOND PROGRESS REPORT

COVERING THE PERIOD SEPTEMBER 1 TO OCTOBER 31, 1949

on

RESEARCH AND DEVELOPMENT ON TITANIUM ALLOYS

Contract No. 33(038)-3736

to

WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO

BATTELLE MEMORIAL INSTITUTE

October 31,1949

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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

to

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.

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 extended.

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 high-strength 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 TiO_2, and aluminum oxide. The stabilized zirconium oxide 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 TiO_2, 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 ingots.

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. W. 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".

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.

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 manganese-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.

Two 2-pound ingots of the 5 per cent chromium, 2 per cent iron, and 0.25 per cent carbon 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

(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.

With 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.

TABLE 16. PHASES PRESENT IN BINARY TITANIUM-GERMANIUM ALLOYS AT TEMPERATURES INDICATED - PROCESS A METAL BASE

Heat No.	Intended Composition, %	As Hot Rolled 1450°F.	Heat Treated 1450°F.	Heat Treated 1550°F.	Heat Treated 1600°F.	Heat Treated 1650°F.	Heat Treated 1700°F.	Heat Treated 1750°F.
WH180	Unalloyed	α	α	α	80 - 20 α + β	15 - 85 α + β	β	β
WH170	0.1 Ge	α	α	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
WH168	1.0 Ge	α	α	90 - 10 α + β	70 - 30 α + β	30 - 70 α + β	β	β

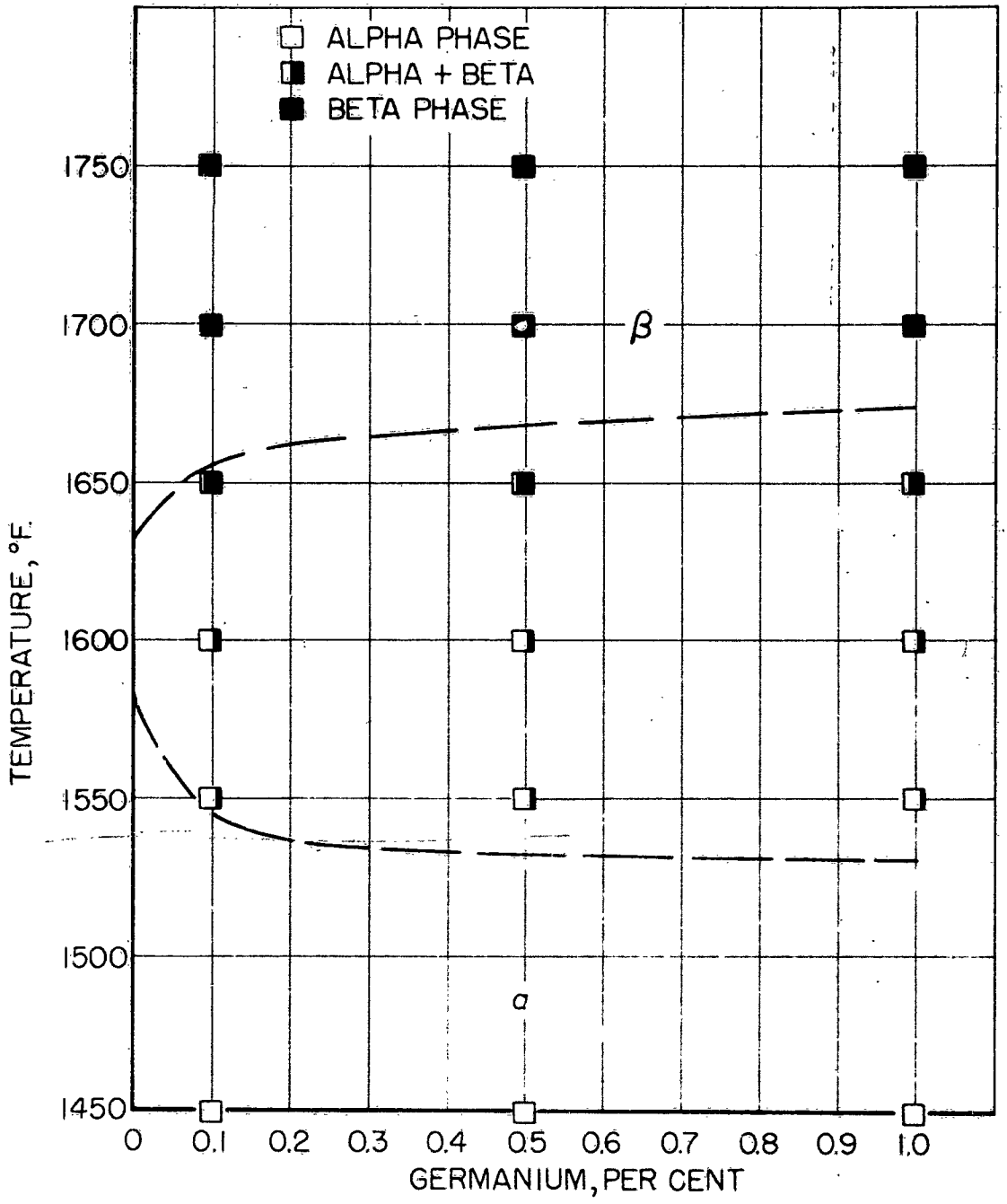

FIGURE 19. TENTATIVE DIAGRAM SHOWING TRANSFORMATION RANGE OF TITANIUM-0 TO 1.0 PER CENT GERMANIUM ALLOYS MADE FROM PROCESS A METAL

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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 (γ) 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 eutectoid 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

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.

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

Heat No.	Composition, % Intended	Actual				As Hot Rolled at 1450°F.	As Hot Rolled Aged 4 Hrs.-750°F.	Heat Treated 1450°F.	Heat Treated 1550°F.	Heat Treated 1600°F.	Heat Treated 1650°F.	Heat Treated 1700°F.	Heat Treated 1750°F.
		C	H	N									
WH247	1.75 Ni	-	-	-	-	α + β	α + β	70 - 30 α + β	50 - 50 α + β	20 - 80 α + β	β	β	β
WH246	2.5 Ni	-	-	-	-	α + β	α + β	60 - 40 α + β	40 - 60 α + β	β	β	β	β
WH245	3.5 Ni	-	-	-	-	50 - 50 α + β	50 - 50 α + β	40 - 60 α + β	15 - 85 α + β	β	β	β	β
WH244	5.0 Ni	-	-	-	-	α + β	α + β	15 - 85 α + β	β	β	β	β	β
WH241	7.5 Ni	-	-	-	-	β + γ*	β + γ*	β + Tr. γ*	β	β	β	β	β
WH237	10.0 Ni	-	-	-	-	β + γ*	β + γ*	β + γ*	β + γ*	β + γ*	β + γ*	β	β
WD1**	10.0 Ni	11.4 Ni	0.07	0.029	0.59	β + γ*	-	β + γ*	β + γ*	β + γ*	β + γ*	β + γ*	β + γ*

* γ indicates nickel-rich phase.
** Data taken from Summary Report - Part III, page 196.

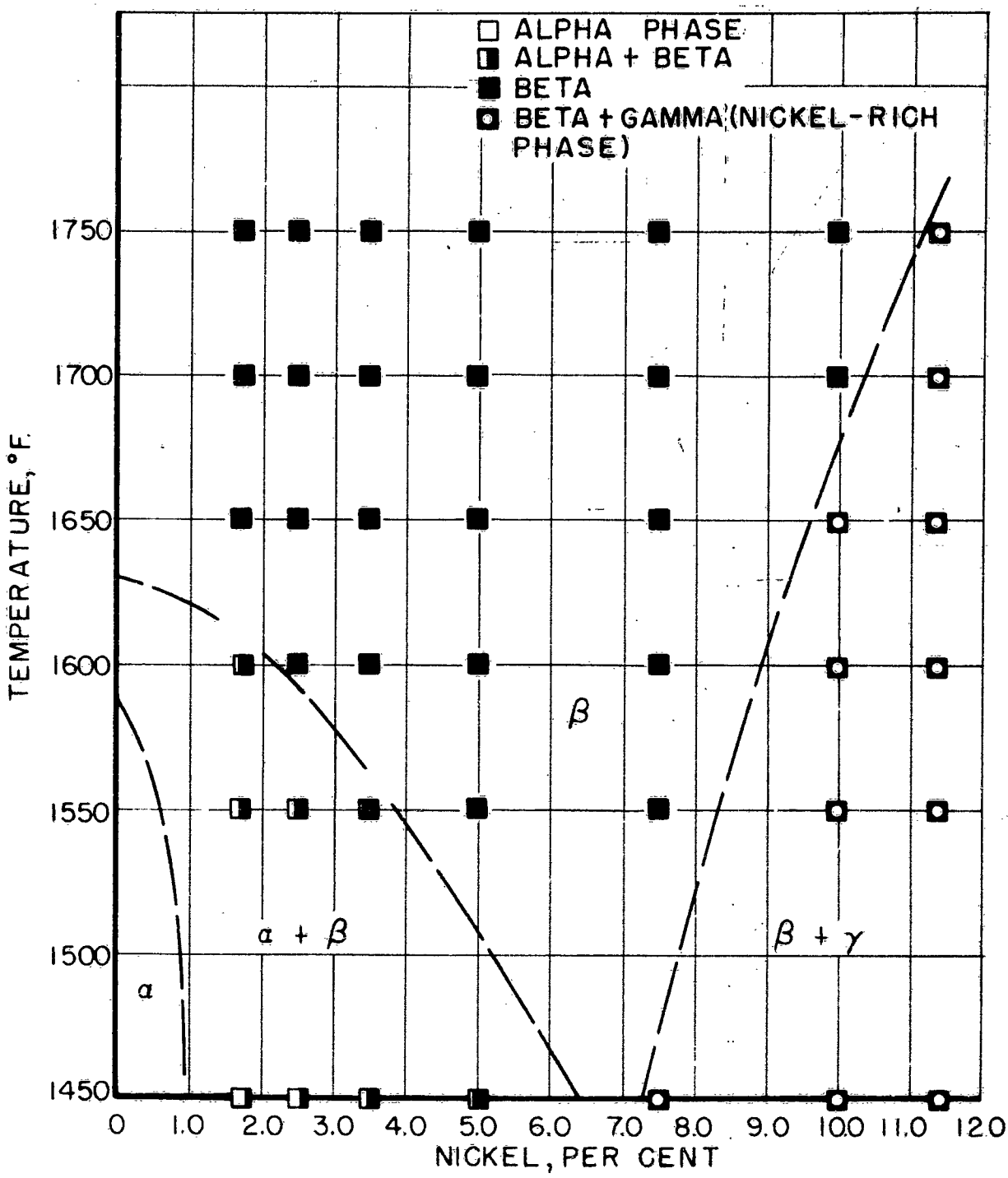

FIGURE 20. TENTATIVE DIAGRAM SHOWING TRANSFORMATION RANGE OF TITANIUM-O TO 10 PER CENT NICKEL ALLOYS MADE FROM PROCESS A METAL.

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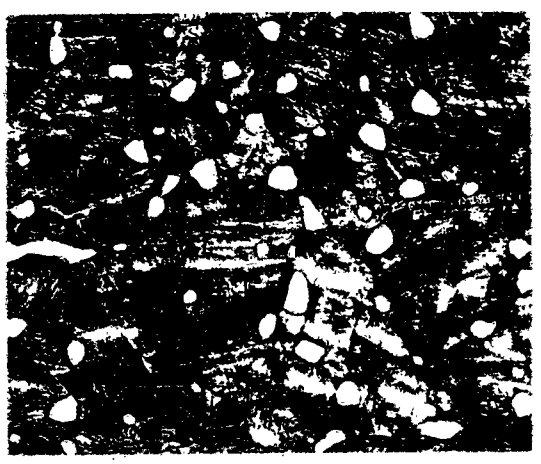
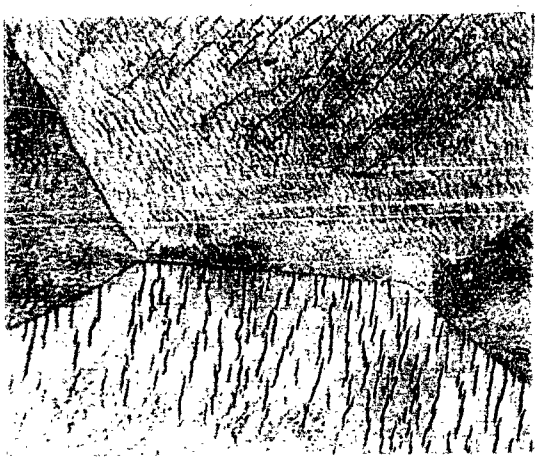
500X 65811

Figure 21. Heat "H245, 3.5% Ni. Quenched from 1600°F. in the beta field. Acicular alpha (transformed beta) is shown.



500X 65810

Figure 22. Heat "H245, 3.5% Ni. Quenched from 1550°F. in the alpha + beta field. Islands of alpha (light) appear in an acicular alpha (transformed beta) matrix.



500X 65809

Figure 23. Heat "H241, 7.5% Ni. Quenched from 1550°F. in the beta field. The nickel-rich phase, shown in Figure 24, is absent when the specimen is quenched from 1550°F.

500X 65808

Figure 24. Heat "H241, 7.5% Ni. Quenched from 1450°F. in the beta + gamma field. The light-colored constituent is the nickel-rich phase in a beta matrix.

Typical structures observed in binary titanium-nickel alloys made from Process A metal.

TABLE 19. HEAT-TREATING AND AGING DATA FOR BINARY ALLOYS OF TITANIUM WITH SILVER, BERYLLIUM, ZIRCONIUM, COLUMBIUM, OR TANTALUM PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	Vickers Hardness After Indicated Treatment(1)								Minimum Bend Radius After Indicated Treatment(2)							
		As Hot Rolled (3)	As Hot Rolled Aged 4 Hrs. - 750°F. (4)	Heat Treated 1450°F. (5)	Heat Treated 1550°F. (6)	Heat Treated 1600°F. (7)	Heat Treated 1650°F. (8)	Heat Treated 1700°F. (9)	Heat Treated 1750°F. (10)	As Hot Rolled (3)	As Hot Rolled Aged 4 Hrs. - 750°F. (4)	Heat Treated 1450°F. (5)	Heat Treated 1550°F. (6)	Heat Treated 1600°F. (7)	Heat Treated 1650°F. (8)	Heat Treated 1700°F. (9)	Heat Treated 1750°F. (10)
							Titanium - Silver										
WH248	Unalloyed	203	216	191	196	196	194	209	222	3/16	3/16	3/16	3/16	1/16	1/16	>1/4*	>1/4*
WH254	2.5 Ag	218	221	176	188	192	206	227	231	3/16	3/16	3/16	3/16	3/32	3/16	3/16	3/16
WH255	3.5 Ag	201	201	182	187	201	204	225	241	1/16	1/16	1/16	3/16	1/16	1/16	3/16	3/16
WH256	5.0 Ag	210	218	168	187	186	191	216	219	3/16	3/16	3/16	3/16	3/16	3/32	1/16	1/16
							Titanium - Beryllium										
WH262	Unalloyed	201	203	179	179	180	188	220	215	3/16	3/16	1/16	1/16	3/16	3/16	3/16	3/16
WH267	0.10 Be	206	198	174	191	200	221	211	217	1/16	1/16	1/16	3/16	3/16	3/16	>1/4*	>1/4*
WH266	0.15 Be	215	224	206	237	210	238	249	247	3/16	1/16	3/16	3/16	3/16	3/16	>1/4*	>1/4*
WH265	0.25 Be	215	213	183	207	266	241	288	255	3/16	3/16	3/16	3/16	3/16	3/16	>1/4*	>1/4*
WH264	0.50 Be	220	221	185	214	276	299	293	281	3/16	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**
WH263	1.0 Be	230	228	199	210	249	241	264	245	>1/4*	3/16	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**
							Titanium - Zirconium										
WH248	Unalloyed	203	216	191	196	196	194	209	222	3/16	3/16	3/16	3/16	1/16	1/16	>1/4*	>1/4*
WH253	1.0 Zr	202	212	171	186	183	212	207	213	1/8	1/8	1/16	1/16	1/16	1/16	3/16	>1/4*
WH252	2.5 Zr	218	223	184	187	202	225	230	227	3/16	3/16	1/16	1/16	3/16	1/16	>1/4*	>1/4*
WH251	3.5 Zr	210	218	177	191	198	192	208	231	3/16	3/16	1/16	3/16	1/16	1/16	>1/4**	>1/4**
WH250	5.0 Zr	249	222	216	254	219	254	254	249	3/16	3/16	3/16	3/16	3/16	3/16	>1/4**	>1/4**
WH249	10.0 Zr	249	235	210	225	242	266	274	290	>1/4*	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**
							Titanium - Columbium										
WH248	Unalloyed	203	216	191	196	196	194	209	222	3/16	3/16	3/16	3/16	1/16	1/16	>1/4*	>1/4*
WH270	1.0 Cb	197	205	160	172	180	182	216	210	3/16	3/16	1/16	3/16	3/16	3/16	3/16	>1/4*
WH269	2.0 Cb	236	219	221	247	233	232	236	232	3/16	3/16	3/16	3/16	3/16	3/16	3/16	3/16
							Titanium - Tantalum										
WH248	Unalloyed	203	216	191	196	196	194	209	222	3/16	3/16	3/16	3/16	1/16	1/16	>1/4*	>1/4*
WH273	1.0 Ta	201	201	177	179	194	200	207	207	3/16	3/16	3/16	3/16	3/16	3/16	3/16	>1/4*
WH272	2.0 Ta	220	220	194	206	198	225	221	230	3/16	3/16	3/16	3/16	1/16	3/16	3/16	3/16

Footnotes on following page.

TABLE 18. PROPERTIES OF BINARY ALLOYS OF TITANIUM WITH SILVER, BERYLLIUM, ZIRCONIUM, COLUMBIUM, OR TANTALUM PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	As Hot Rolled(1) Tensile Strength,(3) p.s.i.	Elong., % in 1 Inch	VHN(4)	Minimum Bend Radius,(5) Inch	Aged 4 Hrs. at 750°F.(2) Tensile Strength,(3) p.s.i.	Elong., % in 1 Inch	VHN(4)	Minimum Bend Radius,(5) Inch
				Titanium - Silver					
WH248	Unalloyed	95,600	21.0	203	3/16	94,700	20.5	216	3/16
WH254	2.5 Ag	93,200	21.5	218	3/16	91,200	20.0	221	3/16
WH255	3.5 Ag	82,700	20.0	201	1/16	83,500	20.0	201	1/16
WH256	5.0 Ag	91,900	20.5	210	3/16	92,900	19.0	218	3/16
				Titanium - Beryllium					
WH262	Unalloyed	85,700	21.5	201	3/16	95,900	20.0	203	3/16
WH267	0.10 Be	91,100	18.0	206	1/16	90,000	18.0	198	1/16
WH266	0.15 Be	96,800	16.5	215	3/16	92,800	15.5	224	1/16
WH265	0.25 Be	100,000	14.0	215	3/16	88,100	14.5	213	3/16
WH264	0.50 Be	95,900	13.5	220	3/16	94,400	14.5	221	3/16
WH263	1.0 Be	97,300	12.0	230	>1/4*	90,100	9.5	228	3/16
				Titanium - Zirconium					
WH248	Unalloyed	95,600	21.0	203	3/16	94,700	20.5	216	3/16
WH253	1.0 Zr	92,500	18.5	202	1/8	94,100	21.0	212	1/8
WH252	2.5 Zr	98,300	18.0	218	3/16	102,400	17.5	223	3/16
WH251	3.5 Zr	96,400	18.5	210	3/16	103,500	17.0	218	3/16
WH250	5.0 Zr	104,300	16.5	249	3/16	117,300	13.5	222	3/16
WH249	10.0 Zr	118,800	15.0	249	>1/4*	114,300	15.0	235	3/16
				Titanium - Columbium					
WH248	Unalloyed	95,600	21.0	203	3/16	94,700	20.5	216	3/16
WH270	1.0 Cb	92,700	19.5	197	3/16	90,000	18.0	205	3/16
WH269	2.0 Cb	108,900	16.5	236	3/16	97,500	18.5	219	3/16
				Titanium - Tantalum					
WH248	Unalloyed	95,600	21.0	203	3/16	94,700	20.5	216	3/16
WH273	1.0 Ta	89,900	21.0	201	3/16	90,300	20.0	201	3/16
WH272	2.0 Ta	99,100	18.0	220	3/16	104,700	17.0	220	3/16

Footnotes on following page.

Footnotes for Table 18:

(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 14-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.

* Some ductility.

** Very little ductility.

Footnotes for Table 19:

(1) 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.

(3) 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.

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 titanium-silver alloys respond to solution heat treatment. Aging the as-hot-rolled alloys for 4 hours at 750°F. did not increase the hardness.

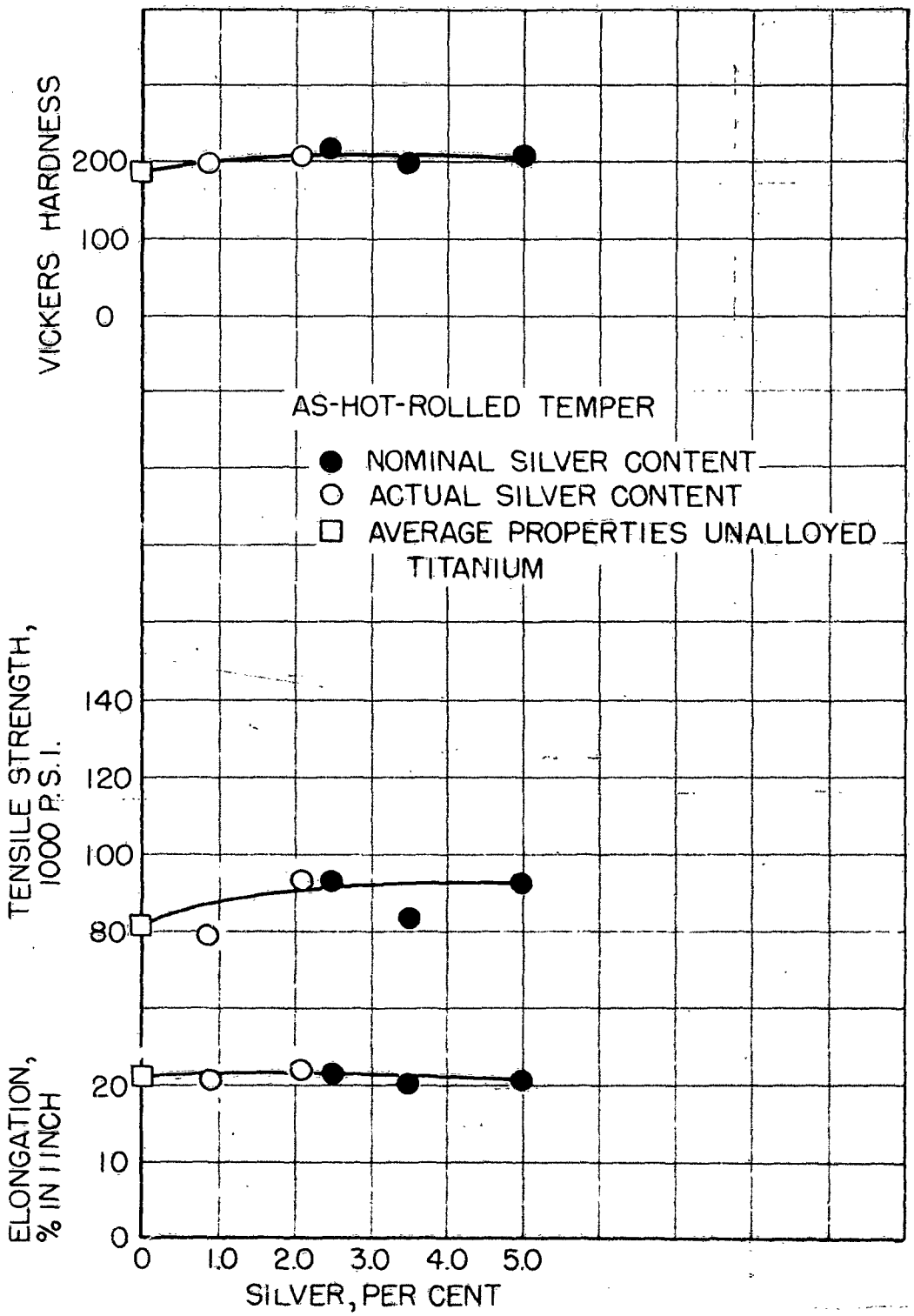


FIGURE 25 EFFECT OF SILVER ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET

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

Heat No.	Composition, % Intended	Actual	C	N	O	As Hot Rolled at 1450°F.	As Hot Rolled Aged 4 Hrs.-750°F.	Heat Treated 1450°F.	Heat Treated 1550°F.	Heat Treated 1600°F.	Heat Treated 1650°F.	Heat Treated 1700°F.	Heat Treated 1750°F.
WC77*	Unalloyed		0.04	0.026	0.10	α	-	α	95 - 5 α + β	85 - 15 α + β	β	β	β
WC94*	1.0 Ag	0.87 Ag	0.03	0.085	0.13	α	-	α	α	90 - 10 α + β	β	β	β
WC96*	2.0 Ag	2.12 Ag	0.02	0.035	0.08	α	-	α	95 - 5 α + β	80 - 20 α + β	10 - 90 α + β	5 - 95 α + β	β
WH254	2.5 Ag	-	-	-	-	α	α	α	90 - 10 α + β	70 - 30 α + β	60 - 40 α + β	β	β
WH255	3.5 Ag	-	-	-	-	α	α	α	95 - 5 α + β	60 - 40 α + β	30 - 70 α + β	β	β
WH256	5.0 Ag	-	-	-	-	α	α	α	α + Tr. β	60 - 40 α + β	40 - 60 α + β	β	β

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

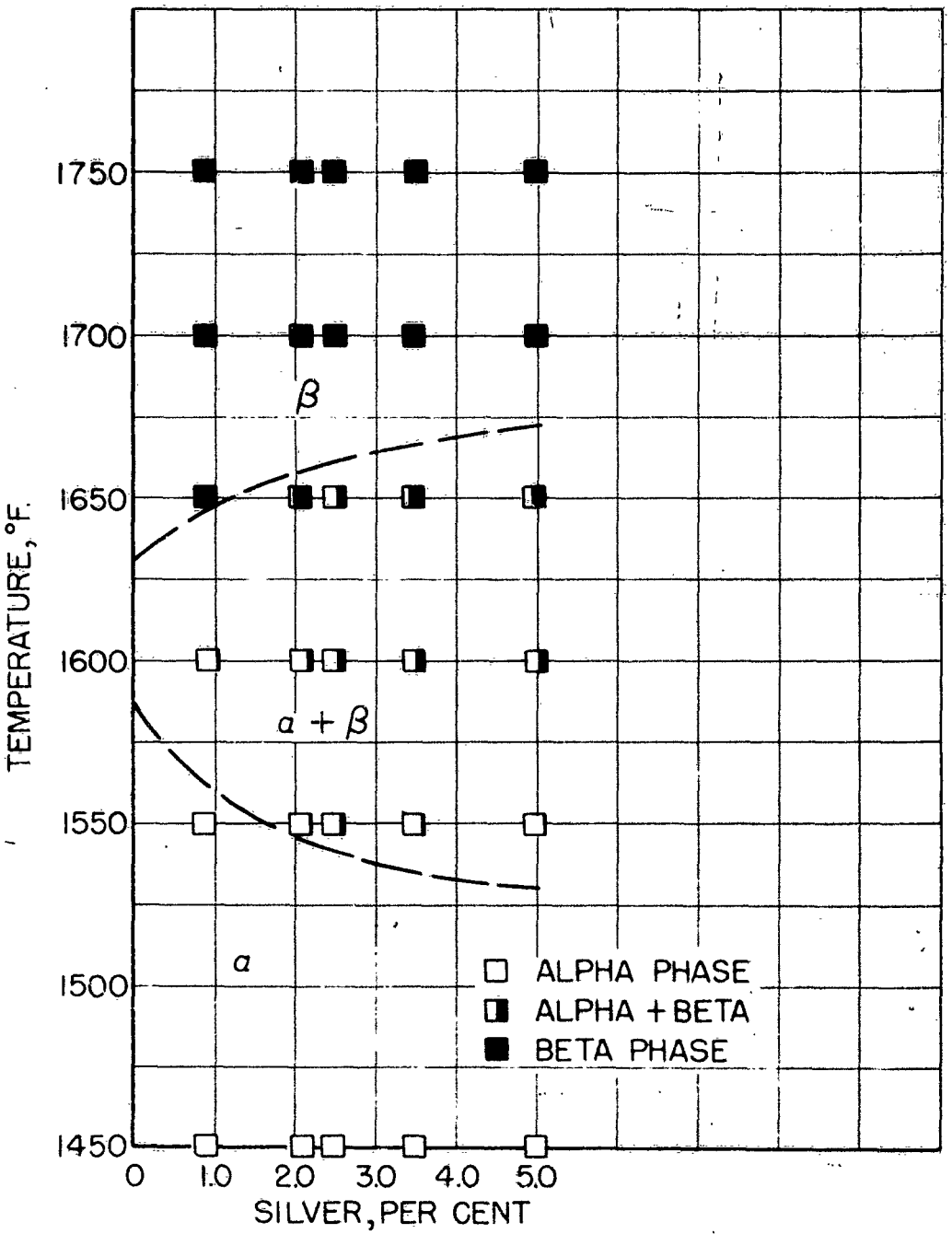

FIGURE 26. TENTATIVE DIAGRAM SHOWING TRANSFORMATION RANGE OF TITANIUM-0 TO 5 PER CENT SILVER ALLOYS MADE FROM PROCESS A METAL

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.

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 as-hot-rolled temper did not respond to an aging treatment of 4 hours at 750°F.

Structure and Transformation Range. Specimens of the titanium-beryllium 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.

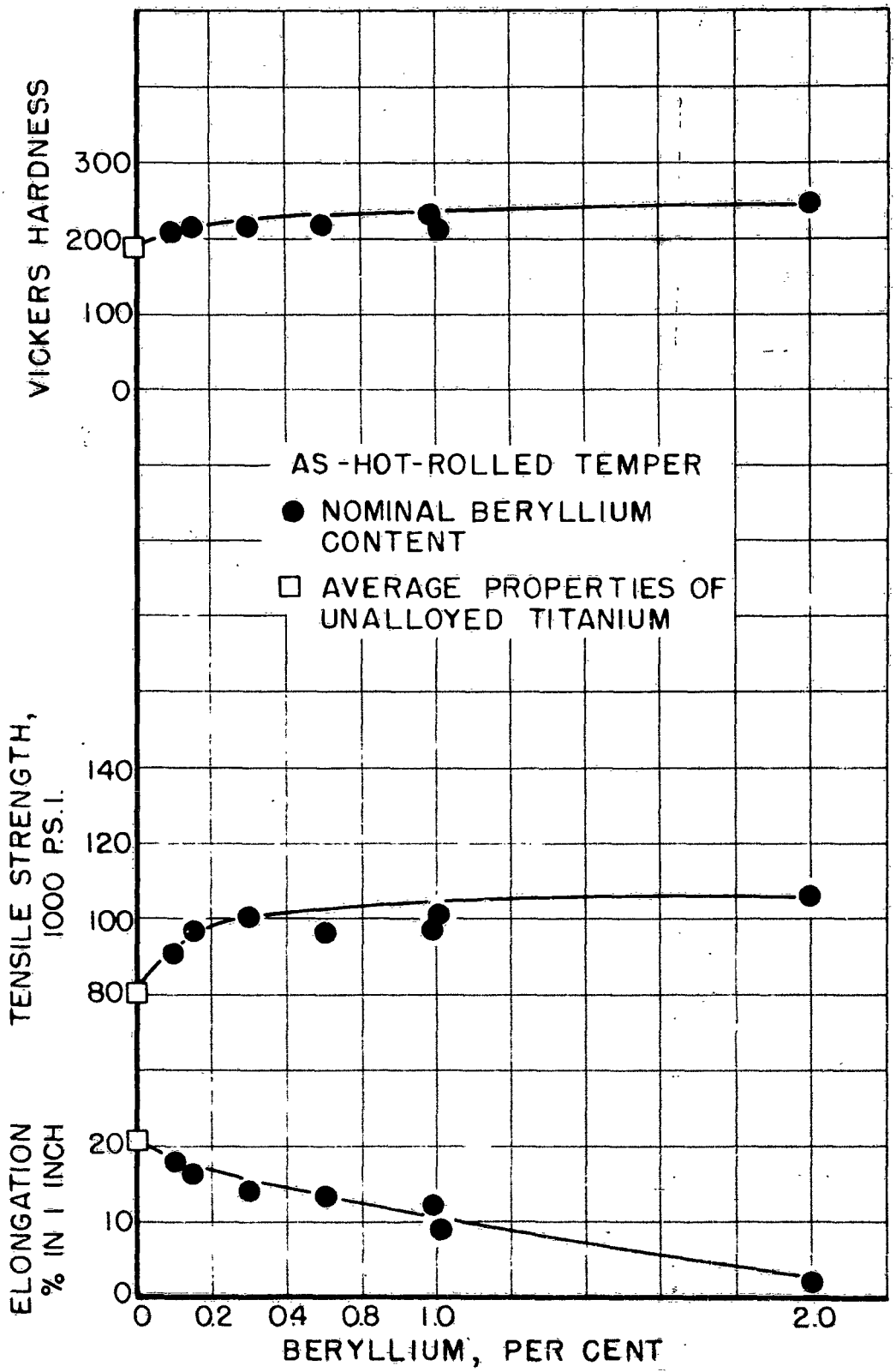

FIGURE 27. EFFECT OF BERYLLIUM ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET.

Heat Treatment. The Vickers 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.

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.

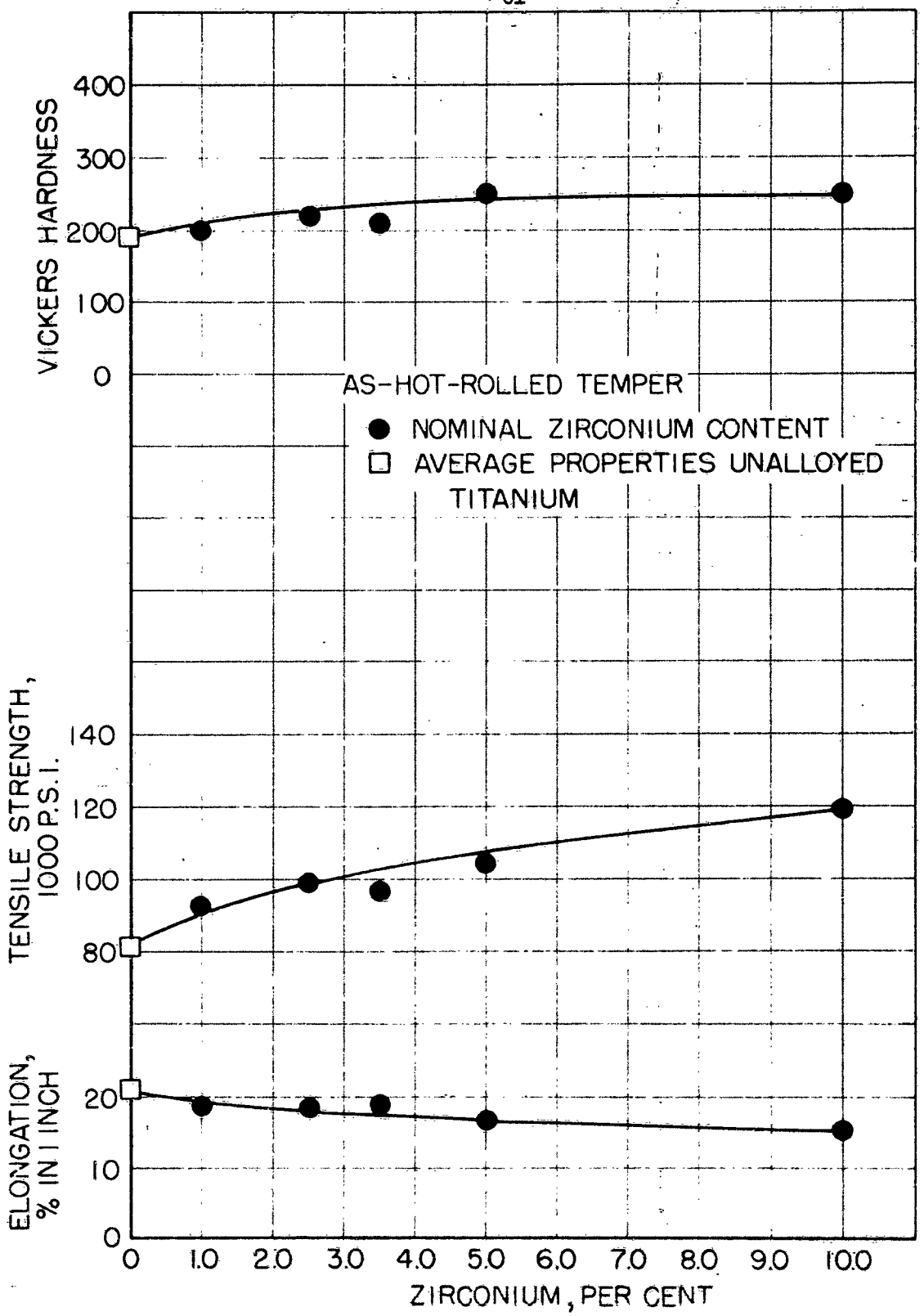

FIGURE 28. EFFECT OF ZIRCONIUM ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET

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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.

Structure and Transformation Range. 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 zirconium-rich 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.

TABLE 21. PHASES PRESENT IN BINARY TITANIUM-ZIRCONIUM ALLOYS AT TEMPERATURES INDICATED - PROCESS A METAL BASE

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.	As Hot Rolled Aged 4 Hrs.-750°F.	Heat Treated 1450°F.	Heat Treated 1550°F.	Heat Treated 1600°F.	Heat Treated 1650°F.	Heat Treated 1700°F.	Heat Treated 1750°F.
WH248	Unalloyed	α	α	α + Tr. β	90 - 10 α + β	80 - 20 α + β	70 - 30 α + β	β	β
WH253	1.0 Zr	α	α	α	α	80 - 20 α + β	20 - 80 α + β	β	β
WH252	2.5 Zr	α	α	α	α + Tr. β	80 - 20 α + β	20 - 80 α + β	β	β
WH251	3.5 Zr	α	α	α + Tr. β	90 - 10 α + β	50 - 50 α + β	30 - 70 α + β	β	β
WH250	5.0 Zr	α + β	α + β	α + β	α + β	50 - 50 α + β	β	β	β
WH249	10.0 Zr	α + β	α + β	70 - 30 α + β	70 - 30 α + β	β	β	β	β

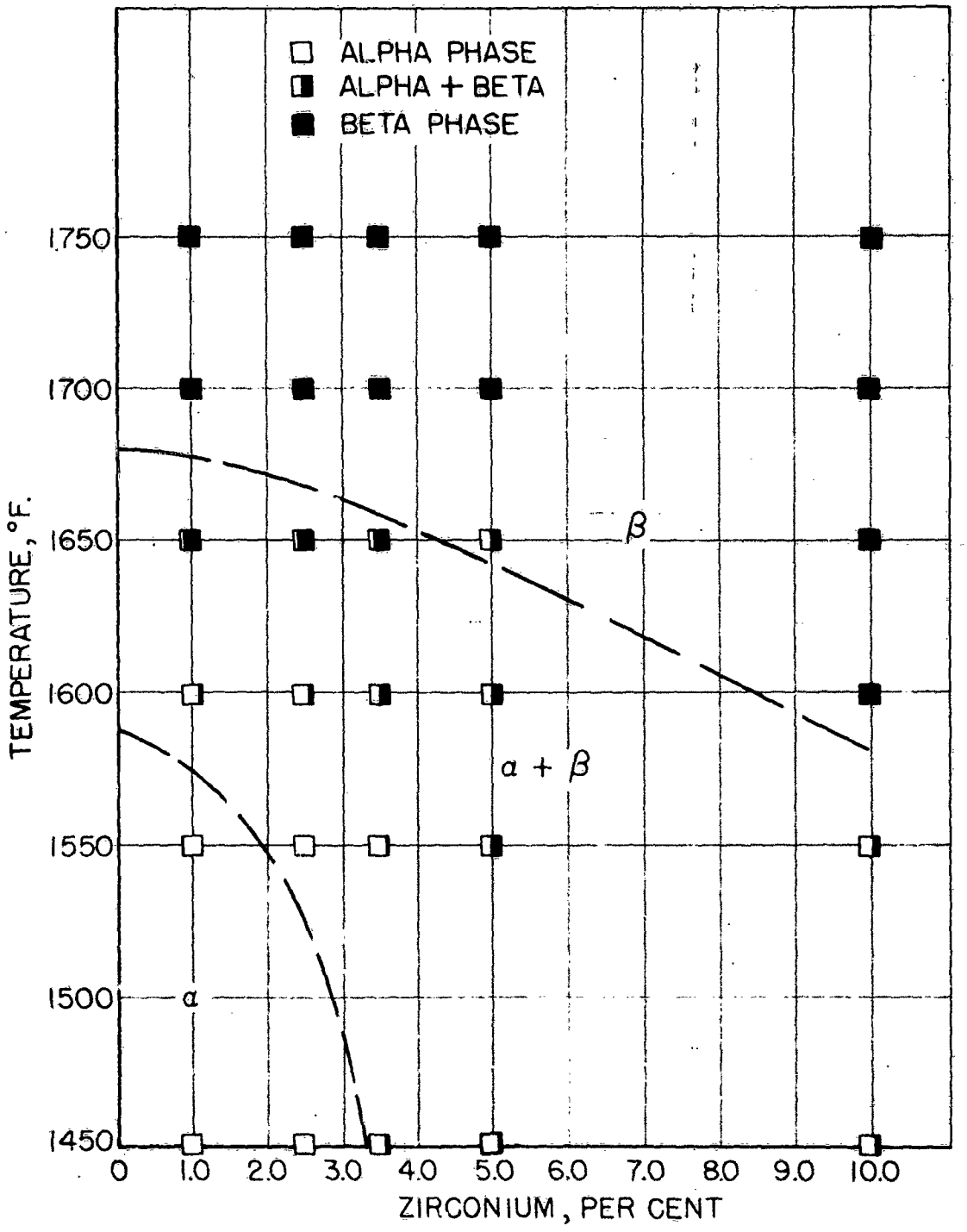

FIGURE 29. TENTATIVE DIAGRAM SHOWING TRANSFORMATION OF TITANIUM-0 TO 10 PER CENT ZIRCONIUM ALLOYS MADE FROM PROCESS A METAL

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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.

Mechanical Properties. The tensile properties, hardnesses, and minimum bend radii of the two titanium-columbium 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 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

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 4 hours at 750°F. Figure 31

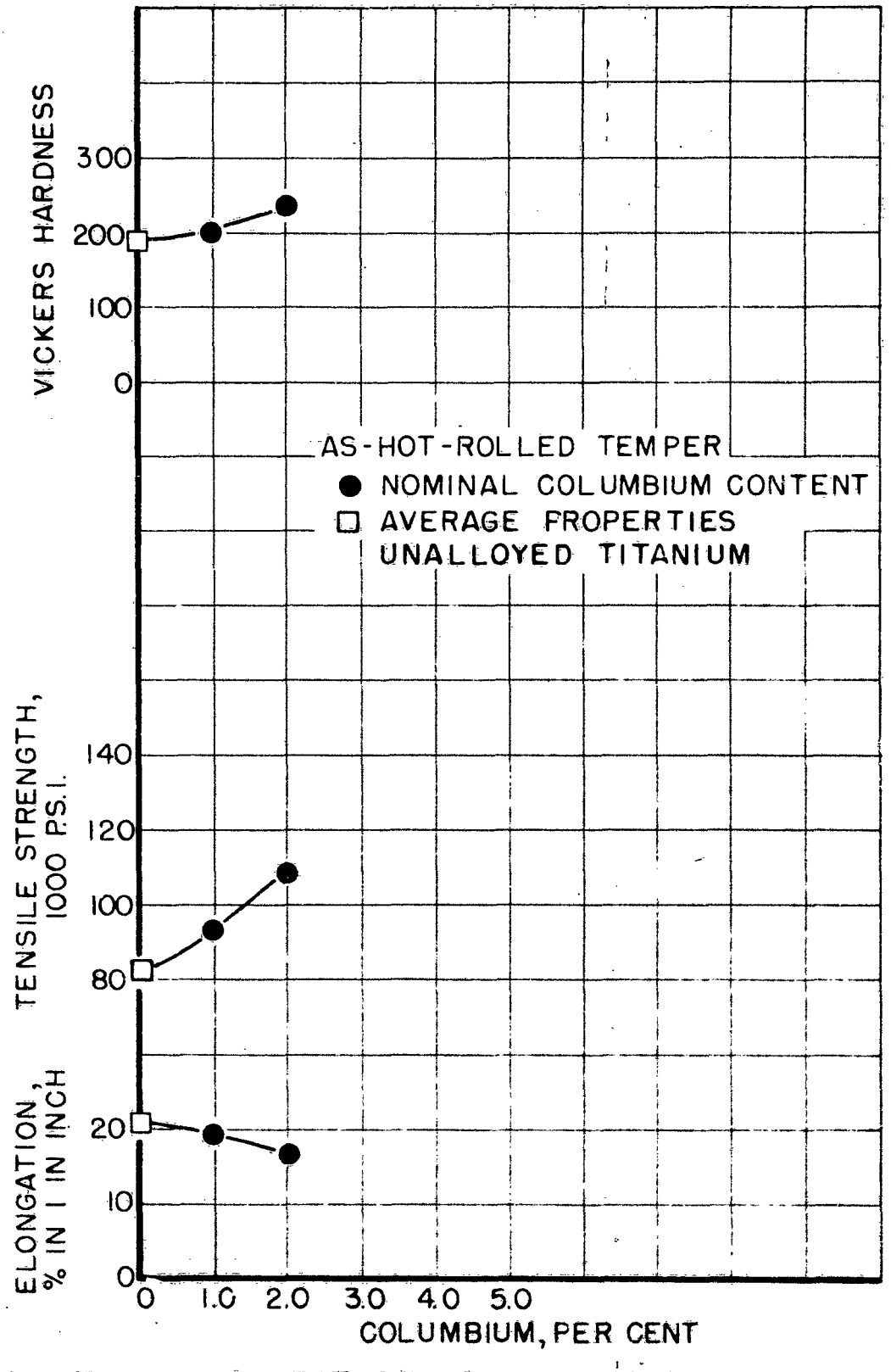

FIGURE 30. EFFECT OF COLUMBIUM ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET

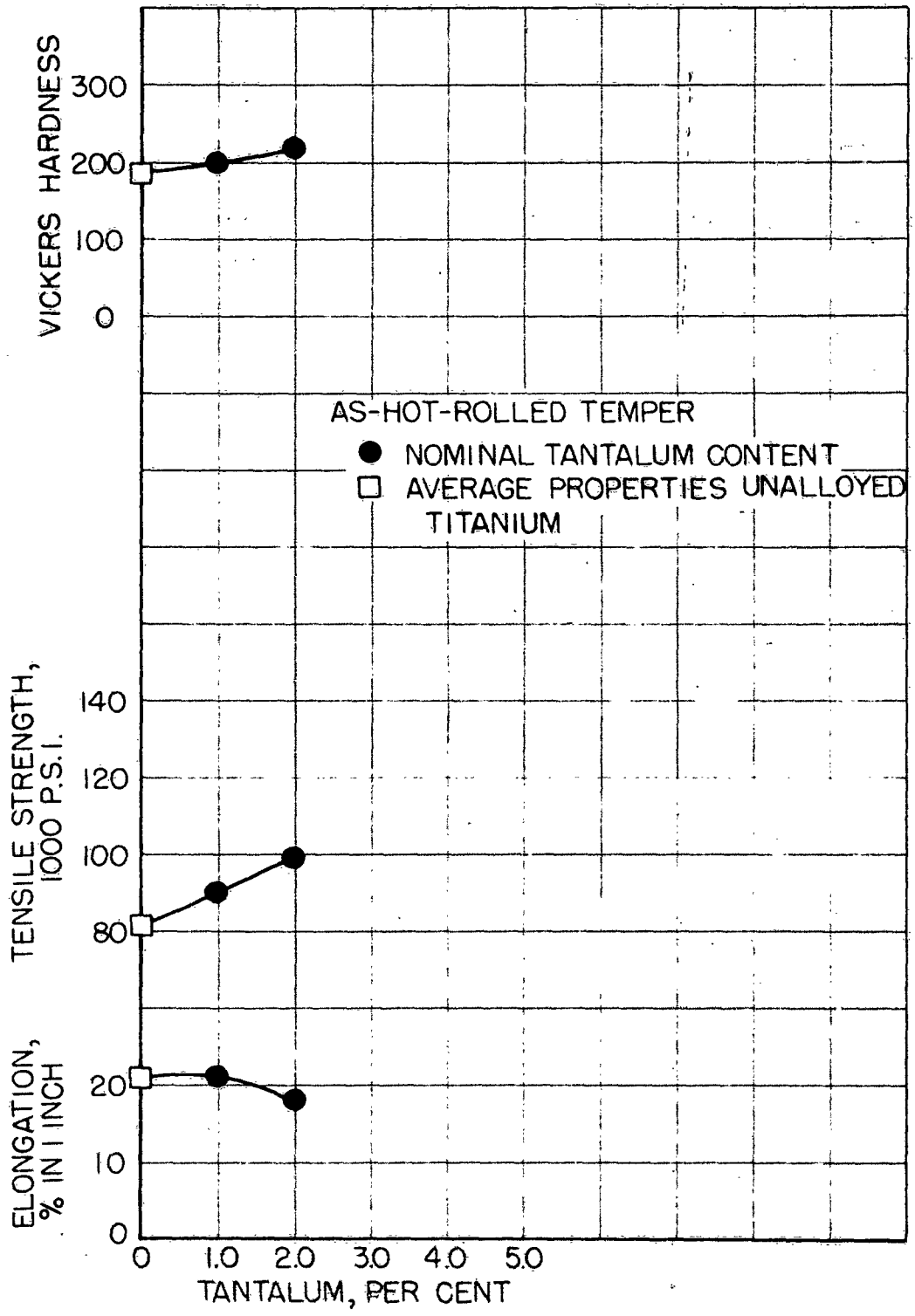

FIGURE 31. EFFECT OF TANTALUM ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM SHEET

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 elongation 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.
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% W.

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 elongation 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.

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:

Heat No.	Composition, %
VH312	5 Mn
VH310	5Mn, 0.25C
VH313	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

(As-Hot-Rolled Temper)

Alloys Prepared Using Metal Additions During Arc Melting					Alloys Prepared Using Alloy Hardeners in Arc Melting				
Heat No.	Intended or Actual Composition, %	Tensile Strength, p.s.i.	Elong., % in 1 Inch	VHN	Heat No.	Intended Composition, %	Tensile Strength, p.s.i.	Elong., % in 1 Inch	VHN
VC41(1)	4.37Mn	157,900	7.5	303					
VH78(1)	5.0Mn	162,800	6.5	315	VH312	5Mn	169,100	5.8	366
VC58(2)	4.80Mn, 0.21C	176,500	5.5	342	VH310	5.0Mn, 0.25C	178,730	6.2	378
VH93(2)	5.0Mn, 0.25C	193,400	2.5	444					
VH162(3)	5.0Mn, 0.1N	173,500	8.5	401	VH313	5.0Mn, 0.1N	183,340	6.0	409

(1) Data from Summary Report, page 182.

(2) Data from Summary Report, page 238a.

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

TABLE 22. TEST OF UNIFORMITY OF THE TENSILE PROPERTIES AND HARDNESS OF TITANIUM ALLOY SHEET. INGOTS MELTED USING A 35 PER CENT MANGANESE ALLOY HARDENER

Heat No.	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
"	X-2	"	Front, side	92,920	18.0	205
"	Y-1	"	Center	92,310	17.0	212
"	Y-2	"	Center	91,600	19.0	210
"	Z-1	"	Rear, side	90,000	18.0	209
"	Z-2	"	Rear, side	88,000	19.5	207
"		"	Avg.	90,360	18.2	210
"H312	X-1	5Mn	Front, side	166,620	6.5	348
"	X-2	"	Front, side	167,220	8.5	343
"	Y-1	"	Center	168,790*	4.5*	368
"	Y-2	"	Center	167,720	4.0	372
"	Z-1	"	Rear, side	174,830	4.0	378
"	Z-2	"	Rear, side	175,860*	4.5*	386
"		"	Avg.	169,100	5.8	366
"H310	X-1	5Mn, 0.25C	Front, side	185,430	4.5	377
"	X-2	" "	Front, side	162,160	6.5	358
"	Y-1	" "	Center	197,120	5.0	378
"	Y-2	" "	Center	181,090	8.0	381
"	Z-1	" "	Rear, side	190,070*	2.5*	398
"	Z-2	" "	Rear, side	167,830	7.0	375
"		" "	Avg.	178,730	6.2	378
"H313	X-1	5.0Mn, 0.1N	Front, side	201,010*	4.0*	425
"	X-2	" "	Front, side	192,310*	4.0*	407
"	Y-1	" "	Center	194,900*	4.0*	420
"	Y-2	" "	Center	189,870	7.0	401
"	Z-1	" "	Rear, side	181,610	7.0	418
"	Z-2	" "	Rear, side	178,530	4.0	386
"		" "	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.

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 Work 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.

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,

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

Heat No.	Spec. No.	Composition, % Intended	Composition, % Actual	Original Properties: Tensile Strength, p.s.i.(8)	Elong., % in 1 Inch	VHN (9)	Minimum Bend Radius, Inch(10)	Redetermined Properties: Tensile Strength, p.s.i.(8)	Yield Strength, p.s.i. at (0.2% Offset)	Elong., % in 1 Inch	Minimum Bend Radius, Inch(10)
WG27(1)	1	3.5Cr,0.25C,2.0Fe	- - 3.93Cr,1.42Fe	191,300	6.0		3/16	176,750	148,100	5.0	3/16
	2			190,000	6.0			169,300	153,500#	6.0	>1/4*
	Avg.			190,700	6.0	373		173,000	150,800	5.5	
WG31(1)	1	5.0Cr,0.25C,2.0Fe	- - 4.66Cr,2.15Fe	183,500	6.0		>1/4**	184,600	169,300	6.0	>1/4**
	2			212,500	2.0			186,700	167,700	4.0	>1/4**
	Avg.			198,000	4.0	401		190,650	168,500	5.0	
WG40(1)	1	5.0Cr,0.25C,2.0Mn	- - 4.63Cr,2.02Mn	196,300	3.0		3/16	188,700	177,400#	3.0	>1/4*
	2			192,500	1.0			183,000	173,200#	3.0	>1/4*
	Avg.			194,400	2.0	340		185,850	175,300	3.0	
WG119(2)	1	3.5Cr,1.0Mo,0.2N	0.044C,0.178N,3.81Cr,0.91Mo	190,000	7.0		3/16	***	***	***	>1/4*
	2			190,000	7.0			193,100	175,350	6.0	1/4
	Avg.			190,000	7.0	379		193,100	175,350	6.0	
WG48(3)	1	5.0Cr,0.25C,0.1N	0.168C,0.086N,4.31Cr	181,300	7.0		3/16	185,800	171,300	7.0	3/16
	2			185,000	5.0			181,700	155,550#	4.0	>1/4*
	Avg.			183,200	6.0	339		183,750	163,400	5.5	
WH93(4)	1	5.0Mn,0.25C	0.212C,0.021N,4.57Mn	195,300	2.0		3/16	183,350	168,150	4.0	>1/4*
	2			193,300	3.0			180,000	164,300	6.0	>1/4*
	Avg.			194,300	2.5	444		181,700	166,200	5.0	
WG172(5)	1	5.0Cr,0.1N	0.022C,0.106N,5.77Cr	189,400	5.0		3/16	170,400	158,600	7.0	3/16
	2			183,800	5.0			166,450	152,250	5.0	3/16
	Avg.			186,600	5.0	295		168,400	155,400	6.0	
WG169(5)	1	5.0Cr,0.2N	0.020C,0.161N,5.87Cr	200,000	3.0		3/16	181,950	173,400	6.0	>1/4*
	2			170,000	5.0			186,700	186,100	3.0	>1/4*
				185,000	4.0	365		184,300	179,750	4.5	
WH131(6)	1	5.0Mn,1.0W	0.036C,0.026N,4.21Mn,0.92W	181,300	4.0		3/16	181,000	161,300	7.0	3/16
	2			200,000	5.0			****	****	****	>1/4*
	Avg.			190,700	4.5	337		181,000	161,300	7.0	
WH29(1)	1	5.0Cr,2.0V,0.25C	0.174C,0.021N,6.12Cr,1.27V	190,700	4.0		3/16	188,900	171,100	3.0	>1/4**
	2			188,600	3.0			189,400	168,950	2.0	3/16
	Avg.			189,700	3.5	378		189,150	170,000	2.5	
WH55(2)	1	5.0Cr,1.0V,0.2N	0.028C,0.163N,5.62Cr,0.69V	214,300	3.0		>1/4*	200,700	-	3.0	>1/4**
	2			201,300	4.0			201,700	182,750#	5.0	>1/4**
	Avg.			207,800	3.5	390		201,200	182,750#	4.0	

TABLE 23. (CONTINUED)

Heat No.	Spec. No.	Composition, % Intended	Composition, % Actual	Original Properties: Tensile Strength, p.s.i.(8)	Original Properties: Elong., % in 1 Inch	Original Properties: VHN (9)	Original Properties: Minimum Bend Radius, Inch(10)	Redetermined Properties: Tensile Strength, p.s.i.(8)	Redetermined Properties: Yield Strength, p.s.i. at (0.2% Offset)	Redetermined Properties: Elong., % in 1 Inch	Redetermined Properties: Minimum Bend Radius, Inch(10)
WG113(2)	1	2.5Cr,1.0Mo,0.2N	0.026C,0.181N,2.89Cr,0.92Mo	191,000	6.0		3/16	172,600	163,200	8.0	3/16
	2			185,900	6.0			175,150	150,950#	8.0	>1/4*
	Avg.			188,500	6.0	378		173,900	157,100	8.0	
WH27(2)	1	5.0Cr,2.0Ni,0.2N	0.024C,0.145N,4.63Cr,1.98Ni	189,300	4.0		3/16	198,900	176,450#	3.0	>1/4*
	2			200,000	2.0			200,000	191,050	6.0	>1/4*
	Avg.			194,700	3.0	417		199,450	183,750	4.5	
WH283(7)	1	3.5Mn,1.0V	0.032C,0.038N,2.43Mn,0.78V	***	***		1/4	138,350	104,000	12.0	3/14
	2			183,300	8.0			165,500	-	6.0	>1/4*
	Avg.			183,300	8.0	245		151,900	-	9.0	
WH293(7)	1	5.0Mn,1.0V	0.044C,0.030N,4.46Mn,0.90V	195,300	3.0		>1/4*	150,350	130,450	7.0	>1/4*
	2			201,600***	3.0***			154,400	119,750	7.0	>1/4*
	Avg.			198,400	3.0	302		152,400	125,000	7.0	
WH292(7)	1	3.5Mn,2.0V	0.032C,0.032N,2.27Mn,1.20V	193,750	2.0		1/4	141,100	111,850	9.0	>1/4*
	2			****	****			169,950	-	5.0	3/16
	Avg.			193,750	2.0	235		155,500	-	7.0	

* Some ductility.
** Very little ductility.
*** Unmelted alloy evident in fracture.
**** Defective specimen.
\# Extrapolated value; curve did not reach 0.2% offset.
(1) Data taken from Summary Report, Table 59.
(2) " " " " " Table 67.
(3) " " " " " Table 56.
(4) " " " " " Table 33.
(5) " " " " " Table 41.
(6) " " " " " Table 51.
(7) Data not previously reported. From ternary titanium-manganese-vanadium series of alloys showing nonuniform distribution of the alloying elements.
(8) Longitudinal 14-gauge specimens 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section.
(9) 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and the rolling direction.
(10) 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 14-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 those of the earlier tests were obtained from a considerable number of these compositions.

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

TABLE 24. EVALUATION OF SELECTED TITANIUM-BASE ALLOYS

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
WH40	X-1	Unalloyed	Front, side	75,700	22.0	178				
"	X-2	"	Front, side	75,200	22.0	178				
"	Y-1	"	Center	75,500	23.0	179				
"	Y-2	"	Center	76,000	21.0	178				
"	Z-1	"	Rear, side	75,000	22.0	176				
"	Z-2	"	Rear, side	74,000	22.0	172				
			Avg.	75,200	22.0	176				
	X-1	3.5Cr,0.25C,2.0Fe	Front, side							
	X-2	" " "	Front, side							
	Y-1	" " "	Center							
	Y-2	" " "	Center							
	Z-1	" " "	Rear, side							
	Z-2	" " "	Rear, side	---						
WH49	X-1	5.0Cr,0.25C,2.0Fe	Front, side	224,200	1.0	429	WH314	194,600	3.0	384
"	X-2	" " "	Front, side	202,900	0.0	435	"	195,900*	2.0*	416
"	Y-1	" " "	Center	204,000	1.0	433	"	184,200	6.0	405
"	Y-2	" " "	Center	Broke in grips		425	"	207,900	3.0	408
"	Z-1	" " "	Rear, side	214,300	1.0	411	"	180,400	7.0	370
"	Z-2	" " "	Rear, side	212,400*	1.0*	421	"	181,100*	5.0*	374
"			Avg.	211,600	1.0	426	"	190,700	5.0	393

TABLE 24. (CONTINUED)

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
	X-1	5.0Cr, 0.25C, 2.0Mn	Front, side				VH299	190,800	2.0	347
	X-2	" " "	Front, side				"	167,300	8.0	336
	Y-1	" " "	Center				"	168,600	2.0	387
	Y-2	" " "	Center				"	200,300	3.0	383
	Z-1	" " "	Rear, side				"	183,000	3.0	406
	Z-2	" " "	Rear, side				"	156,300	9.0	325
			Avg.				"	177,700	4.5	364
WJ43	X-1	3.5Cr, 1.0Mo, 0.2N	Front, side	-	-	-				
"	X-2	" " "	Front, side	-	-	-				
"	Y-1	" " "	Center	188,600	5.0	393				
"	Y-2	" " "	Center	173,500*	4.0*	417				
"	Z-1	" " "	Rear, side	177,500	6.0	399				
"	Z-2	" " "	Rear, side	170,900	6.0	412				
			Avg.	177,600	5.2	405				
	X-1	5.0Cr, 0.25C, 0.1N	Front, side				VH300	167,100	9.0	403
	X-2	" " "	Front, side				"	168,000	8.0	376
	Y-1	" " "	Center				"	180,100*	5.0*	374
	Y-2	" " "	Center				"	200,000	3.0	397
	Z-1	" " "	Rear, side				"	196,500	3.0	380
	Z-2	" " "	Rear, side				"	200,900	-	366
			Avg.				"	185,400	5.6	382

TABLE 24. (CONTINUED)

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
"J46	X-1	5.0Mn,0.25C	Front, side	177,900	4.0	397				
"	X-2	" "	Front, side	189,800	4.0	373				
"	Y-1	" "	Center	175,900	5.0	373				
"	Y-2	" "	Center	178,800	6.0	370				
"	Z-1	" "	Rear, side	185,900	6.0	392				
"	Z-2	" "	Rear, side	173,000	6.0	390				
			Avg.	180,200	5.0	382				
	X-1	5.0Cr,0.1N	Front, side				"H302	180,700*	3.0*	396
	X-2	" "	Front, side				"	182,200	4.0	387
	Y-1	" "	Center				"	189,900	5.0	390
	Y-2	" "	Center				"	188,000	5.0	390
	Z-1	" "	Rear, side				"	183,000	6.0	368
	Z-2	" "	Rear, side				"	183,800	7.0	360
			Avg.					184,600	5.0	382
"J41	X-1	5.0Cr,0.2N	Front, side	206,500	1.0	386				
"	X-2	" "	Front, side	193,500	2.0	391				
"	Y-1	" "	Center	202,100	3.0	403				
"	Y-2	" "	Center	188,600	3.0	420				
"	Z-1	" "	Rear, side	202,200	4.0	412				
"	Z-2	" "	Rear, side	183,100	4.0	404				
			Avg.	196,000	2.8	403				

TABLE 24. (CONTINUED)

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
" J50	X-1	5.0Mn, 1.0"	Front, side	187,600	4.0	372				
"	X-2	" "	Front, side	187,500	4.0	380				
"	Y-1	" "	Center	187,600	4.0	381				
"	Y-2	" "	Center	177,800	5.0	370				
"	Z-1	" "	Rear, side	175,000*	5.0*	376				
"	Z-2	" "	Rear, side	173,200*	5.0*	375				
			Avg.	181,500	4.5	375				
" J38	X-1	5.0Cr, 2.0V, 0.25C	Front, side	185,600	4.0	379				
"	X-2	" " "	Front, side	184,800	4.0	361				
"	Y-1	" " "	Center	192,900	4.0	370				
"	Y-2	" " "	Center	194,300	3.0	386				
"	Z-1	" " "	Rear, side	195,600	4.0	370				
"	Z-2	" " "	Rear, side	196,400	4.0	413				
"			Avg.	191,600	3.8	379				
" J42	X-1	5.0Cr, 1.0V, 0.2N	Front, side	-	-	-				
"	X-2	" " "	Front, side	-	-	-				
"	Y-1	" " "	Center	177,400	6.0	359				
"	Y-2	" " "	Center	178,300	6.0	383				
"	Z-1	" " "	Rear, side	180,300	5.0	379				
"	Z-2	" " "	Rear, side	180,300	5.0	401				
"			Avg.	179,100	5.5	380				

TABLE 24. (CONTINUED)

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
J44	X-1	2.5Cr,1.0Mo,0.2N	Front, side	178,100*	4.0*	369				
"	X-2	" " "	Front, side	182,000	5.0	347				
"	Y-1	" " "	Center	173,700	6.0	364				
"	Y-2	" " "	Center	179,600	4.0	361				
"	Z-1	" " "	Rear, side	186,300	4.0	421				
"	Z-2	" " "	Rear, side	184,700	4.0	427				
			Avg.	180,700	4.5	381				
	X-1	5.0Cr,2.0Ni,0.2N	Front, side							
	X-2	" " "	Front, side							
	Y-1	" " "	Center							
	Y-2	" " "	Center							
	Z-1	" " "	Rear, side							
	Z-2	" " "	Rear, side							
J39	X-1	3.5Mn,1.0V	Front, side	153,500	8.0	434				
"	X-2	" "	Front, side	153,500	8.0	417				
"	Y-1	" "	Center	156,000	7.0	429				
"	Y-2	" "	Center	157,600	7.0	358				
"	Z-1	" "	Rear, side	166,100*	6.0*	399				
"	Z-2	" "	Rear, side	165,000	7.0	383				
			Avg.	158,600	7.0	403				
	X-1	5.0Mn,1.0V	Front, side							
	X-2	" "	Front, side							
	Y-1	" "	Center							
	Y-2	" "	Center							
	Z-1	" "	Rear, side							
	Z-2	" "	Rear, side							

TABLE 24. (CONTINUED)

Alloys Prepared Using Alloy Hardeners(1)							Alloys Prepared Using Pure Metals			
Heat No.	Spec. No.	Intended Composition, %	Relative Location of Specimen	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)	Heat No.	Tensile Strength, p.s.i.(2)	Elong., % in 1 Inch	VHN (3)
"J47	X-1	3.5Mn, 2.0V	Front, side	164,100	6.0	370				
"	X-2	" "	Front, side	175,500	6.0	373				
"	Y-1	" "	Center	184,800	7.0	363				
"	Y-2	" "	Center	171,100	7.0	369				
"	Z-1	" "	Rear, side	171,900	7.0	366				
"	Z-2	" "	Rear, side	171,900	7.0	387				
			Avg.	173,200	6.6	371				
"J45	X-1	5.0Mn, 0.1N	Front, side	181,800*	6.0*	379				
	X-2	" "	Front, side	187,000	6.0	363				
	Y-1	" "	Center	192,700	6.0	384				
	Y-2	" "	Center	195,800*	5.0*	388				
	Z-1	" "	Rear, side	190,000	5.0	364				
	Z-2	" "	Rear, side	185,800	6.0	366				
			Avg.	188,900	5.6	374				

* Very slight evidence of nonuniform structure in fracture.

(1) Chromium added as a 58%Cr eutectic alloy, manganese as a 35%Mn master alloy, vanadium as a 35%V master alloy, iron as a 35%Fe master alloy, nickel as a 34%Ni 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 powders.

(2) 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.

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 boride-lined carbon crucibles was completed. Additional melts were made in hot-pressed titanium carbide, hot-pressed zirconium 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.

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

TABLE 25. DATA ON REFRACTORIES TESTED AND RESULTS OBTAINED

Crucible Material	Melting Atmosphere	Wetting 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	-	267(2)	Ditto	Ditto
B lining on graphite crucible	Argon	Yes	-	306(2)	See Footnote 5	Ditto
Hot-pressed ZrO_2 (stabilized with CaO)	Argon	Only in one spot	-	440(3)	Not complete	Ditto
CaO-compressed; porosity - 1.6%	Argon	Very little wetting	-	Not complete		Some attack
CaO (fluxed with 2% TiO_2) compressed; porosity - 0.8%	Argon	Wet 1/2 diameter of crucible	-	Not complete		Some attack
Al_2O_3 compressed; porosity - 1.6%	Argon	Wet only one side of crucible	-	Not complete		Some attack

(1) Indicated by meniscus on top of ingot.
(2) Vickers hardness of the titanium ingot from which the forged-rod melting stock was made was 210. 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.
(3) Vickers hardness of the titanium ingot from which the forged-rod melting stock was made was 160.
(4) The carbide phase appears in a dendritic pattern uniformly distributed throughout the sample.
(5) 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.

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.

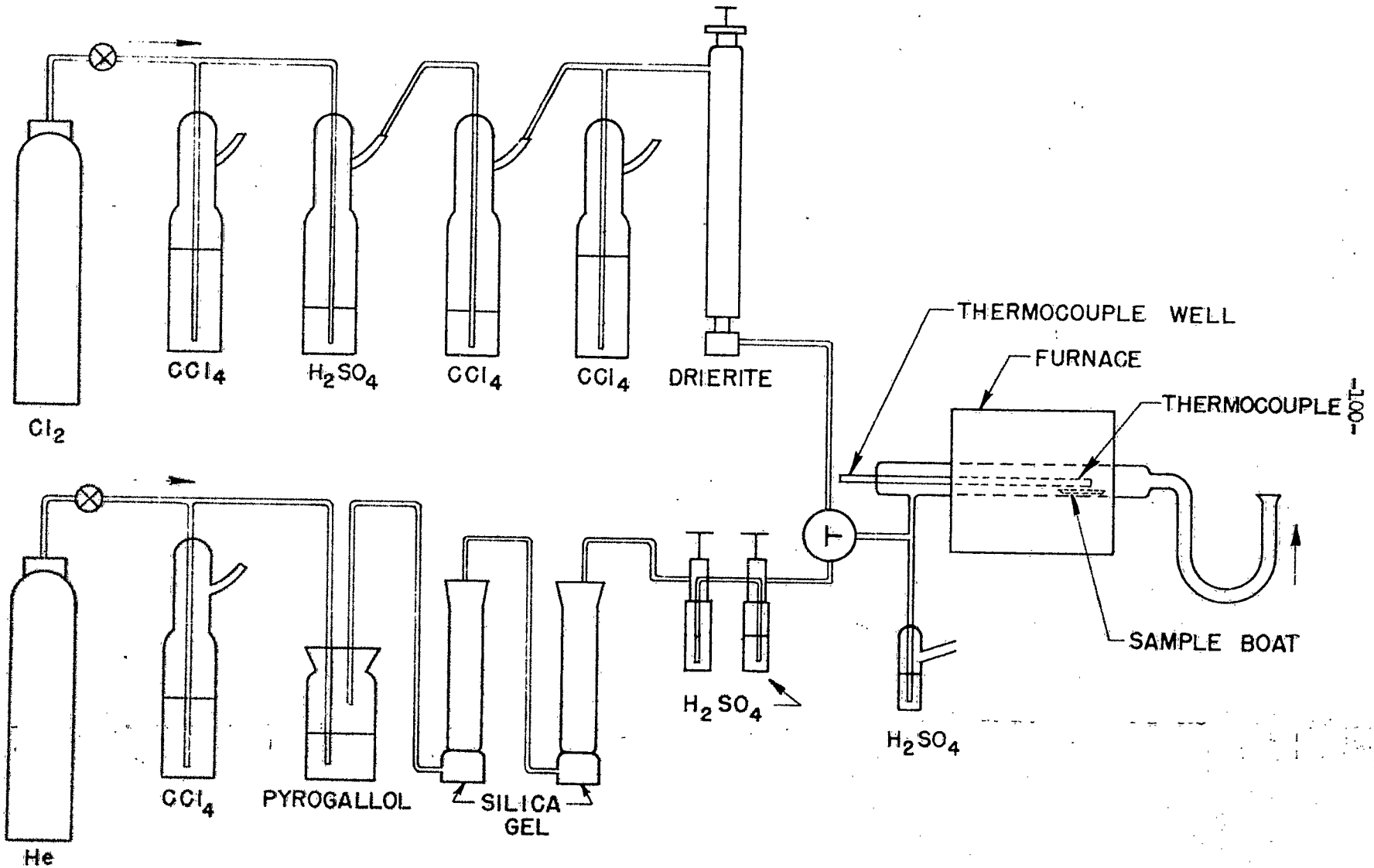

FIGURE 32. SCHEMATIC DRAWING SHOWING CHLORINATION TRAIN

0-13483

TABLE 26. TYPICAL ANALYSIS OF BUREAU OF MINES TITANIUM POWDER

Element	Per Cent
C	0.02
N	0.02
Fe	0.14
Mg	0.46
Cl	0.04
Si	0.02
H_2	0.12
Al	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.

TABLE 27. SUMMARY OF RUNS

Run No.	Average Temperature, °C.	Time, Hours	Sample	% Residue	Boat Consumption, Mg	
1	450	5	Bureau of Mines powder	1.2		
2	450	2	Ditto	0.8		
3	450	4	"	8.		(2)
4	525	5	"			(2)
5	525	6-1/2	"	3.73 (1)		(2)
6	525	6-1/2	"	0.9 (1)		(3)
7	550	1-1/2	"	0.8	0.0	
8	550	2-1/2	"			(2)
9	550	7-1/2	Du Pont sponge	0.32		
10	575	1	Bureau of Mines powder			(2)
11	650	7	Ditto	0.54	15	
12	650	6	"	0.75	3	(2)
13	675	5	"	0.72	60	
14	675	4-1/2		0.5	126.3	
15	850	3-1/2	Residue Run No. 6	2.7	26.2	(3)
16	850	5	Residue Runs Nos. 9, 11, 13	3.3	26.4	(3)
17	875	6	Residue Run No. 12	4	2.4	

(1) No CCl_4 used.
(2) Boat cracked during run.
(3) Leco HF-C boat used.

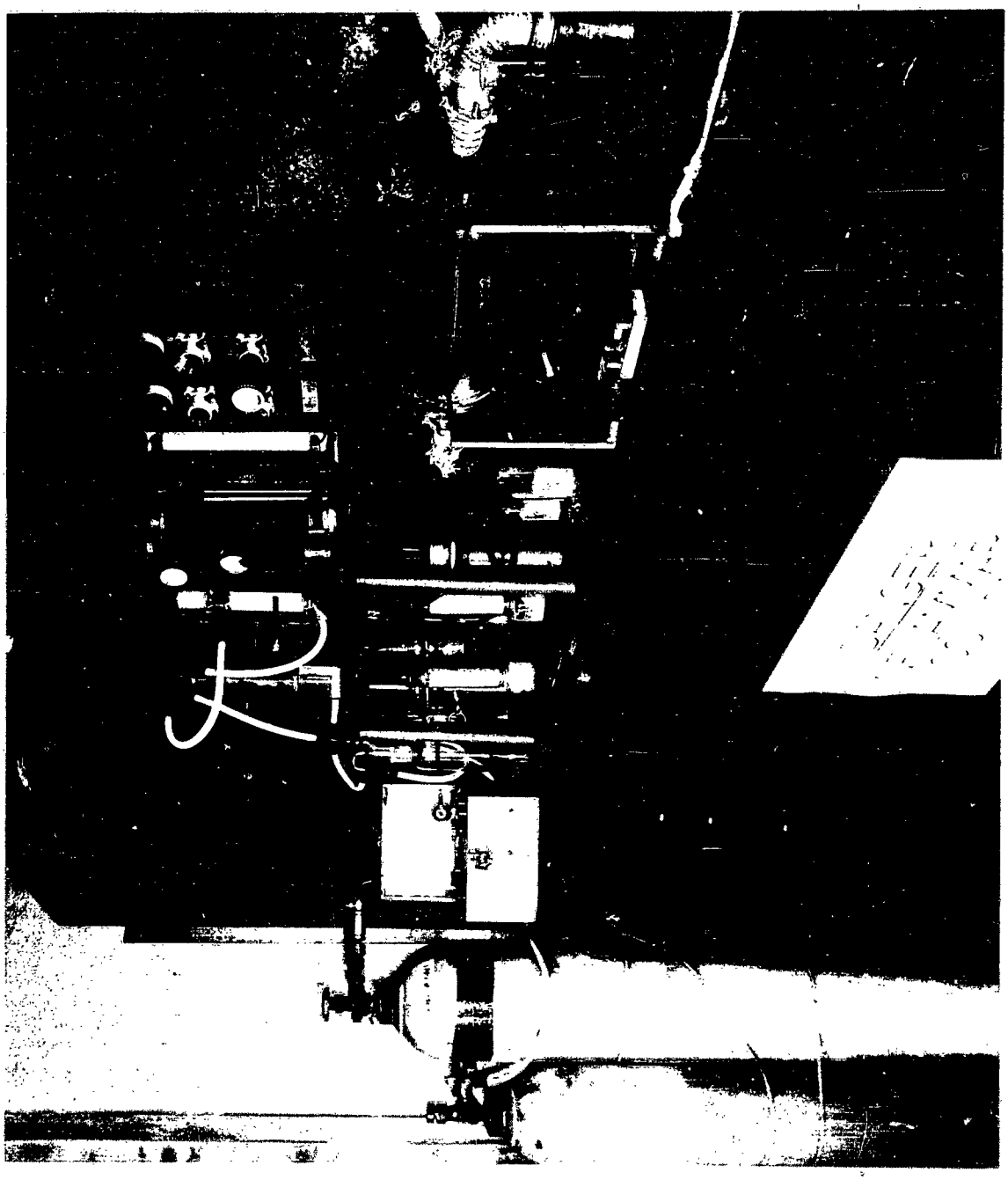

Figure 3. Photograph of the apparatus.

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 decompose. Richardson 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 chlorine alone, chemical analyses account for a large percentage of the oxygen expected to be found in the samples. However, uncertainty exists as to loss of oxygen and the oxidation state of titanium associated with oxygen. Therefore, without additional information, it is impossible to establish a definite factor for calculating oxygen content from the chemical analyses.

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* The following spinels could be present: $MgAl_2O_4$; $FeTiO_4$; $MgTiO_4$.

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 laboratory.

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.

Preparation of the Standard Samples

Samples with known weights of oxygen added as TiO_2 were prepared from iodide titanium crystal bar as follows:

(1) V. A. Aluise, et al., Anal. Chem. 24, 477-81, (1952).
(2) S. S. Zimmerstein and F. T. Eppig, Anal. Chem. 21, 463-7 (1949).
(3) H. W. Richardson, et al., Trans. Brit. Ceram. Soc., 51, 149-70 (1952).

A small capsule of iodide titanium was prepared by drilling a hole in the iodide titanium, adding a weighed amount of dried C.P. TiO_2 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 TiO_2 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.

TABLE 28. OXYGEN STANDARDS FOR ANALYSIS

Sample No.	Total Wt. of Sample Before Melting, Grams	Total Wt. of Sample After Double Melting, Grams	Loss or Gain in Weight During Melting, Grams	TiO_2 Added, Grams	Per Cent Oxygen in Sample*	VHN(1) Top	VHN(1) Bottom	Designation Assigned to Sample Sent to Dr. Derge
1	7.9246	7.9231	-0.0015	None	None	97.8	97.9	2
2	7.7944	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
4	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	1
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	0.0488	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.

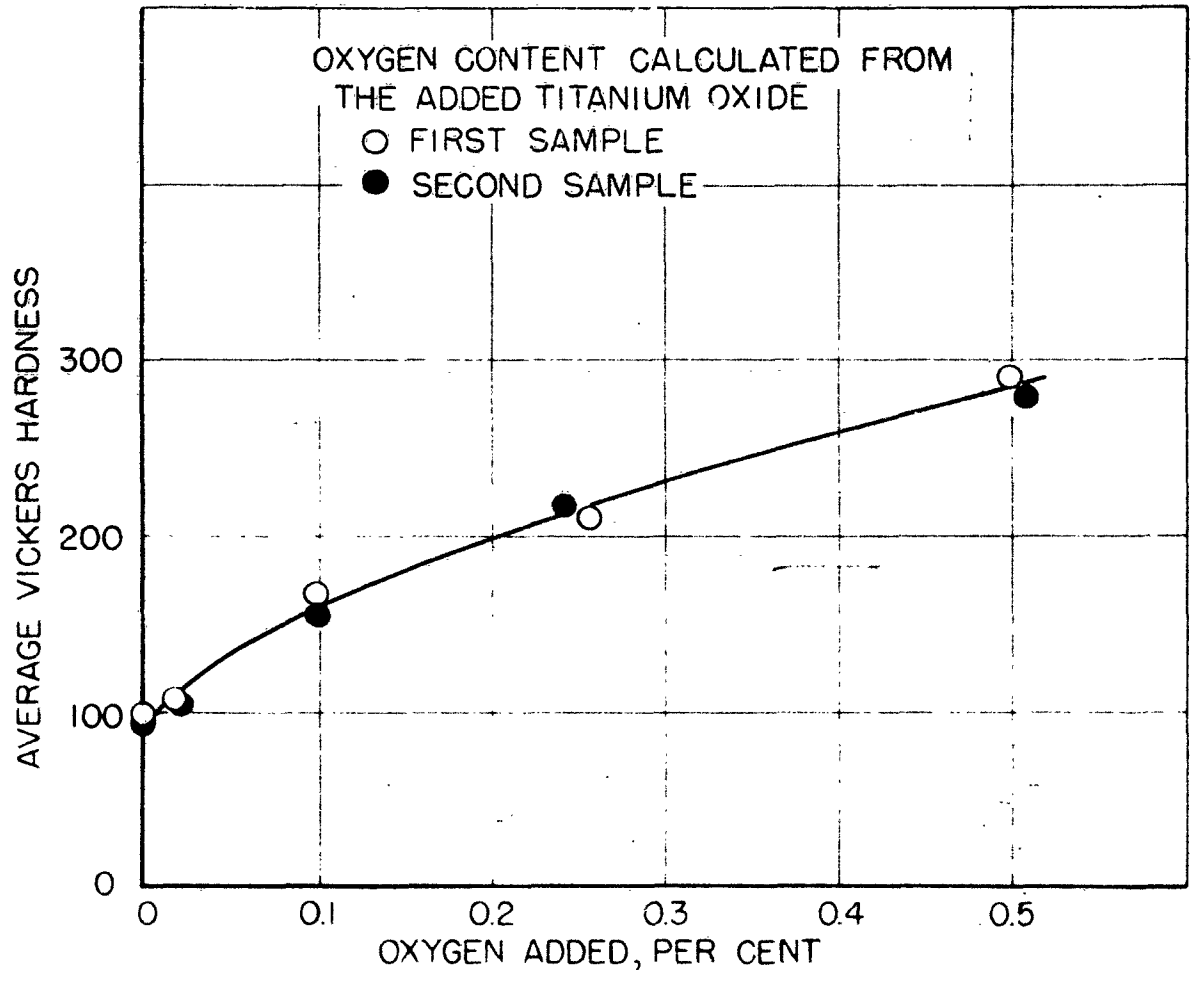

FIGURE 34. RELATION BETWEEN THE VICKERS HARDNESS AND THE OXYGEN CONTENT OF IODINE TITANIUM SAMPLES

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

Apparatus and General Technique. The modification of the vacuum-fusion 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.

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 copper oxide tube, Q, and the freezing trap, M. The pressure in the collection system is read on the dibutyl phthalate manometer, C.

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

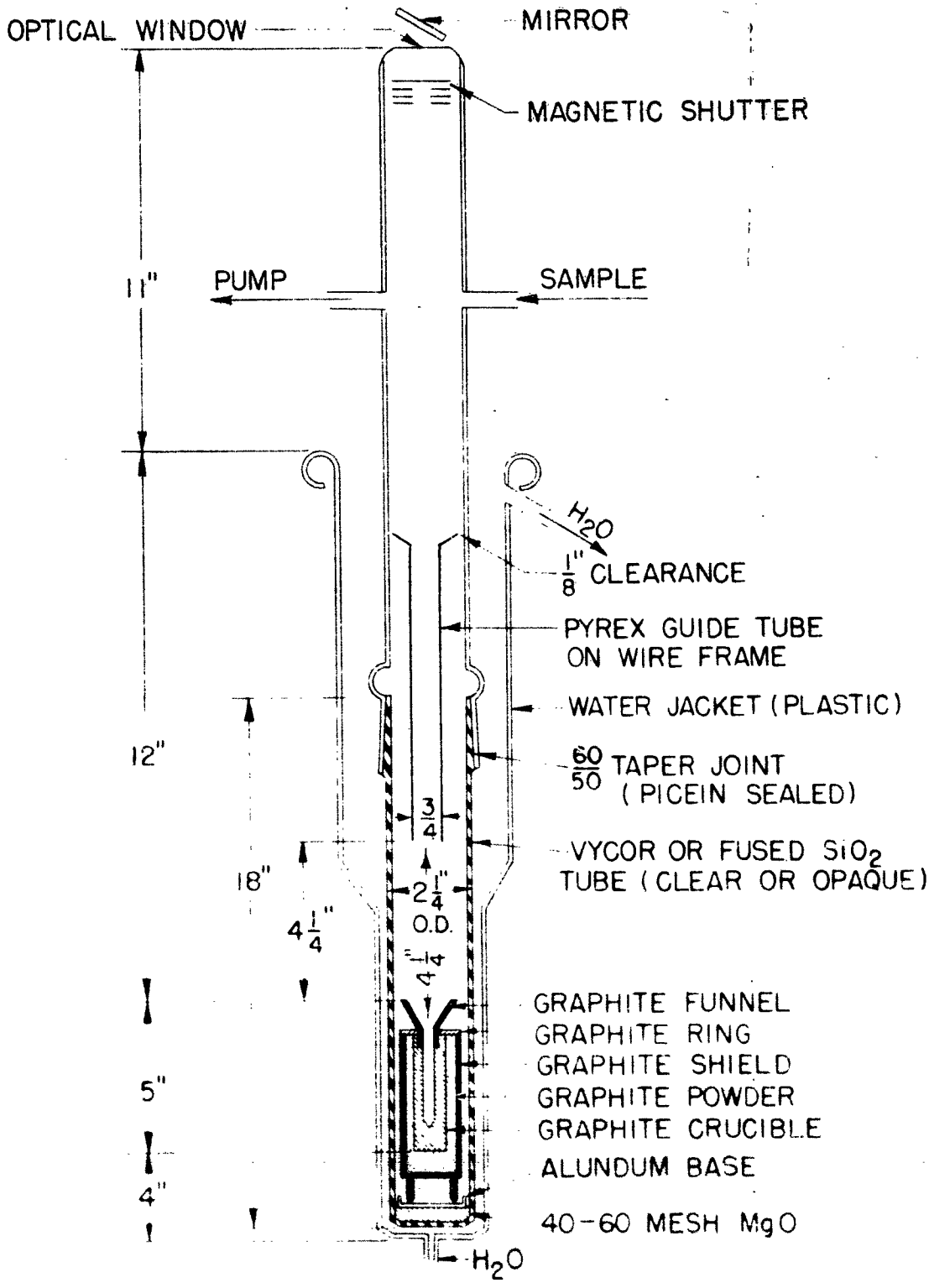

FIGURE 36 FURNACE ASSEMBLY FOR DERGE'S APPARATUS

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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; or the large volume, to stopcock, 7; depending upon the amount of gas extracted. After measurement, the gas is oxidized (CO to CO_2 and H_2 to H_2O) in the copper oxide tube, and the H_2O is frozen out in trap, M, by means of a dry ice–acetone mixture. After 10 minutes, the residual gases (CO_2 and N_2) are collected, in 5 minutes, in a suitable volume and measured. Liquid nitrogen is then put on the trap, M, and the CO_2 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 4 to 8 hours. When 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 1400°C. and degassed, and a third blank is taken. The blanks at 1400°C. and 1700°C. are usually similar in volume.

A. MERCURY LIFT
B. SAMPLE STORAGE ARM
C. MAGNETIC SHUTTER
D. OPTICAL WINDOW
E. MIRROR
F. GUIDE FUNNEL
G. INDUCTION FURNACE
H. MERCURY DIFFUSION PUMP (SINGLE STAGE)
I. MERCURY DIFFUSION PUMP (TWO STAGE)
J. STORAGE BULB
K. MERCURY DIFFUSION PUMP (SINGLE STAGE)
L. MERCURY DIFFUSION PUMP (SINGLE STAGE)
M. FREEZING TRAP (DRY ICE-ACETONE OR LIQUID NITROGEN.)
N. MEDIUM VOLUME
O. DIBUTYL PHTHALATE MANOMETER
P. LARGE VOLUME
Q. CuO TUBE
R. HEATER

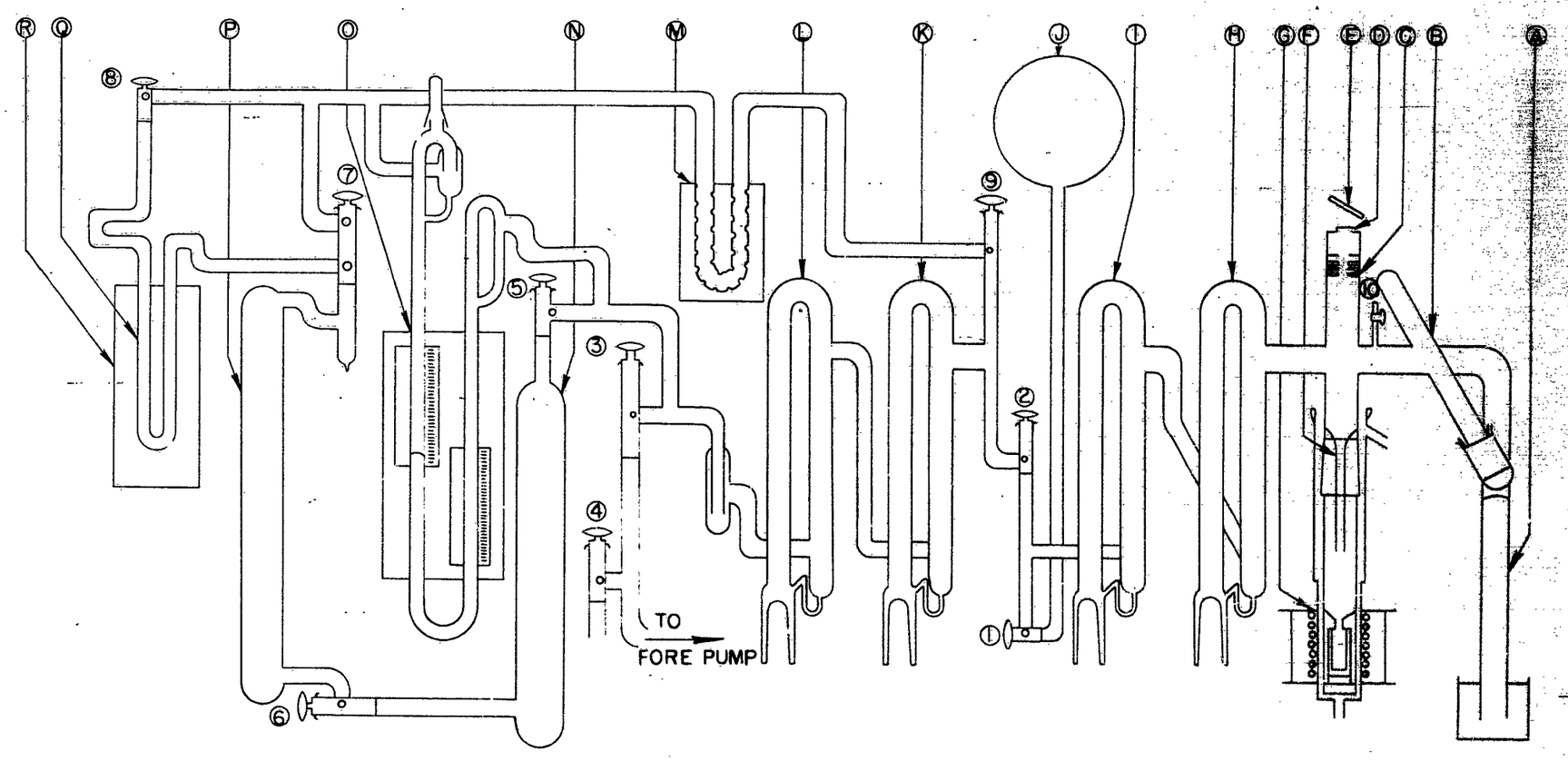

FIGURE 35. DERGE VACUUM - FUSION APPARATUS.

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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.

The copper oxide is made by oxidizing 1/4" by 1/4" squares of copper 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:

"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 added 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.

While 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

TABLE 29. RESULTS OF VACUUM-FUSION ANALYSIS

Battelle Sample No.	Per Cent Oxygen Added	Sample No. Assigned to the Specimen Before Sending to Dr. Derge	Run No.	Vacuum-Fusion Analysis, % H	O	N	Average VHN of Sample
			1	0.0121	0.0135	0.0007	
1	None	2	2	0.0125	0.0142	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	1	0.0088	0.083	0.0063	106
			1	0.0106	0.042*	0.0033*	
5	0.100	1	2	0.0102	0.094	0.0041	166
			3	0.0149	0.056**	0.045**	
			3 recalc.	-	0.080	0.0037	
			Avg.	0.0126	0.087	0.0039	
			1	0.0136	0.124	0.0005	
6	0.100	4	2	0.0128	0.146	0.0008	155
			Avg.	0.0132	0.135	0.0006	
			1	0.0097	0.094	0.0023	
7	0.257	7	2	0.0108	0.136	0.015	210
			Avg.	0.0103	0.125	0.0087	
			1	0.0106	0.304	0.014	
8	0.243	10	2	0.0234	0.307	0.017	217
			Avg.	0.0170	0.306	0.016	
			1	0.0146	0.562	0.0016	
9	0.500	6	2	0.0127	0.513***	0.0015	289
			Avg.	0.0137	0.538	0.0016	
			1	0.0098	0.547	0.0007	
10	0.510	8	2	0.0089	0.485***	0.0008	280
			3	0.0159	0.518	0.0008	
			Avg.	0.0115	0.517	0.0008	

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

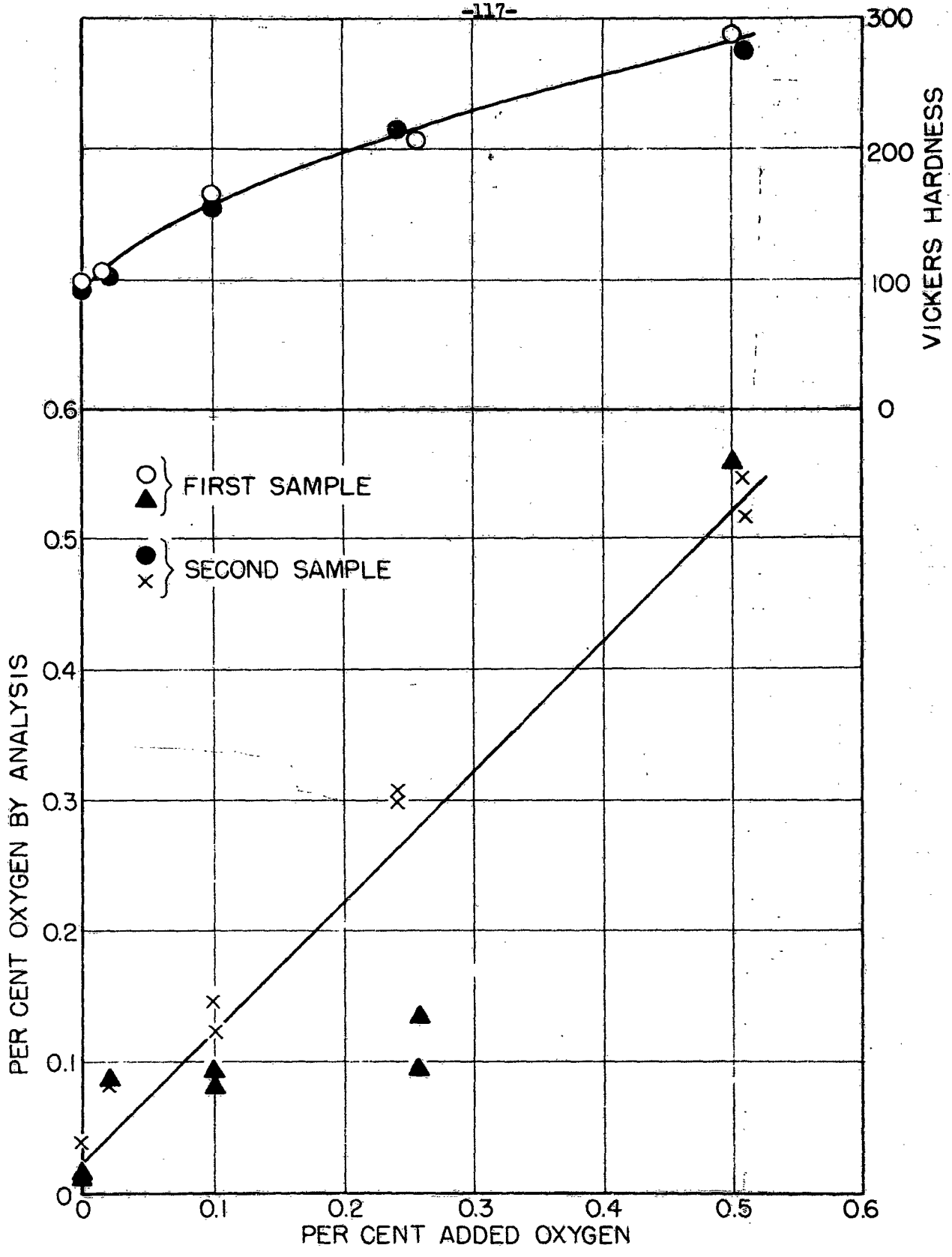

FIGURE 37. RELATIONSHIP OF THE PER CENT ADDED OXYGEN TO THE PER CENT OXYGEN BY ANALYSIS AND THE HARDNESS OF THE SAMPLES

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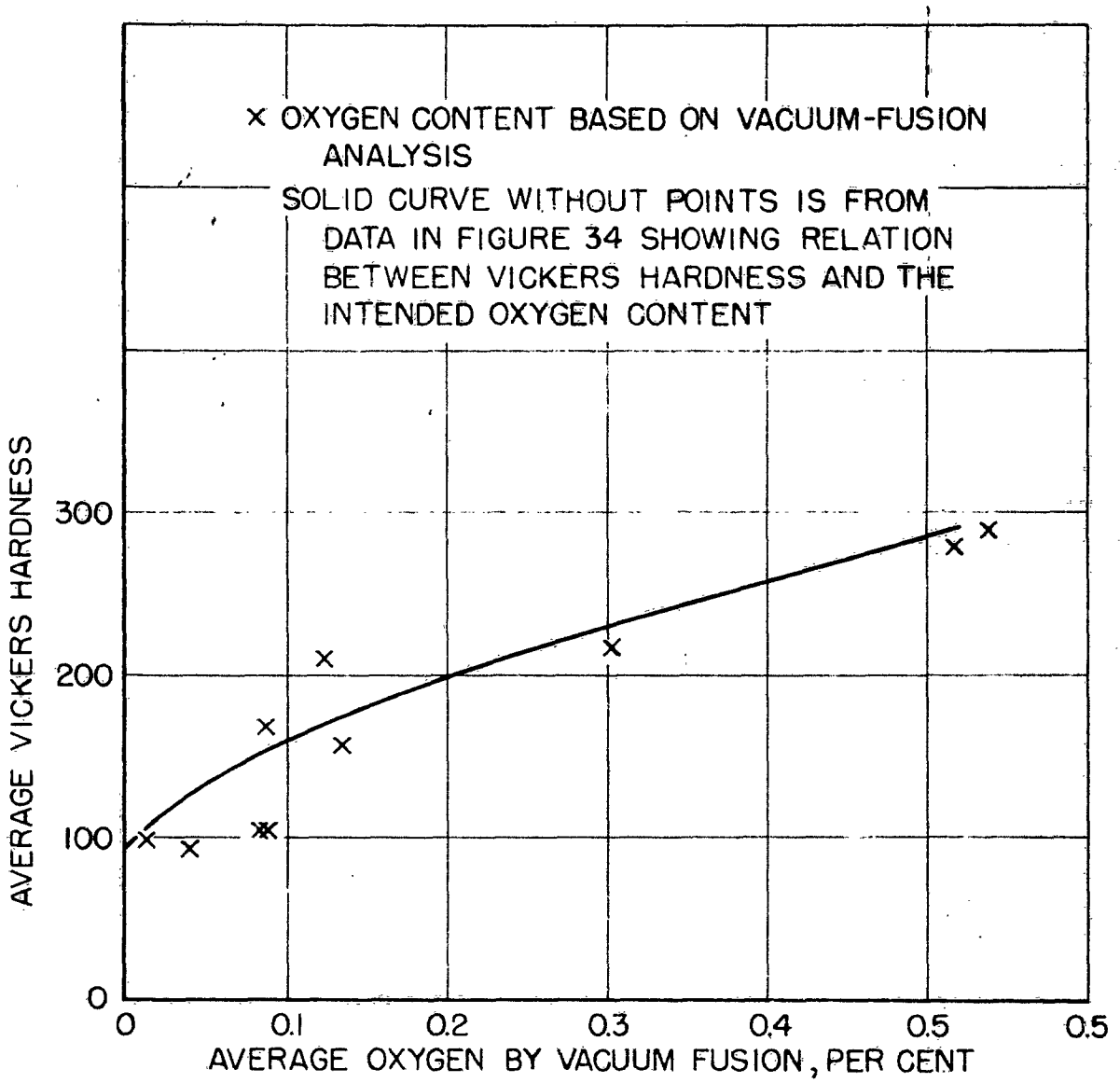

FIGURE 38. RELATION BETWEEN THE VICKERS HARDNESS OF THE SAMPLES AND THE OXYGEN CONTENT

O-13680

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 Ta_2C, 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.

Quotations are being obtained from the Norton Company on hot-pressed 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, ThO_2-ZrO_2, TiO_2-ZrO_2, ThO_2-CaO, and ZrO_2-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.

mr
November 7, 1949

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ABSTRACT:

Progress is reported in development of titanium alloys. Phase relations in titanium - 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 solvus 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 ductility of Process A titanium. Ternary alloys of managese and carbon, manganese and vanadium, and molybdenum and tungsten, prepared by adding the pure metals during arc melting, had quite erratic tensile properties when tested after fabrication to sheet. Tests were completed on evaluation of "hot-pressed" titanium carbide and graphite crucibles.

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