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First Progress Report Covering the Period May 18 to September 18, 1949 on Research and Development on Titanium Alloys (None)

Simmons, W. O.; Greenidge, C. T.; Craighead, C. M.; and others
Battelle Memorial Institute, Columbus, Ohio
(Same) for AMC, Wright-Patterson Air Force Base, Dayton, Ohio

1st

Aug'49 Unclass. U.S. English 59 photos, tables, diagr, graphs (Same)

Progress is reported in the development of titanium alloys. During the period reported, titanium binary alloys of germanium and nickel were studied, as well as titanium-molybdenum and titanium-manganese ternary alloys. Carbon, copper, chromium, manganese, iron, and cobalt were added to the titanium-molybdenum binary alloys, and nitrogen, copper, molybdenum and cobalt were added to titanium-manganese alloys. The alloys were tested in both the as-hot-rolled temper and after aging the hot-rolled sheet four hours at 750°F. Tensile strengths and elongations for various alloys are listed. Some of the alloys were tested after solution heat treatment at 1600°F. Tests were also completed on "hot pressed" crucibles of tungsten carbide, titanium carbide, and zirconium carbide as refractories for melting titanium. Melts were made in graphic crucibles lines with tantalum carbide and tungsten boride.

Copies obtainable from CADO.

Titanium alloys

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

USAF Contr. No. AF33(038)-3736

FIRST PROGRESS REPORT

August 31, 1949

to

WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO

on

RESEARCH AND DEVELOPMENT ON
TITANIUM ALLOYS

G-1261-13

BATTELLE
MEMORIAL INSTITUTE
505 King Avenue
COLUMBUS 1, OHIO

FORM 49

FIRST PROGRESS REPORT

COVERING THE PERIOD MAY 18 TO SEPTEMBER 18, 1949

on

RESEARCH AND DEVELOPMENT ON TITANIUM ALLOYS

Contract No. 33(038)-3736

to

WRIGHT-PATTERSON AIR FORCE BASE
DAYTON, OHIO

BATTELLE MEMORIAL INSTITUTE

August 31, 1949

BATTELLE MEMORIAL INSTITUTE
INDUSTRIAL AND SCIENTIFIC RESEARCH
COLUMBUS 1, OHIO

September 16, 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 twenty-five (25) copies and one (1) reproducible of the first progress report prepared under Contract No. AF 33(038)-3736.

Part III of the Summary Report, dated July 30, 1949, under Contract W-33-038 ac-21229, describes the development of analytical methods and the study of refractories carried out during the period May 18, 1948, to May 18, 1949. In addition, it contains data obtained during the interval May 18, 1949, to July 30, 1949, on alloys which were in process at the expiration of the preceding contract, May 18, 1949. At the request of Mr. J. B. Johnson, this latter information obtained during the first two and a half months of the present contract was submitted in lieu of the first regular bimonthly progress report.

The attached report contains an account of the following:

1. A description of the alloy development work, including the data on alloys prepared during the period May 18, 1949, to September 18, 1949. Data obtained during this period on alloys already under study on May 18, 1949, are included in the above-mentioned Summary Report.

2. The progress made during the period May 18, 1949, to September 18, 1949, on the development of analytical methods for oxygen in titanium.

3. The results of the study during the period May 18, 1949, to September 18, 1949, on the development of refractories for holding molten titanium.

Very truly yours,

L. W. Eastwood

LWE:ec
Enc. (26)

FIRST PROGRESS REPORT

COVERING THE PERIOD MAY 18 TO SEPTEMBER 18, 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

August 31, 1949

SUMMARY

In the 4-month period covered by this report, May 18 to September 18, 1949, titanium binary alloys of germanium and nickel were studied. Also investigated were titanium-molybdenum and titanium-manganese ternary alloys. Additions of carbon, copper, chromium, manganese, iron, and cobalt were made to the titanium-molybdenum binary alloys and additions of nitrogen, copper, molybdenum, and cobalt were made to titanium-manganese alloys.

The alloys were tested in both the as-hot-rolled temper and after aging the hot-rolled sheet 4 hours at 750°F. The following alloys were found to have tensile strengths and elongations in the range of 156,000 to 180,000 p.s.i. and 5 to 11 per cent, respectively.

1. Titanium - 5 per cent molybdenum base alloys with additions of 1 per cent copper, 2 per cent copper, 1 per cent manganese, and 2 per cent iron.

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2. Titanium - 3.5 per cent manganese base alloys with additions of 0.1 per cent nitrogen, 1.0 per cent cobalt, and 2.0 per cent cobalt.

3. Titanium - 5 per cent manganese base alloys with additions of 0.1 per cent nitrogen, and 2.0 per cent cobalt.

Aging the hot-rolled sheet at 750°F. for 4 hours increased the tensile strength and hardness of some alloys without a serious reduction in elongation values, as shown in the following table.

Heat No.	Intended Composition, Per Cent	Condition	Tensile Strength, p.s.i.	Elongation, Per Cent in 1 In.	Vickers Hardness
WH38	5 Mo, 1 Cu	As hot rolled	171,700	5.5	298
		Aged 4 hrs. 750°F.	180,300	5.0	327
WH182	3.5 Mo, 1 Mn	As hot rolled	147,200	4.0	288
		Aged 4 hrs. 750°F.	170,600	6.0	302
WH227	3.5 Mo, 1 Fe	As hot rolled	167,500	6.0	285
		Aged 4 hrs. 750°F.	175,400	5.0	317
WH166	7.5 Mn, 0.1 N	As hot rolled	184,400	3.5	403
		Aged 4 hrs. 750°F.	190,000	5.0	498
WH159	2.5 Mn, 0.2 N	As hot rolled	134,400	5.5	272
		Aged 4 hrs. 750°F.	152,900	6.5	272
WH151	3.5 Mn, 1.0 Co	As hot rolled	166,700	6.0	297
		Aged 4 hrs. 750°F.	201,800	3.0	285
WH150	2.5 Mn, 2.0 Co	As hot rolled	143,600	10.0	262
		Aged 4 hrs. 750°F.	176,700	6.0	307

Some of the alloys were tested after solution heat treatment at 1600°F., but the properties in this condition were not so good as those for the as-hot-rolled temper. Further testing of solution-heat-treated alloys does not appear justified.

Tests were completed on "hot pressed" crucibles of tungsten carbide, titanium carbide, and zirconium carbide as refractories for melting titanium. Melts were made in graphite crucibles lined with tantalum carbide and tungsten boride, but the examination of these melts has not been completed.

The determination of oxygen in titanium by the $Cl_2 - CCl_4$ method, three modifications of the vacuum-fusion technique, and the vacuum-fusion mass-spectrograph method has been investigated. None of these techniques, to date, has resulted in consistent and reproducible oxygen values.

INTRODUCTION

During the period, May 18 to September 18, 1949, covered in this report, 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 one hundred and forty 0.5-pound ingots were made and submitted for fabrication during the period May 18 to August 31. 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.

In addition to the above alloys, two large ingots of Process A titanium were made at the request of Mr. J. B. Johnson. Heat Ti354, 11 pounds gross, and Heat Ti363, 8.5 pounds gross, were melted in the large arc-melting furnace. These ingots were scalped to 6 and 5.1 pounds, respectively, and shipped to Mr. E. A. Gee of E. I. du Pont de Nemours and Company. The analyses and hardnesses of the two ingots were as follows:

	Ingot Ti354				Ingot Ti363			
	C,%	N,%	W,%	Brinell	C,%	N,%	W,%	Brinell
Top	0.08	0.010	0.12	137	0.03	0.010	0.09	131
Middle	0.05	0.009	0.11	137	0.04	0.008	0.18	131
Bottom	0.04	0.013	0.12	137	0.06	0.007	0.19	131

EVALUATION OF EXPERIMENTAL TITANIUM-BASE ALLOYS

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

The alloy evaluation work covered in the present bimonthly report has been directed toward the investigation of:

1. Binary titanium-germanium alloys.
2. Binary titanium-nickel alloys.
3. Ternary titanium-molybdenum base alloys.
4. Ternary titanium-manganese base alloys.

These alloys have been tested in both the as-hot-rolled condition and after aging the alloys in the as-hot-rolled temper for 4 hours at 750°F.

The data for the titanium-germanium and titanium-nickel binary alloys are shown in Tables 1 and 2. Figures 1 and 2 graphically illustrate the effects, respectively, of germanium and nickel on the tensile strength, elongation, and Vickers hardness of as-hot-rolled Process A titanium sheet.

The addition of 0.1 to 1.0 per cent germanium to Process A metal produced essentially no effect on the tensile strength, elongation, or Vickers hardness. Likewise, the titanium-germanium alloys were not responsive to solution or aging treatments.

Nickel increased the tensile strength of as-hot-rolled Process A titanium sheet to 120,000 p.s.i. at 10 per cent nickel, while the elongation decreased to 2 per cent, and the Vickers hardness increased to 300. Aging the as-hot-rolled titanium-nickel alloys for 4 hours at 750°F. produced no beneficial effect on the tensile properties. Solution heat treatment of the titanium-nickel alloys at temperatures ranging from 1450 to 1750°F. resulted in an increase in the Vickers hardness. The higher nickel alloys showed the greatest increase in hardness. The bend ductility of the binary titanium-nickel alloys either in the as-hot-rolled or heat-treated tempers was poor.

The present data do not justify further investigation of binary titanium-germanium or titanium-nickel alloys.

The effect of additions of carbon, copper, chromium, manganese, iron, cobalt, and nickel on the properties of titanium-molybdenum base alloys are shown in Tables 3 and 4. Figures 3 to 9 graphically illustrate the changes in tensile strength, elongation, and hardness resulting

TABLE 1. PROPERTIES OF BINARY ALLOYS OF TITANIUM WITH GERMANIUM OR NICKEL PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
					Titanium - Germanium								
WH180	Unalloyed	83,000	20.5	193	3/32	84,700	19.0	188	3/16	92,900	14.5	181	3/16
WH170	0.10 Ge	87,100	16.5	190	3/64	85,700	18.0	190	1/16	77,500	16.5	194	3/32
WH169	0.50 Ge	77,400	16.5	206	3/16	88,400	18.5	202	1/16	80,400	17.5	203	3/16
WH168	1.0 Ge	83,000	18.0	215	3/16	90,000	21.0	208	3/32	86,000	18.0	198	3/16
					Titanium - Nickel								
WH242	Unalloyed	90,700	22.5	202	1/16	86,700	22.5	196	3/16				
WH247	1.75 Ni	102,000	14.0	218	>1/4*	97,500	15.5	213	1/4				
WH246	2.5 Ni	102,000	12.5	219	1/4	104,000	9.5	288	>1/4*				
WH245	3.5 Ni	115,000	8.5	283	>1/4*	114,300	5.5	305	>1/4*				
WH244	5.0 Ni	112,200	5.5	236	>1/4*	117,700	7.5	236	>1/4*				
WH241	7.5 Ni	120,000	2.5	266	>1/4**	120,700	2.5	278	>1/4**				
WH237	10.0 Ni	122,200	2.0	305	>1/4**	106,900	1.5	299	>1/4**				
WH238	15.0 Ni	(7)	-	-	-	-	-	-	-				

(1) As hot rolled at 1450°F.
(2) Hot-rolled sheet aged 4 hours at 750°F. in air and air cooled.
(3) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(4) Average of 2 longitudinal 14-gauge specimens, 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section.
(5) 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and to the rolling direction.
(6) Minimum bend radius without cracking on a single longitudinal specimen 3 inches long by 0.5 inch wide. Tested with the surface skin present.
(7) Sheet was too brittle to shear into specimens.
* Some ductility.
** Very little ductility.

TABLE 2. HEAT-TREATING AND AGING DATA FOR BINARY ALLOYS OF TITANIUM WITH GERMANIUM OR NICKEL 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 - Germanium											
WH180	Unalloyed	193	188	199	199	181	202	222	232	3/32	3/16	3/32	3/16	3/16	3/16	3/16	>1/4*
WH170	0.10 Ge	190	190	170	181	194	201	206	233	3/64	1/16	3/64	3/64	3/32	3/32	3/16	>1/4*
WH169	0.50 Ge	206	202	176	168	203	198	199	212	3/16	1/16	3/64	3/64	3/16	3/16	3/16	3/16
WH168	1.0 Ge	215	208	194	190	198	214	218	224	3/16	3/32	1/8	1/8	3/16	3/16	3/16	3/16
						Titanium - Nickel											
WH242	Unalloyed	202	196	175	181	186	185	175	213	1/16	3/16	1/16	1/16	1/8	1/8	3/16	3/16
WH247	1.75 Ni	218	213	225	242	233	259	275	307	>1/4*	1/4	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**
WH246	2.5 Ni	219	288	253	251	294	254	270	316	1/4	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH245	3.5 Ni	283	305	337	358	394	374	283	317	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH244	5.0 Ni	236	236	519	461	383	328	529	487	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH241	7.5 Ni	266	278	508	544	519	498	530	542	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH237	10.0 Ni	305	299	542	530	519	544	548	528	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH238(11)	15.0 Ni	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

(1) 10-kg. load. Hardness at the center of the cross section of the sheet specimen 90° to the surface and to the rolling direction. Average of at least 5 readings.
(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 in air 1/2 hour at 1450°F. and quenched in cold water.
(6) Heated in air 1/2 hour at 1550°F. and quenched in cold water.
(7) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(8) Heated in air 1/2 hour at 1650°F. and quenched in cold water.
(9) Heated in air 1/2 hour at 1700°F. and quenched in cold water.
(10) Heated in air 1/2 hour at 1750°F. and quenched in cold water.
(11) Sheet was surface and edge cracked. No tests were made.
* Some ductility.
** Very little ductility.

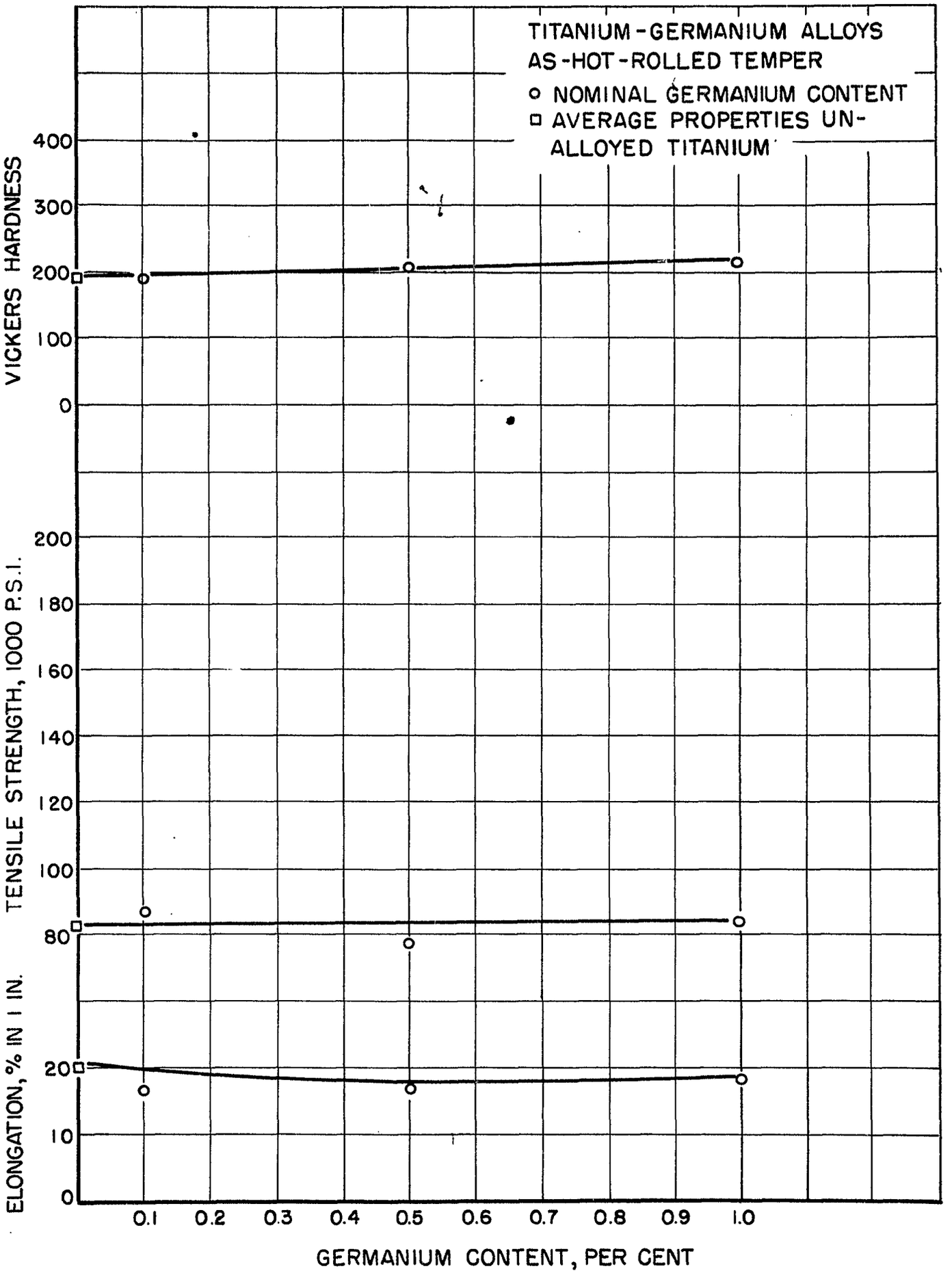

FIGURE 1. EFFECT OF GERMANIUM ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM BINARY ALLOY SHEET

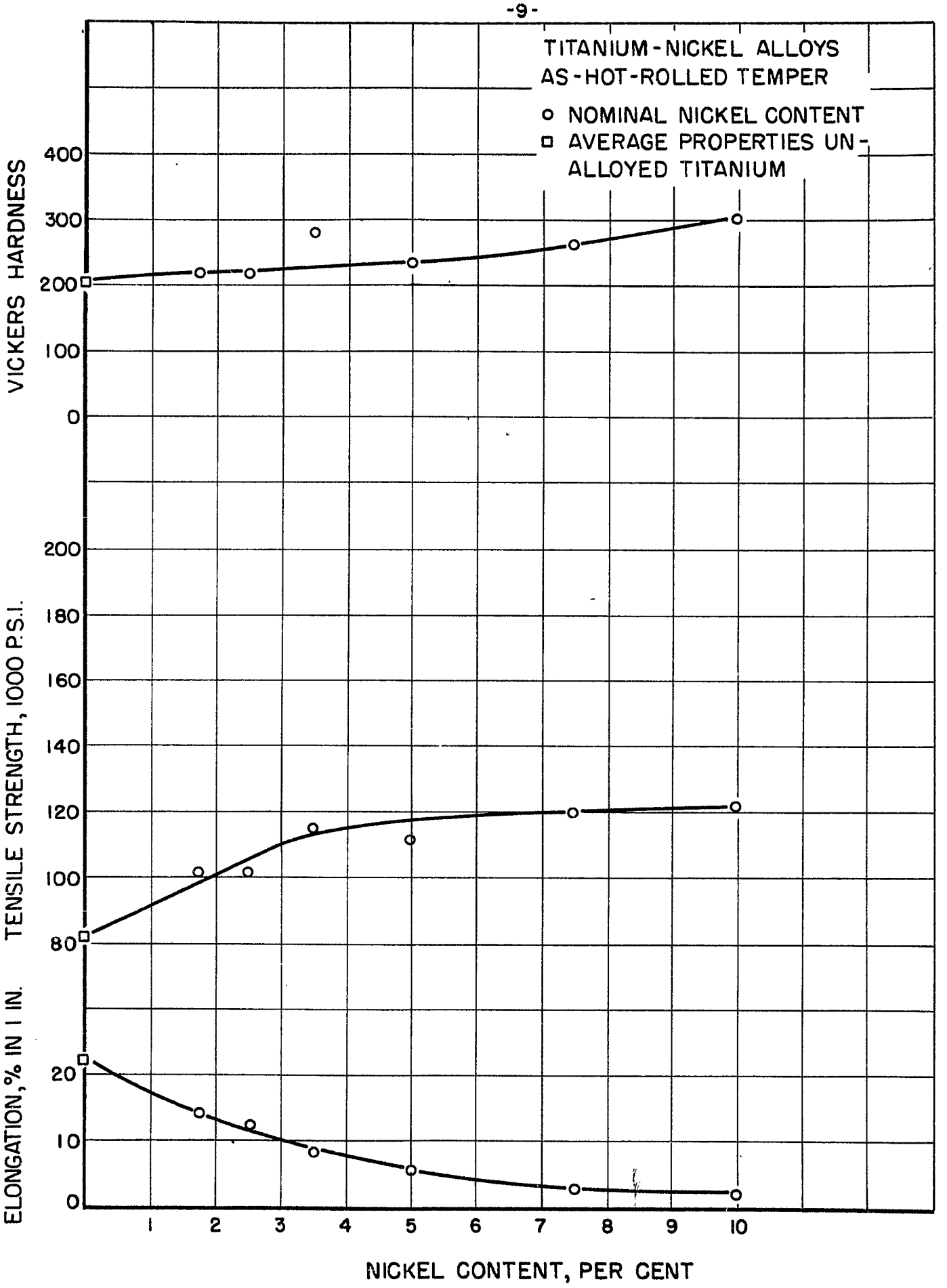

FIGURE 2. EFFECT OF NICKEL ON THE PROPERTIES OF AS-HOT-ROLLED PROCESS A TITANIUM BINARY ALLOY SHEET

TABLE 3. PROPERTIES OF TITANIUM-MOLYBDENUM ALLOYS WITH ADDITIONS OF CARBON, COPPER, CHROMIUM, MANGANESE, IRON, COBALT, OR NICKEL PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
						Titanium - Molybdenum - Carbon							
						0.25% Carbon							
WH233	Unalloyed	88,000	21.5	202	1/16	97,400	21.0	205	1/16				
WH234	2.5 Mo, 0.25 C	128,100	15.0	268	>1/4*	129,000	13.0	279	3/16				
WH236	3.5 Mo, 0.25 C	141,000	10.0	295	>1/4*	149,700	4.0	304	>1/4*				
WH240	5.0 Mo, 0.25 C	146,600	9.0	296	>1/4*	155,300	2.0	320	>1/4**				
						0.5% Carbon							
WH235	2.5 Mo, 0.50 C	143,200	7.5	282	>1/4*	154,700	5.5	297	>1/4*				
WH239	3.5 Mo, 0.50 C	143,200	6.0	279	>1/4*	149,400	7.5	313	>1/4**				
WH243	5.0 Mo, 0.50 C	149,400	6.5	294	>1/4*	161,900	3.0	348	>1/4**				
						Titanium - Molybdenum - Copper							
						1.0% Copper							
WH206	Unalloyed	90,400	20.5	204	3/16	90,000	17.5	199	3/16				
WH213	2.5 Mo, 1.0 Cu	130,000	8.0	264	3/16	133,200	6.0	263	>1/4*				
WH42	3.5 Mo, 1.0 Cu	140,000	9.0	281	>1/4*	156,200	5.0	304	>1/4*				
WH38	5.0 Mo, 1.0 Cu	171,700	5.5	298	>1/4*	180,300	5.0	327	>1/4*				
						2.0% Copper							
WH212	2.5 Mo, 2.0 Cu	138,600	7.5	290	3/16	145,800	2.5	305	>1/4*				
WH209	3.5 Mo, 2.0 Cu	139,100	5.0	279	>1/4*	154,100	4.0	301	>1/4*				
WH171	5.0 Mo, 2.0 Cu	174,700	5.0	315	3/16	178,000	3.0	338	>1/4*				
						Titanium - Molybdenum - Chromium							
						1.0% Chromium							
WH190	Unalloyed	73,600	21.0	181	3/64	74,100	22.0	185	3/32	67,900	18.0	175	3/4
WH194	2.5 Mo, 1.0 Cr	122,500	9.0	262	3/16	126,800	5.0	266	3/16	130,700	2.5	278	>1/4*
WH197	3.5 Mo, 1.0 Cr	151,200	4.0	252	3/16	146,700	3.5	272	>1/4*	119,700	3.0	297	>1/4*
WH201	5.0 Mo, 1.0 Cr	166,100	2.0	356	>1/4*	160,800	3.5	377	>1/4**	113,200	9.0	264	3/16
						2.0% Chromium							
WH195	2.5 Mo, 2.0 Cr	147,700	4.5	320	3/16	154,800	4.5	315	3/16	125,400	2.0	297	>1/4*
WH198	3.5 Mo, 2.0 Cr	158,800	4.0	272	>1/4*	178,500	3.5	324	>1/4*	122,000	3.5	305	>1/4*
WH204	5.0 Mo, 2.0 Cr	158,400	3.5	322	>1/4*	167,900	2.0	361	>1/4*	106,100	8.0	247	3/16

TABLE 3. (Continued)

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
						3.0% Chromium							
WH191	2.5 Mo, 3.0 Cr	162,700	4.0	358	>1/4*	166,000	5.0	358	>1/4**	103,700	3.0	264	>1/4*
WH200	3.5 Mo, 3.0 Cr	155,700	3.0	384	>1/4*	190,900	2.0	383	>1/4*	114,500	2.5	283	>1/4**
WH205	5.0 Mo, 3.0 Cr	195,400	2.5	400	3/16	193,300	2.0	409	>1/4**	111,300	2.5	292	>1/4*
						Titanium - Molybdenum - Manganese 1.0% Manganese							
WH172	Unalloyed	84,900	18.0	190	3/64	83,000	21.5	187	3/32	82,800	14.0	189	3/32
WH186	2.5 Mo, 1.0 Mn	141,100	4.5	275	3/16	130,000	8.0	294	3/16	138,500	2.0	327	>1/4*
WH182	3.5 Mo, 1.0 Mn	147,200	4.0	288	3/16	170,600	6.0	302	>1/4*	132,900	3.5	319	3/16
WH178	5.0 Mo, 1.0 Mn	152,700	5.0	338	>1/4*	178,600	3.0	344	>1/4*	120,000	8.0	281	>1/4*
						2.0% Manganese							
WH183	2.5 Mo, 2.0 Mn	165,000	7.0	326	3/16	149,400	5.0	324	3/16	138,600	1.5	332	>1/4*
WH179	3.5 Mo, 2.0 Mn	167,700	4.0	290	>1/4*	161,700	3.0	317	>1/4*	132,900	2.0	324	3/16
WH173	5.0 Mo, 2.0 Mn	178,800	7.0	351	>1/4*	168,800	2.0	406	>1/4*	126,700	3.0	304	>1/4*
						Titanium - Molybdenum - Iron 1.0% Iron							
WH207	Unalloyed	89,700	22.0	199	3/32	88,700	21.0	191	1/16				
WH218	2.5 Mo, 1.0 Fe	139,400	10.5	248	3/16	141,300	3.5	265	>1/4*				
WH227	3.5 Mo, 1.0 Fe	167,500	6.0	285	>1/4	175,400	5.0	317	>1/4*				
WH223	5.0 Mo, 1.0 Fe	184,200	4.0	317	3/16	177,200	2.5	348	>1/4*				
						2.0% Iron							
WH210	2.5 Mo, 2.0 Fe	136,000	6.0	274	3/16	151,300	4.0	306	>1/4*				
WH222	3.5 Mo, 2.0 Fe	154,700	3.5	308	>1/4	182,000	2.0	337	>1/4**				
WH226	5.0 Mo, 2.0 Fe	164,400	6.5	317	3/16	160,000	3.5	340	>1/4*				
						Titanium - Molybdenum - Cobalt 1.0% Cobalt							
WH180	Unalloyed	83,000	20.5	193	3/32	84,700	19.0	188	3/16	92,900	14.5	181	3/16
WH175	2.5 Mo, 1.0 Co	134,000	9.0	268	3/16	133,400	7.0	296	3/16	132,700	2.5	367	>1/4**
WH184	3.5 Mo, 1.0 Co	155,200	5.0	303	3/16	150,400	2.5	326	>1/4*	161,200	2.0	370	>1/4*
WH188	5.0 Mo, 1.0 Co	180,700	4.5	306	>1/4*	182,500	3.0	331	>1/4*	134,300	2.5	316	>1/4*
						2.0% Cobalt							
WH181	2.5 Mo, 2.0 Co	144,700	7.0	280	3/16	150,900	5.5	296	3/16	56,700	1.0	377	>1/4**
WH185	3.5 Mo, 2.0 Co	163,000	5.0	290	>1/4*	176,000	2.0	312	>1/4*	130,000	2.0	331	>1/4**
WH189	5.0 Mo, 2.0 Co	183,000	4.0	311	>1/4*	160,000	3.0	350	>1/4*	122,600	1.5	417	>1/4**

TABLE 3. (Continued)

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
						Titanium - Molybdenum - Nickel							
						1.0% Nickel							
WH187	Unalloyed	80,800	18.0	185	3/64	79,500	18.0	181	1/16	74,000	15.5	178	3/32
WH203	2.5 Mo, 1.0 Ni	122,700	4.0	263	3/16	136,500	7.0	268	3/16	138,000	1.5	330	>1/4**
WH199	3.5 Mo, 1.0 Ni	157,200	5.0	253	3/16	141,700	8.0	282	>1/4*	147,700	3.0	314	>1/4**
WH193	5.0 Mo, 1.0 Ni	170,100	4.5	316	3/16	179,200	2.0	315	>1/4*	128,300	8.5	301	>1/4*
						2.0% Nickel							
WH202	2.5 Mo, 2.0 Ni	144,300	8.0	265	>1/4*	139,700	5.5	275	3/16	85,700	0.5	366	>1/4**
WH196	3.5 Mo, 2.0 Ni	143,300	2.5	328	3/16	151,700	4.0	353	>1/4*	52,100	0.0	453	>1/4**
WH192	5.0 Mo, 2.0 Ni	185,400	3.0	382	3/16	185,000	2.5	390	>1/4*	100,900	0.0	417	>1/4**

(1) As hot rolled at 1450°F.
(2) Hot-rolled sheet aged 4 hours at 750°F. in air and air cooled.
(3) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(4) Average of 2 longitudinal 14-gauge specimens, 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section.
(5) 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and to the rolling direction.
(6) Minimum bend radius without cracking on a single longitudinal specimen 3 inches long by 0.5 inch wide. Tested with the surface skin present.
* Some ductility.
** Very little ductility.

TABLE 4. HEAT-TREATING AND AGING DATA FOR TITANIUM-MOLYBDENUM ALLOYS WITH ADDITIONS OF CARBON, COPPER, CHROMIUM, MANGANESE, IRON, COBALT, OR NICKEL PREPARED FROM PROCESS A METAL

		Vickers Hardness After Indicated Treatment(1)								Minimum Bend Radius After Indicated Treatment(2)							
Heat No.	Intended Composition, %	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 - Molybdenum - Carbon										
							0.25% Carbon										
WH233	Unalloyed	202	205	177	178	188	186	198	223	1/16	1/16	1/16	1/16	1/16	1/16	1/16	1/16
WH234	2.5 Mo, 0.25 C	268	279	287	272	283	289	290	302	>1/4*	3/16	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH236	3.5 Mo, 0.25 C	295	304	303	269	285	289	287	303	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	1/4**
WH240	5.0 Mo, 0.25 C	296	320	328	267	285	264	274	294	>1/4*	>1/4**	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	1/4**
							0.5% Carbon										
WH235	2.5 Mo, 0.50 C	282	297	299	277	295	309	302	312	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	1/4**
WH239	3.5 Mo, 0.50 C	279	313	304	266	285	294	299	285	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH243	5.0 Mo, 0.50 C	294	348	324	283	278	266	281	312	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
							Titanium - Molybdenum - Copper										
							1.0% Copper										
WH206	Unalloyed	204	199	179	185	206	202	201	219	3/16	3/16	3/16	3/16	3/16	1/8	1/8	>1/4*
WH213	2.5 Mo, 1.0 Cu	264	263	250	283	285	306	296	302	3/16	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4*	>1/4**
WH42	3.5 Mo, 1.0 Cu	281	304	244	274	301	297	287	313	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH38	5.0 Mo, 1.0 Cu	298	327	293	277	266	276	292	278	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
							2.0% Copper										
WH212	2.5 Mo, 2.0 Cu	290	305	319	354	337	339	354	376	3/16	>1/4*	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**
WH209	3.5 Mo, 2.0 Cu	279	301	287	309	321	339	351	327	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH171	5.0 Mo, 2.0 Cu	315	338	283	302	302	309	302	317	3/16	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
							Titanium - Molybdenum - Chromium										
							1.0% Chromium										
WH190	Unalloyed	181	185	155	153	175	195	191	199	3/64	3/32	3/64	3/64	3/64	3/32	3/16	3/16
WH194	2.5 Mo, 1.0 Cr	262	266	239	295	278	286	286	277	3/16	3/16	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH197	3.5 Mo, 1.0 Cr	252	272	248	268	297	281	287	263	3/16	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH201	5.0 Mo, 1.0 Cr	356	377	261	262	264	259	274	266	>1/4*	>1/4**	3/16	3/16	3/16	>1/4*	>1/4*	>1/4**
							2.0% Chromium										
WH195	2.5 Mo, 2.0 Cr	320	315	256	294	297	291	290	294	3/16	3/16	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH198	3.5 Mo, 2.0 Cr	272	324	281	298	305	262	284	286	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
WH204	5.0 Mo, 2.0 Cr	322	361	298	256	247	257	256	261	>1/4*	>1/4*	3/32	3/32	3/16	>1/4*	>1/4*	>1/4**

TABLE 4. (Continued)

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)
							3.0% Chromium										
WH191	2.5 Mo, 3.0 Cr	358	358	276	292	264	270	272	282	>1/4*	>1/4**	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**
WH200	3.5 Mo, 3.0 Cr	384	383	354	351	283	262	327	285	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH205	5.0 Mo, 3.0 Cr	400	409	330	307	292	277	264	283	3/16	>1/4**	3/16	3/16	>1/4*	>1/4*	>1/4*	>1/4**
							Titanium - Molybdenum - Manganese										
							1.0% Manganese										
WH172	Unalloyed	190	187	169	176	189	201	207	193	3/64	3/32	3/64	3/64	3/32	3/32	3/16	3/16
WH186	2.5 Mo, 1.0 Mn	275	294	291	310	327	302	342	353	3/16	3/16	3/16	3/16	>1/4*	>1/4*	>1/4*	>1/4*
WH182	3.5 Mo, 1.0 Mn	288	302	350	318	319	329	314	324	3/16	>1/4*	3/16	3/16	3/16	>1/4*	>1/4*	>1/4*
WH178	5.0 Mo, 1.0 Mn	338	344	333	273	281	312	294	294	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**
							2.0% Manganese										
WH183	2.5 Mo, 2.0 Mn	326	324	391	359	332	369	368	373	3/16	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**
WH179	3.5 Mo, 2.0 Mn	290	317	398	333	324	355	370	355	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**
WH173	5.0 Mo, 2.0 Mn	351	406	363	363	304	322	321	375	>1/4*	>1/4*	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**
							Titanium - Molybdenum - Iron										
							1.0% Iron										
WH207	Unalloyed	199	191	171	160	170	188	199	216	3/32	1/16	1/16	1/16	3/32	3/32	3/16	>1/4*
WH218	2.5 Mo, 1.0 Fe	248	265	310	370	376	339	358	363	3/16	>1/4*	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH227	3.5 Mo, 1.0 Fe	285	317	319	363	353	389	364	399	>1/4	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH223	5.0 Mo, 1.0 Fe	317	348	407	328	319	383	382	394	3/16	>1/4*	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**
							2.0% Iron										
WH210	2.5 Mo, 2.0 Fe	274	306	407	421	425	408	442	401	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH222	3.5 Mo, 2.0 Fe	308	337	473	371	413	442	296	339	>1/4	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH226	5.0 Mo, 2.0 Fe	317	340	368	376	409	444	361	264	3/16	>1/4*	3/16	>1/4*	3/16	>1/4*	>1/4**	>1/4**
							Titanium - Molybdenum - Cobalt										
							1.0% Cobalt										
WH180	Unalloyed	193	188	199	199	181	202	222	232	3/32	3/16	3/32	3/16	3/16	3/16	3/16	>1/4*
WH175	2.5 Mo, 1.0 Co	268	296	297	356	367	336	336	354	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH184	3.5 Mo, 1.0 Co	303	326	314	349	370	408	330	396	3/16	>1/4*	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**
WH188	5.0 Mo, 1.0 Co	306	331	355	314	316	310	321	372	>1/4*	>1/4*	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**

TABLE 4. (Continued)

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)
							2.0% Cobalt										
WH181	2.5 Mo, 2.0 Co	280	296	390	380	377	499	373	421	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH185	3.5 Mo, 2.0 Co	290	312	390	372	331	455	331	483	>1/4*	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH189	5.0 Mo, 2.0 Co	311	350	357	421	417	401	401	390	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
							Titanium - Molybdenum - Nickel										
							1.0% Nickel										
WH187	Unalloyed	185	181	163	172	178	187	201	213	3/64	1/16	3/64	3/64	3/32	3/32	3/16	3/16
WH203	2.5 Mo, 1.0 Ni	263	268	254	319	330	331	306	309	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH199	3.5 Mo, 1.0 Ni	253	282	308	350	314	345	332	326	3/16	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH193	5.0 Mo, 1.0 Ni	316	315	404	351	301	311	330	323	3/16	>1/4*	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**
							2.0% Nickel										
WH202	2.5 Mo, 2.0 Ni	265	275	349	401	366	334	363	373	>1/4*	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH196	3.5 Mo, 2.0 Ni	328	353	446	387	453	446	488	417	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH192	5.0 Mo, 2.0 Ni	382	390	402	405	417	453	394	455	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**

(1) 10-kg. load. Hardness at the center of the cross section of the sheet specimen 90° to the surface and to the rolling direction. Average of at least 5 readings.
(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 in air 1/2 hour at 1450°F. and quenched in cold water.
(6) Heated in air 1/2 hour at 1550°F. and quenched in cold water.
(7) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(8) Heated in air 1/2 hour at 1650°F. and quenched in cold water.
(9) Heated in air 1/2 hour at 1700°F. and quenched in cold water.
(10) Heated in air 1/2 hour at 1750°F. and quenched in cold water.
* Some ductility.
** Very little ductility.

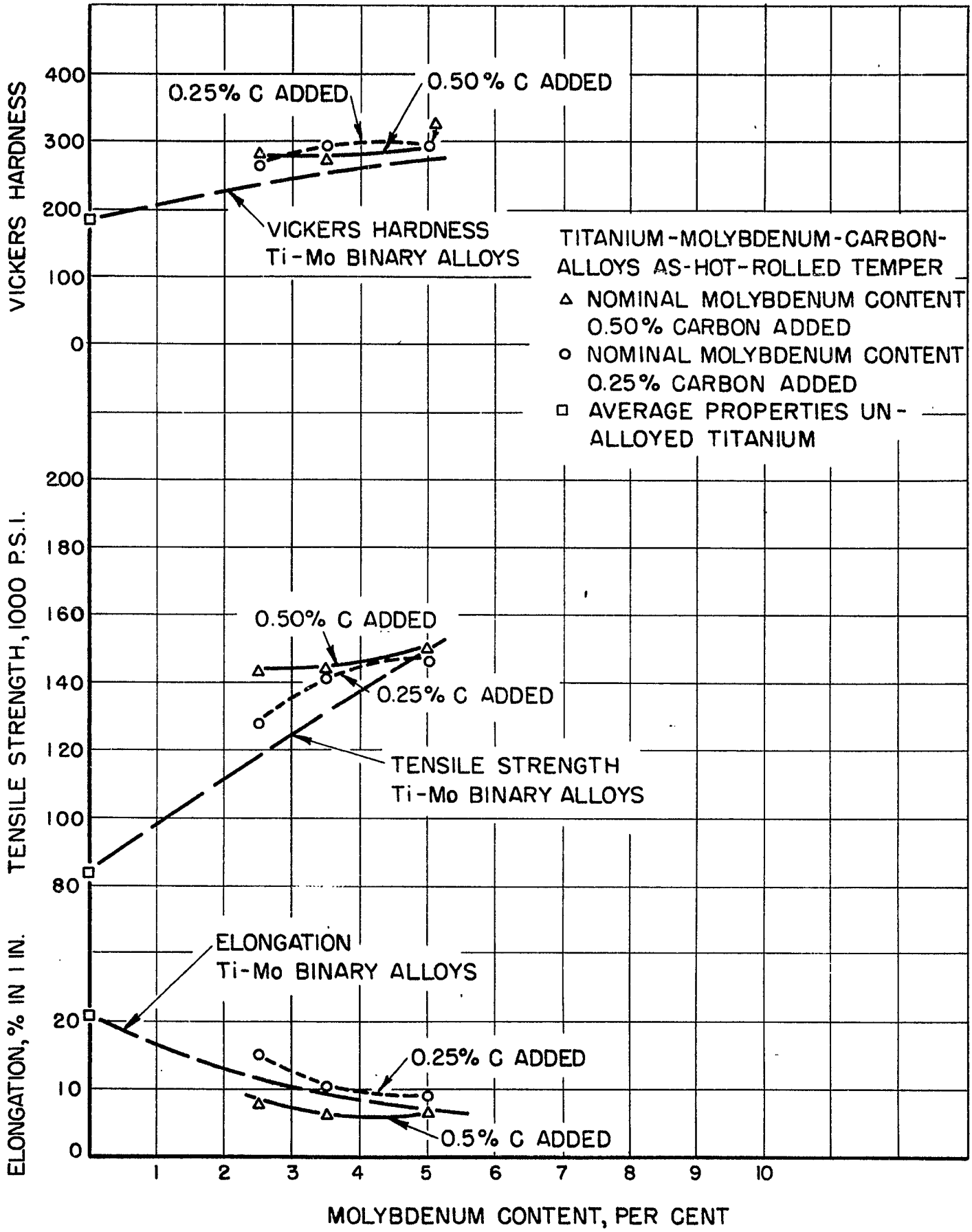

FIGURE 3. EFFECT OF CARBON ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MOLYBDENUM ALLOYS-PREPARED FROM PROCESS A METAL

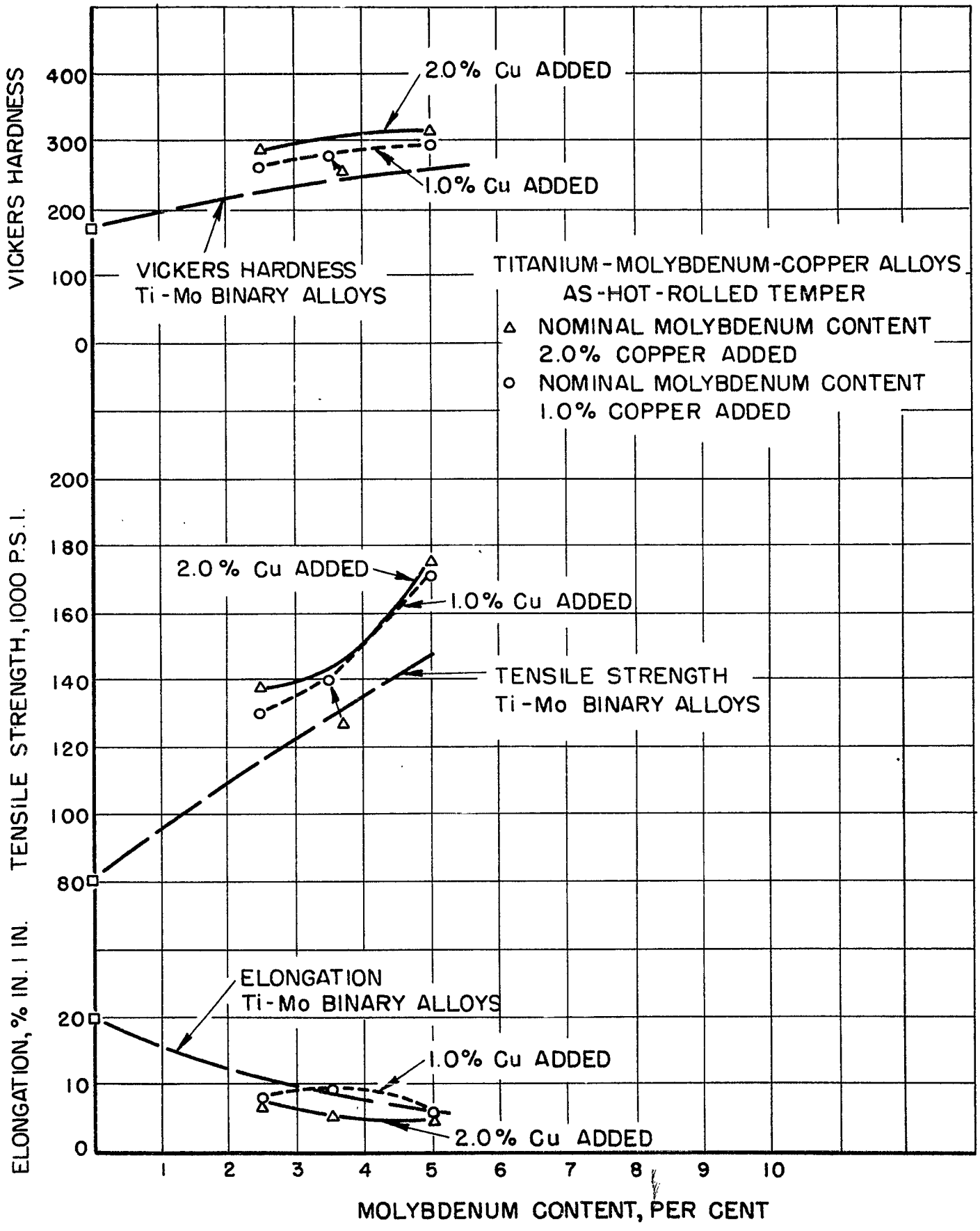

FIGURE 4. EFFECT OF COPPER ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MOLYBDENUM ALLOYS-PREPARED FROM PROCESS A METAL

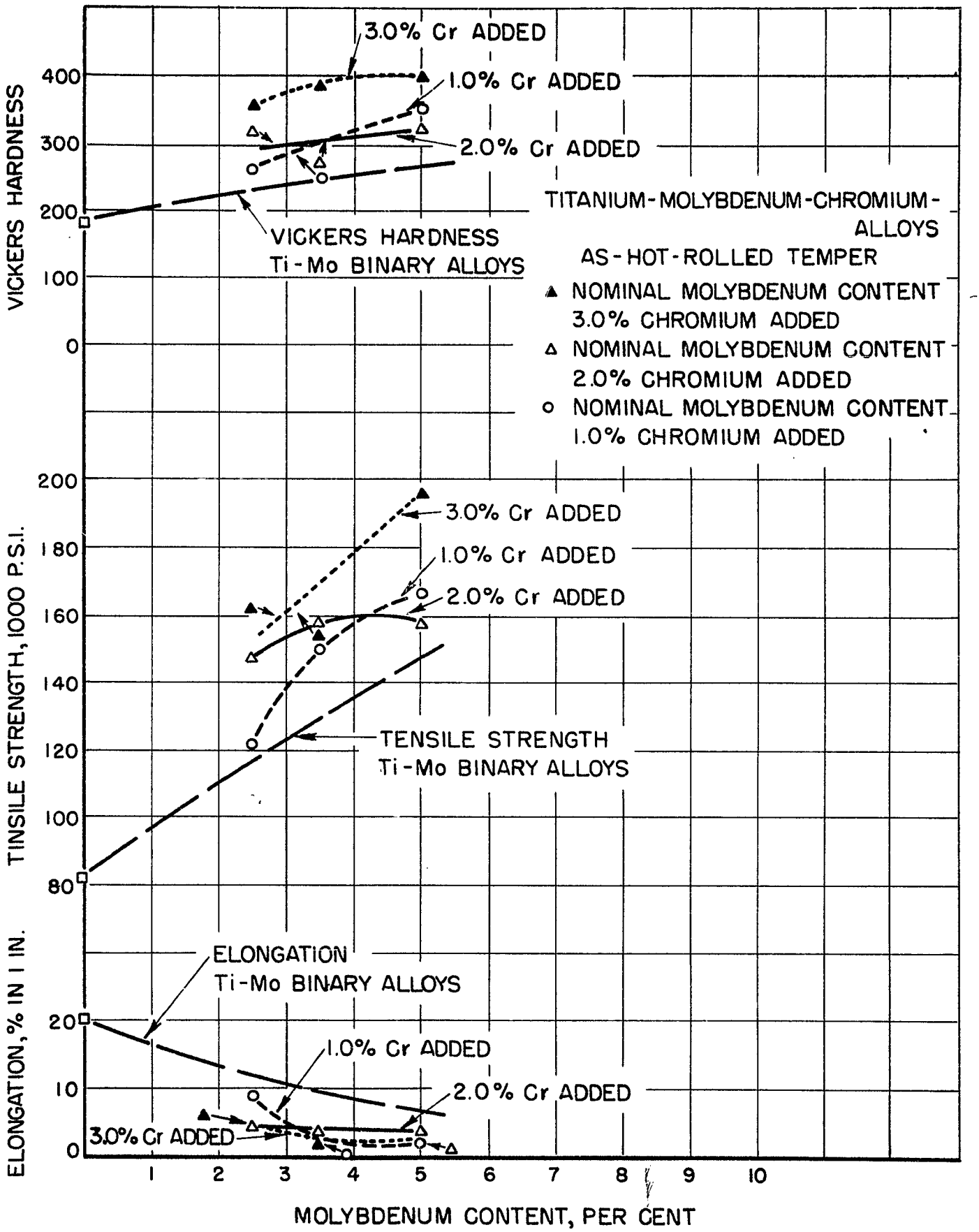

FIGURE 5. EFFECT OF CHROMIUM ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MOLYBDENUM ALLOYS. PREPARED FROM PROCESS A METAL

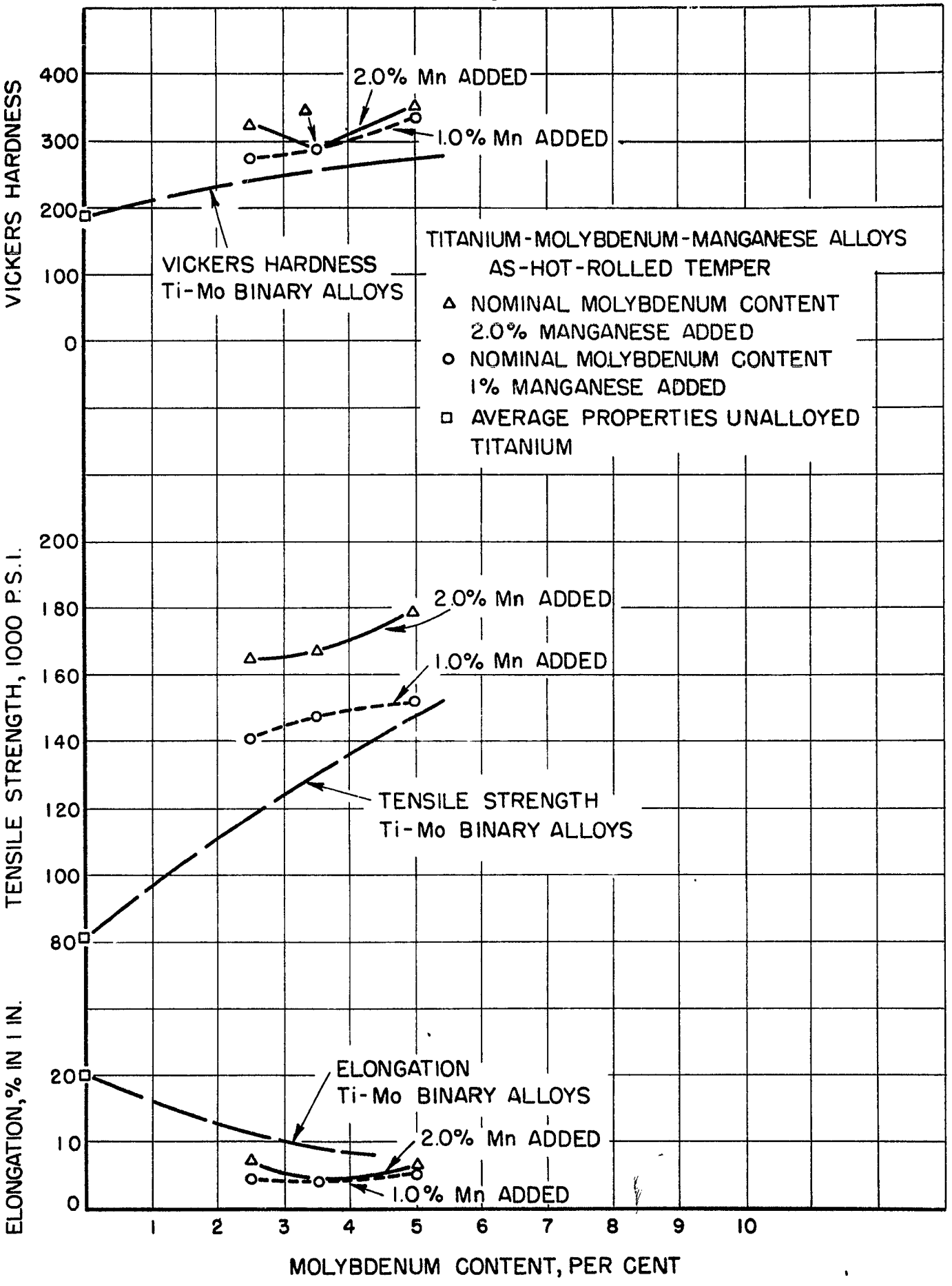

FIGURE 6. EFFECT OF MANGANESE ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM MOLYBDENUM ALLOYS-PREPARED FROM PROCESS A METAL

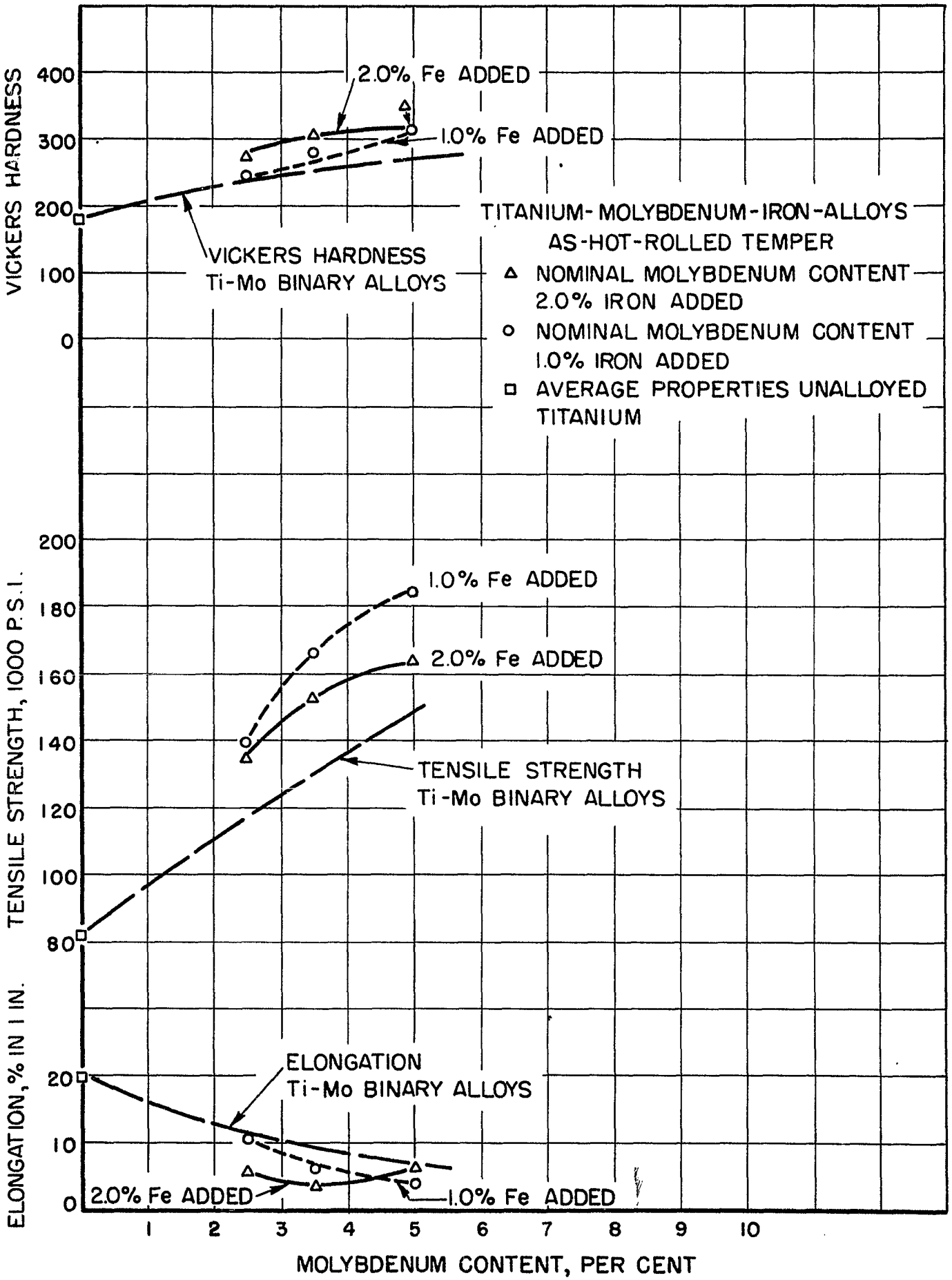

FIGURE 7. EFFECT OF IRON ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MOLYBDENUM ALLOYS. PREPARED FROM PROCESS A METAL

0-13287

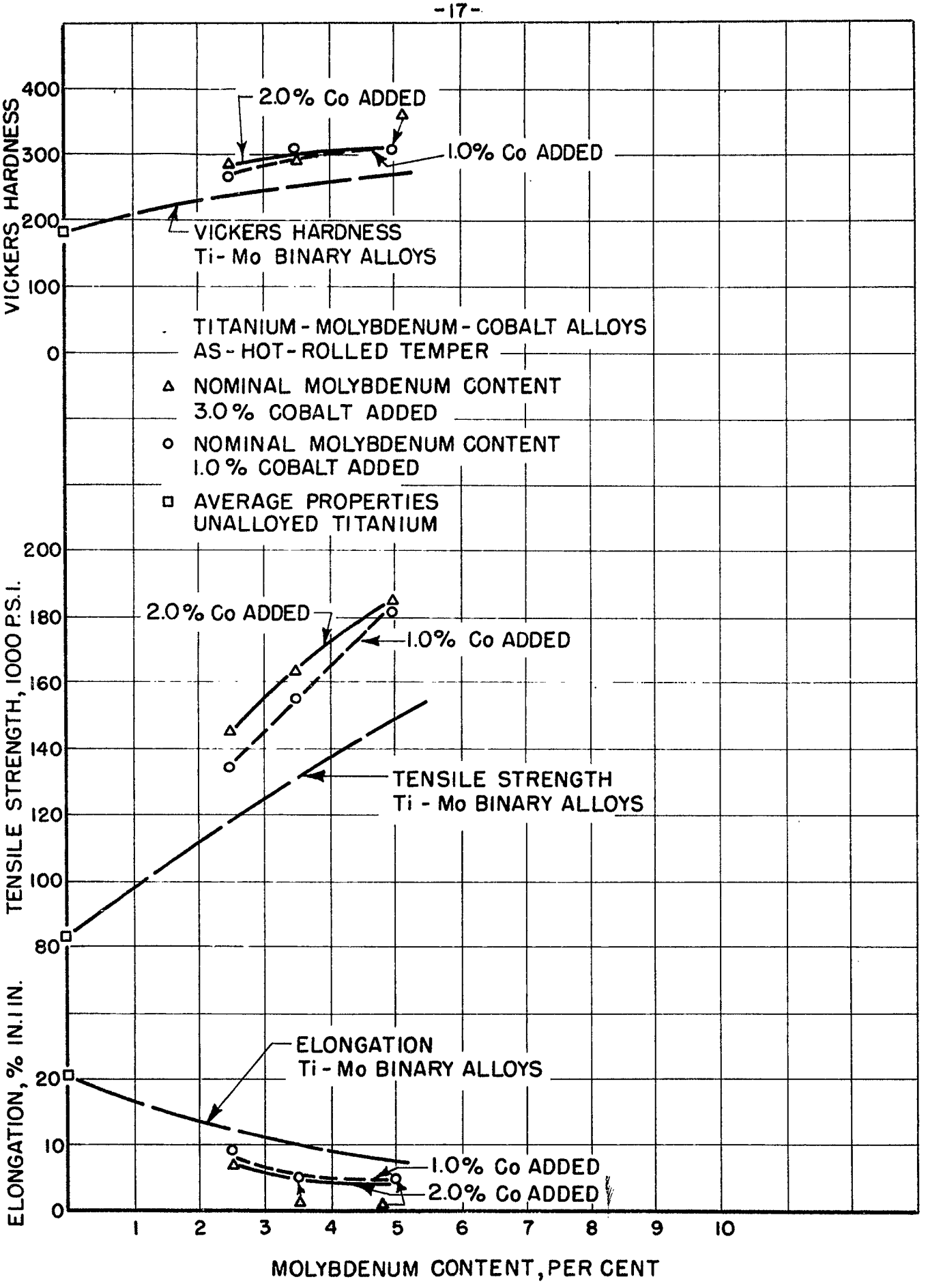

FIGURE 8. EFFECT OF COBALT ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM MOLYBDENUM ALLOYS-PREPARED FROM PROCESS A METAL

0-13288

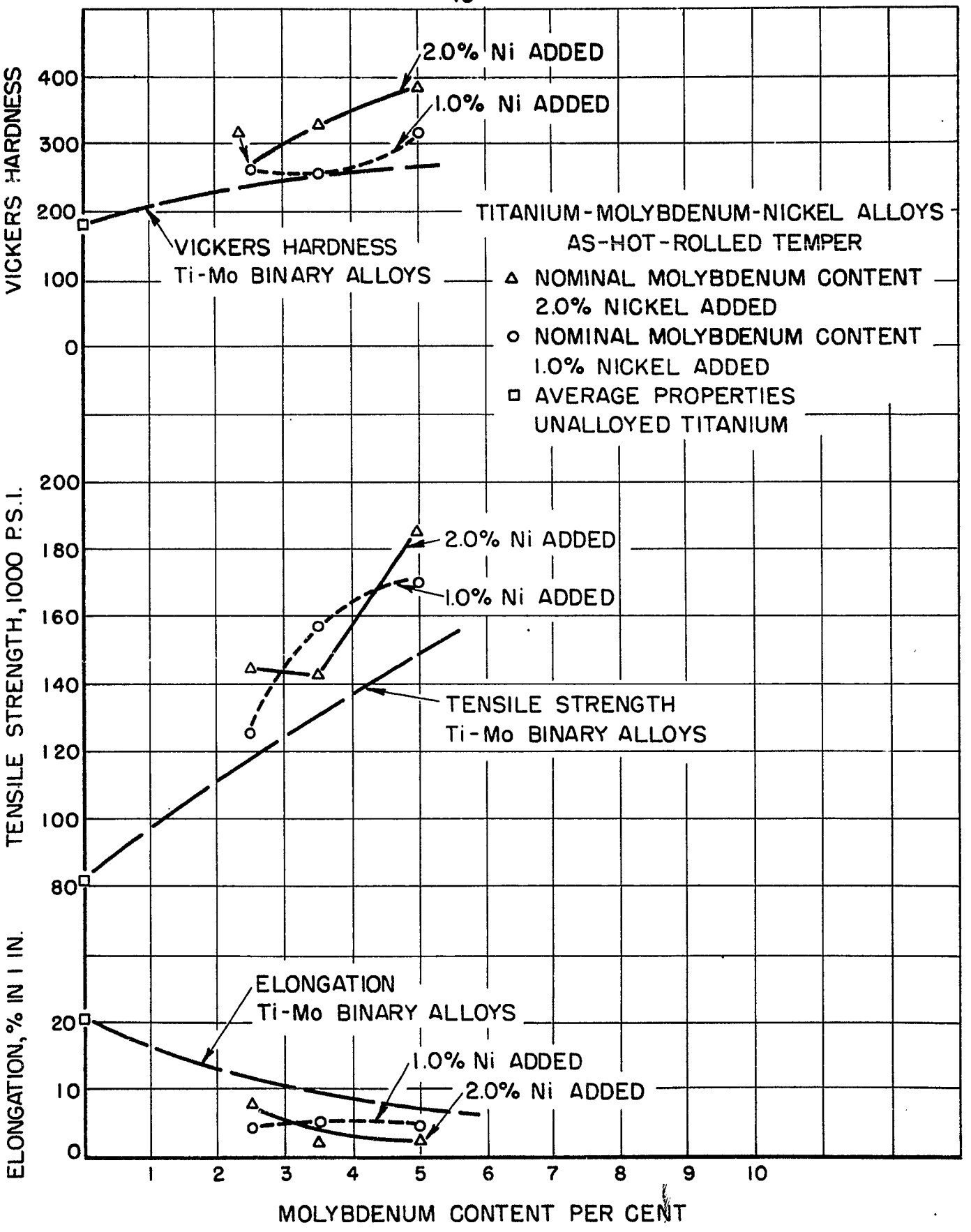

FIGURE 9. EFFECT OF NICKEL ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MOLYBDENUM ALLOYS-PREPARED FROM PROCESS A METAL.

from the above additions to binary titanium-molybdenum base alloys in the as-hot-rolled condition. In these graphs, the tensile properties and hardness of as-hot-rolled binary titanium-molybdenum alloys have been reproduced as dashed lines from data reported previously.

In general, the tensile strength and hardness of the titanium-molybdenum alloys were increased, with a corresponding decrease in elongation, by these additions. However, the addition of 1.0 and 2.0 per cent copper, 2.0 per cent manganese, and 2.0 per cent iron to the titanium - 5 per cent molybdenum alloys showed a large increase in tensile strength with essentially the same elongation values as the binary alloys. The properties of these alloys were:

Heat No.	Intended Composition, Per Cent	Tensile Strength, p.s.i.	Elongation, Per Cent in 1 Inch	VHN	Minimum Bend Radius
WC84(1)	5.0 Mo	144,900	6.5	268	>1/4*
WC38	5.0 Mo, 1.0 Cu	171,700	5.5	298	>1/4*
WH171	5.0 Mo, 2.0 Cu	174,700	5.0	315	3/16*
SH173	5.0 Mo, 1.0 Mn	178,800	7.0	351	>1/4*
WH226	5.0 Mo, 2.0 Fe	164,400	6.5	317	3/16

(1) Data from Table 18, page 162, of previous progress report.

* Some bend ductility.

The addition of 3.0 per cent chromium, 1.0 per cent iron, 2.0 per cent cobalt, or 2.0 per cent nickel to the titanium - 5 per cent molybdenum base gave tensile strengths of 195,400, 184,200, 183,000, and 185,400 p.s.i., respectively. However, the elongation of these alloys was lower than that of the binary base.

These alloys in the as-hot-rolled temper were aged 4 hours at 750°F. and the tensile properties determined. As shown by the data in Table 3, the tensile strength of many of these alloys in the lower ranges of molybdenum can be improved by aging the as-hot-rolled sheet without too great a sacrifice in ductility.

The properties of titanium-manganese base alloys containing additions of nitrogen, copper, molybdenum, and cobalt are listed in Tables 5 and 6. Figures 10 to 13 illustrate graphically the effect of the above additions on the tensile strength, elongation, and hardness of the titanium-manganese base. In these figures, the curves for the binary titanium-manganese alloys have been reproduced as dashed lines from previous data.

The addition of 0.1 per cent nitrogen to the titanium-manganese base increased the tensile strength about 10,000 p.s.i. without any sacrifice in elongation. With 0.2 per cent nitrogen added, a further increase in tensile strength was obtained, but at a considerable sacrifice in ductility. The alloys containing 7.5 and 10.0 per cent manganese and 0.2 per cent nitrogen were too brittle to shear.

The ternary titanium-manganese base alloys which had good tensile strength and elongation values in the as-hot-rolled temper were:

TABLE 5. PROPERTIES OF TITANIUM-MANGANESE ALLOYS WITH ADDITIONS OF NITROGEN, COPPER, MOLYBDENUM, OR COBALT PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) pls.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
						Titanium - Manganese - Nitrogen							
						0.1% Nitrogen							
WH155	Unalloyed	88,000	10.5	175	3/64	75,000	23.0	184	1/32	74,300	17.5	174	3/32
WH156	1.75 Mn, 0.10 N	161,600	11.0	321	3/16	127,300	11.5	337	3/16	128,700	1.5	359	>1/4**
WH158	2.5 Mn, 0.10 N	146,000	9.5	314	3/16	128,200	1.5	309	3/16	159,100	2.0	410	>1/4**
WH160	3.5 Mn, 0.10 N	156,900	11.0	350	3/16	156,200	6.0	341	>1/4*	108,700	1.0	425	>1/4**
WH162	5.0 Mn, 0.10 N	173,500	8.5	401	>1/4*	136,000	1.0	474	>1/4**	111,600	1.0	428	>1/4**
WH166	7.5 Mn, 0.10 N	184,400	3.5	403	>1/4**	190,000	5.0	498	>1/4**	121,900	1.0	389	>1/4**
WH164	10.0 Mn, 0.10 N	(7)	-	455	-	-	-	-	-	-	-	-	-
						0.2% Nitrogen							
WH157	1.75 Mn, 0.20 N	143,700	8.5	339	>1/4*	139,900	4.5	341	>1/4**	161,300	2.0	374	>1/4**
WH159	2.5 Mn, 0.20 N	134,400	5.5	272	3/16	152,900	6.5	272	>1/4**	131,300	2.0	481	>1/4**
WH161	3.5 Mn, 0.20 N	167,200	2.5	368	3/16	175,000	2.0	370	>1/4*	143,800	0.0	446	>1/4**
WH163	5.0 Mn, 0.20 N	176,000	2.0	405	>1/4*	218,300	0.0	413	>1/4**	157,300	0.0	427	>1/4**
WH167	7.5 Mn, 0.20 N	(7)	-	403	-	-	-	-	-	-	-	-	-
WH165	10.0 Mn, 0.20 N	(7)	-	455	-	-	-	-	-	-	-	-	-
						Titanium - Manganese - Copper							
						1.0% Copper							
WH122	Unalloyed	78,600	22.5	189	3/64	75,900	21.5	184	1/16	75,000	19.5	166	3/32
WH125	1.75 Mn, 1.0 Cu	114,300	12.5	264	3/16	120,300	11.5	271	3/16	123,200	5.5	290	>1/4**
WH133	2.5 Mn, 1.0 Cu	136,600	10.0	294	3/16	110,900	5.0	306	3/16	121,400	0.5	401	>1/4**
WH137	3.5 Mn, 1.0 Cu	201,400	1.0	245	3/16	120,000	6.5	265	>1/4*	111,300	1.5	300	>1/4**
WH144	5.0 Mn, 1.0 Cu	195,000	2.0	417	>1/4**	164,100	2.0	472	3/16	90,700	1.0	414	>1/4**
						2.0% Copper							
WH126	1.75 Mn, 2.0 Cu	167,300	4.5	261	3/16	170,000	2.0	267	>1/4*	112,000	1.0	375	>1/4**
WH134	2.5 Mn, 2.0 Cu	139,800	6.0	266	>1/4*	153,900	5.0	289	>1/4*	101,800	2.0	390	>1/4**
WH138	3.5 Mn, 2.0 Cu	138,800	5.0	240	3/16	120,400	8.0	254	>1/4*	122,500	1.0	394	>1/4**
WH145	5.0 Mn, 2.0 Cu	166,600	7.5	351	>1/4**	148,500	1.0	364	>1/4*	91,700	0.0	413	>1/4**

TABLE 5. (Continued)

Heat No.	Intended Composition, %	As Hot Rolled at 1450°F.(1) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Hot Rolled at 1450°F. and Aged 4 Hrs. at 750°F.(2) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch	Heat Treated at 1600°F.(3) Tensile Strength,(4) p.s.i.	Elong., % in 1 Inch	VHN(5)	Minimum Bend Radius,(6) Inch
						Titanium - Manganese - Molybdenum							
						1.0% Molybdenum							
WH220	Unalloyed	88,800	21.5	199	3/16	90,100	19.5	203	3/16				
WH221	1.75 Mn, 1.0 Mo	129,700	9.0	275	3/16	126,500	10.0	285	3/16				
WH225	2.5 Mn, 1.0 Mo	131,600	9.0	349	3/16	132,500	9.0	270	3/16				
WH229	3.5 Mn, 1.0 Mo	151,100	6.0	313	3/16	142,300	5.0	335	3/16				
WH231	5.0 Mn, 1.0 Mo	165,100	3.5	331	3/16	167,500	6.5	353	>1/4**				
						2.0% Molybdenum							
WH224	1.75 Mn, 2.0 Mo	131,400	8.5	301	3/16	144,200	7.0	304	1/4				
WH228	2.5 Mn, 2.0 Mo	147,700	6.0	281	3/16	157,700	6.5	304	>1/4*				
WH230	3.5 Mn, 2.0 Mo	184,200	3.5	372	3/16	160,000	4.5	351	>1/4*				
WH232	5.0 Mn, 2.0 Mo	184,400	3.5	390	>1/4**	167,600	3.5	392	>1/4**				
						Titanium - Manganese - Cobalt							
						1.0% Cobalt							
WH146	Unalloyed	82,900	22.5	194	3/32	79,000	21.0	193	3/32	82,900	18.0	199	3/32
WH147	1.75 Mn, 1.0 Co	121,000	12.5	232	>1/4**	117,900	9.0	238	3/16	113,700	1.5	333	>1/4**
WH148	2.5 Mn, 1.0 Co	140,700	12.0	290	3/16	145,400	6.5	318	3/16	122,800	2.0	424	>1/4**
WH151	3.5 Mn, 1.0 Co	166,700	6.0	297	3/16	201,800	3.0	285	>1/4*	140,700	2.0	395	>1/4**
WH153	5.0 Mn, 1.0 Co	150,400	7.0	380	3/16	160,300	1.5	408	3/16	92,800	1.0	407	>1/4**
						2.0% Cobalt							
WH149	1.75 Mn, 2.0 Co	128,800	11.0	259	3/16	122,700	4.0	291	3/16	-	-	406	>1/4**
WH150	2.5 Mn, 2.0 Co	143,600	10.0	262	3/16	176,700	6.0	307	>1/4**	110,700	1.5	459	>1/4**
WH152	3.5 Mn, 2.0 Co	160,200	8.5	324	3/16	163,300	7.5	341	>1/4*	-	-	421	>1/4**
WH154	5.0 Mn, 2.0 Co	179,400	3.0	378	>1/4*	182,800	2.0	394	>1/4*	88,300	0.0	408	>1/4**

(1) As hot rolled at 1450°F.
(2) Hot-rolled sheet aged 4 hours at 750°F. in air and air cooled.
(3) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(4) Average of 2 longitudinal 14-gauge specimens, 3 inches long by 0.375 inch wide with a 0.250-inch-wide reduced section.
(5) 10-kg. load. Hardness at the center of the cross section of the sheet 90° to the surface and to the rolling direction.
(6) Minimum bend radius without cracking on a single longitudinal specimen 3 inches long by 0.5 inch wide. Tested with the surface skin present.
(7) Sheet was too brittle to shear into specimens.
* Some ductility.
** Very little ductility.

TABLE 6. HEAT-TREATING AND AGING DATA FOR TITANIUM-MANGANESE ALLOYS WITH ADDITIONS OF COPPER, MOLYBDENUM, OR COBALT PREPARED FROM PROCESS A METAL

Heat No.	Intended Composition, %	Vickers Hardness After Indicated Treatment(1): 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)	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)
						Titanium - Manganese - Nitrogen											
						0.1% Nitrogen											
WH155	Unalloyed	175	184	153	161	174	190	201	220	3/64	1/32	1/32	3/64	3/32	3/16	3/16	>1/4**
WH156	1.75 Mn, 0.10 N	321	337	336	275	359	353	285	404	3/16	3/16	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH158	2.5 Mn, 0.10 N	314	309	386	446	410	411	445	361	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH160	3.5 Mn, 0.10 N	350	341	374	402	425	437	446	459	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH162	5.0 Mn, 0.10 N	401	474	383	417	428	442	445	464	>1/4*	>1/4**	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH166	7.5 Mn, 0.10 N	403	498	405	464	389	370	473	434	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH164(11)	10.0 Mn, 0.10 N	455	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
						0.2% Nitrogen											
WH157	1.75 Mn, 0.20 N	339	341	345	348	374	397	409	413	>1/4*	>1/4**	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH159	2.5 Mn, 0.20 N	272	272	385	320	481	373	425	470	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH161	3.5 Mn, 0.20 N	368	370	407	442	446	447	447	478	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH163	5.0 Mn, 0.20 N	405	413	395	432	427	437	464	464	>1/4*	>1/4**	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH167(11)	7.5 Mn, 0.20 N	403	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
WH165(11)	10.0 Mn, 0.20 N	455	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
						Titanium - Manganese - Copper											
						1.0% Copper											
WH122	Unalloyed	189	184	165	174	166	204	218	232	3/64	1/16	3/64	3/64	3/32	3/16	>1/4*	>1/4*
WH125	1.75 Mn, 1.0 Cu	264	271	287	338	290	299	377	316	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH133	2.5 Mn, 1.0 Cu	294	306	346	325	401	405	284	412	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH137	3.5 Mn, 1.0 Cu	245	265	394	318	300	433	408	341	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH144	5.0 Mn, 1.0 Cu	417	472	394	394	414	413	370	446	>1/4**	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
						2.0% Copper											
WH126	1.75 Mn, 2.0 Cu	261	267	421	425	375	357	437	429	3/16	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH134	2.5 Mn, 2.0 Cu	266	289	310	323	390	383	348	425	>1/4*	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH138	3.5 Mn, 2.0 Cu	240	254	225	333	394	411	294	317	3/16	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH145	5.0 Mn, 2.0 Cu	351	364	447	459	413	446	433	421	>1/4**	>1/4*	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**

TABLE 6. (Continued)

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 - Manganese - Molybdenum										
							1.0% Molybdenum										
WH220	Unalloyed	199	203	176	172	199	188	221	182	3/16	3/16	1/16	1/16	1/16	3/16	3/16	3/16
WH221	1.75 Mn, 1.0 Mo	275	285	253	373	299	330	366	380	3/16	3/16	3/16	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**
WH225	2.5 Mn, 1.0 Mo	349	270	318	339	344	383	387	394	3/16	3/16	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**
WH229	3.5 Mn, 1.0 Mo	313	335	393	390	405	410	367	373	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH231	5.0 Mn, 1.0 Mo	331	353	413	404	400	387	383	419	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
							2.0% Molybdenum										
WH224	1.75 Mn, 2.0 Mo	301	304	284	342	327	340	345	338	3/16	1/4	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH228	2.5 Mn, 2.0 Mo	281	304	315	357	340	378	390	383	3/16	>1/4*	1/4	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**
WH230	3.5 Mn, 2.0 Mo	372	351	302	368	363	383	390	398	3/16	>1/4*	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**
WH232	5.0 Mn, 2.0 Mo	390	392	401	363	372	360	348	390	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
							Titanium - Manganese - Cobalt										
							1.0% Cobalt										
WH146	Unalloyed	194	193	178	184	199	217	226	234	3/32	3/32	3/64	3/32	3/32	3/16	>1/4*	>1/4**
WH147	1.75 Mn, 1.0 Co	232	238	323	335	333	339	366	384	>1/4**	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH148	2.5 Mn, 1.0 Co	290	318	390	446	424	437	464	436	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH151	3.5 Mn, 1.0 Co	297	285	321	409	395	379	469	429	3/16	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH153	5.0 Mn, 1.0 Co	380	408	390	434	407	423	443	468	3/16	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
							2.0% Cobalt										
WH149	1.75 Mn, 2.0 Co	259	291	397	390	406	438	401	439	3/16	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH150	2.5 Mn, 2.0 Co	262	307	318	409	459	394	473	429	3/16	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH152	3.5 Mn, 2.0 Co	324	341	430	489	421	478	422	483	3/16	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**
WH154	5.0 Mn, 2.0 Co	378	394	401	386	408	408	446	441	>1/4*	>1/4*	>1/4*	>1/4**	>1/4**	>1/4**	>1/4**	>1/4**

(1) 10-kg load. Hardness at the center of the cross section of the sheet specimen 90° to the surface and to the rolling direction. Average of at least 5 readings.
(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 in air 1/2 hour at 1450°F. and quenched in cold water.
(6) Heated in air 1/2 hour at 1550°F. and quenched in cold water.
(7) Heated in air 1/2 hour at 1600°F. and quenched in cold water.
(8) Heated in air 1/2 hour at 1650°F. and quenched in cold water.
(9) Heated in air 1/2 hour at 1700°F. and quenched in cold water.
(10) Heated in air 1/2 hour at 1750°F. and quenched in cold water.
(11) Sheet was too brittle to shear into specimens.
* Some ductility.
** Very little ductility.

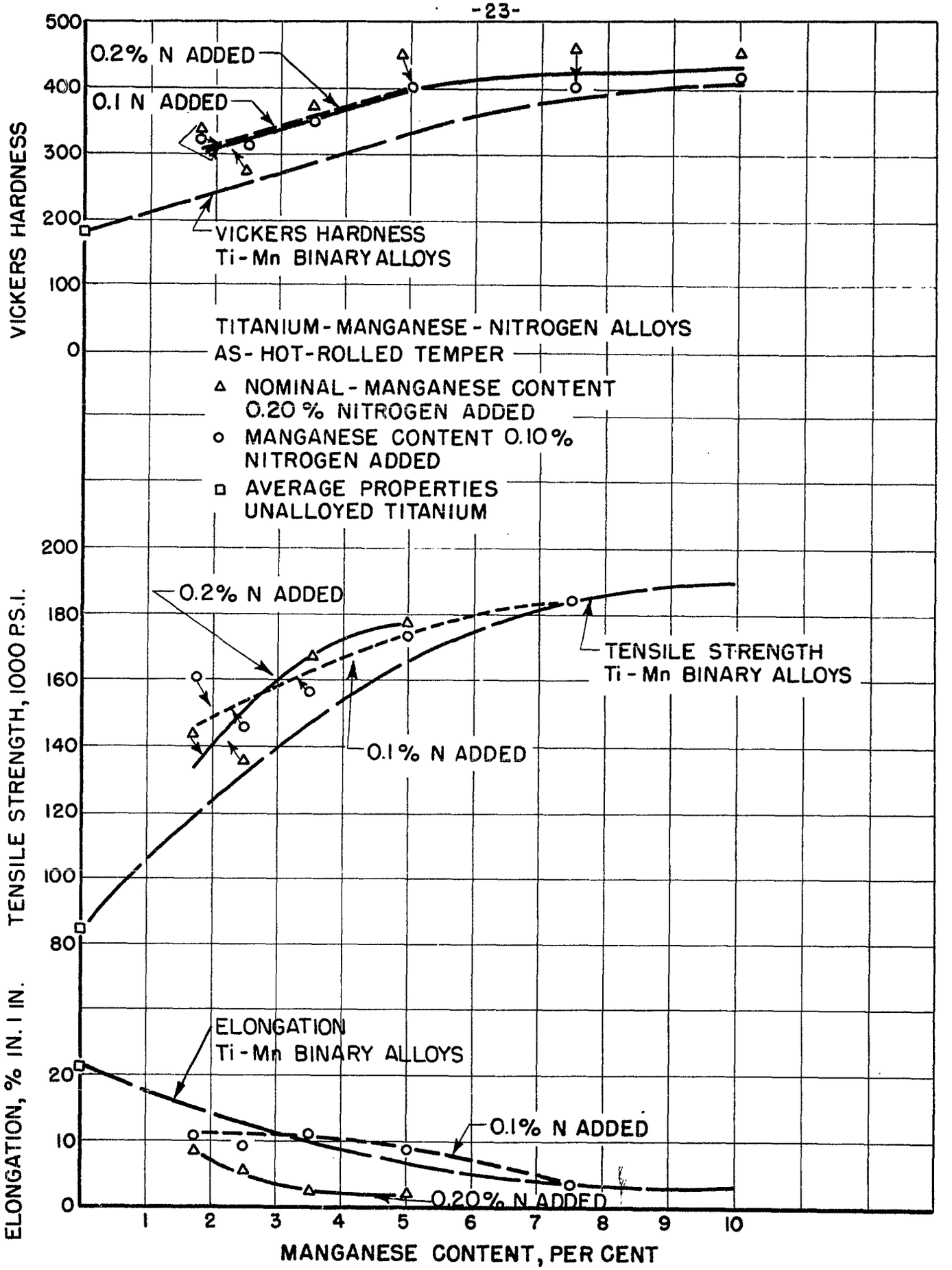

FIGURE 10. EFFECT OF NITROGEN ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MANGANESE ALLOYS - PREPARED FROM PROCESS A METAL

0-13290

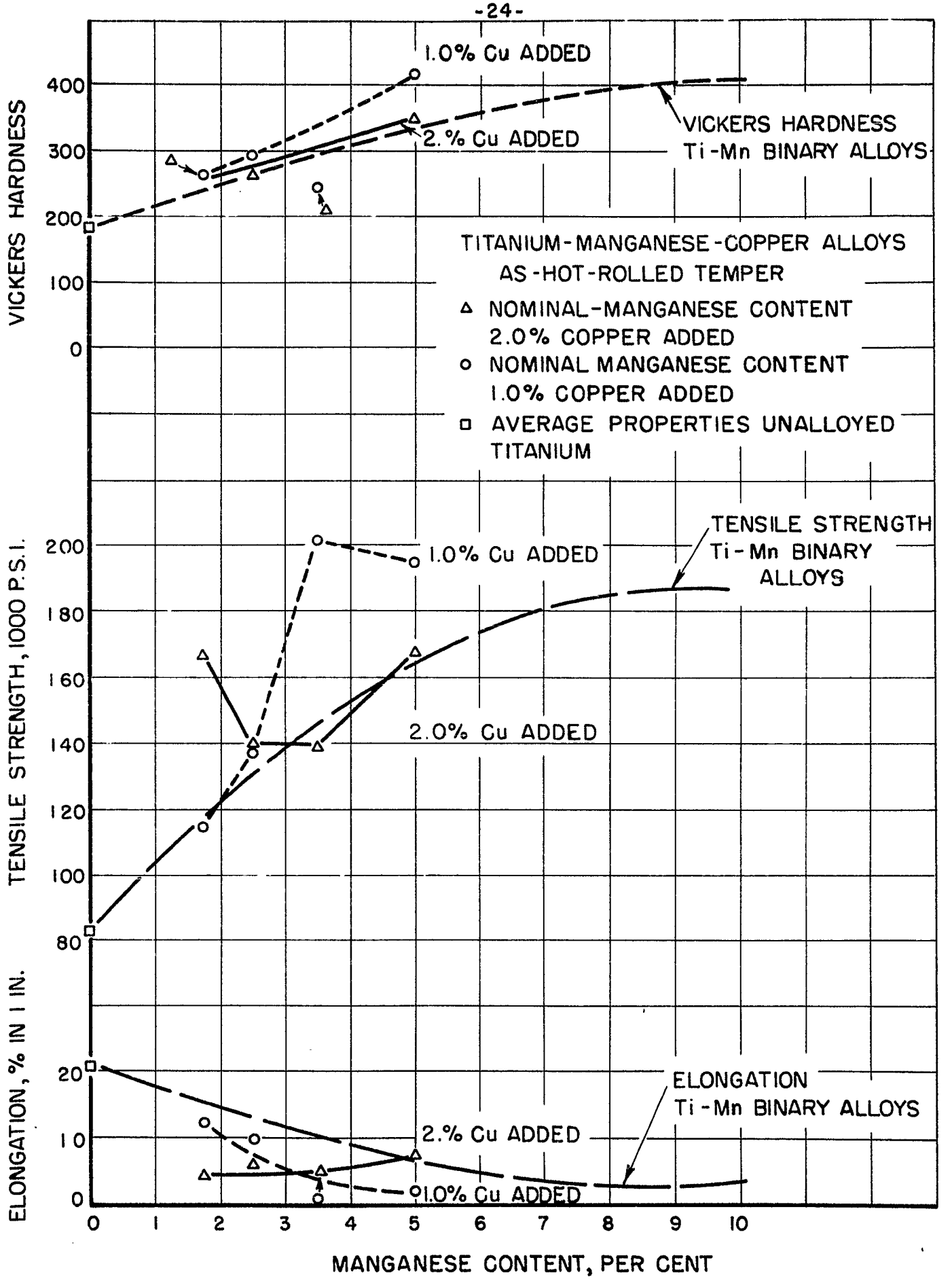

FIGURE 11. EFFECT OF COPPER ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MANGANESE ALLOYS-PREPARED FROM PROCESS A METAL

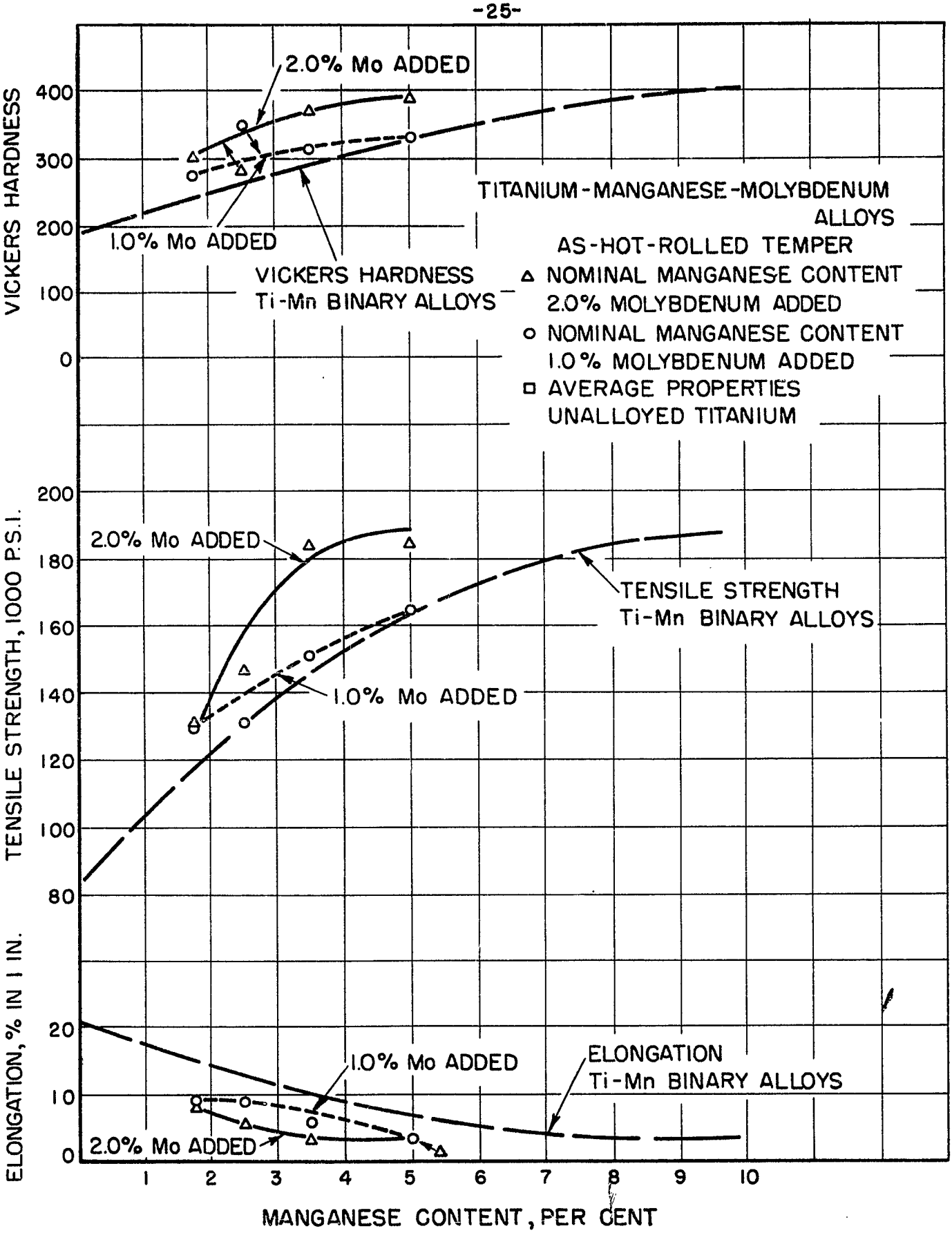

FIGURE 12. EFFECT OF MOLYBDENUM ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MANGANESE ALLOYS-PREPARED FROM PROCESS A METAL

0-13292

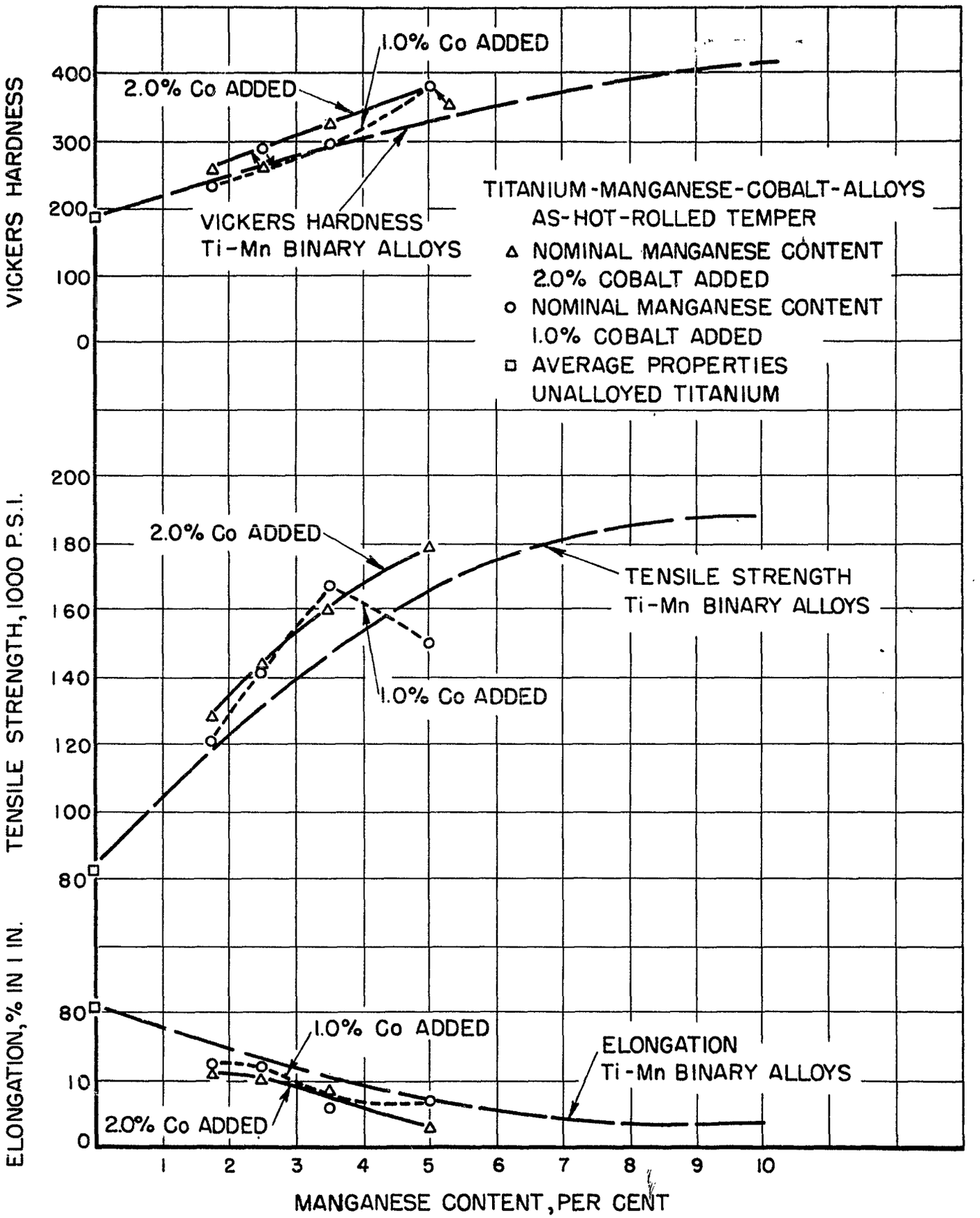

FIGURE 13. EFFECT OF COBALT ON THE PROPERTIES OF AS-HOT-ROLLED TITANIUM-MANGANESE ALLOYS-PREPARED FROM PROCESS A METAL

0-13293

Heat No.	Intended Composition, Per Cent	As-Hot-Rolled Temper: Tensile Strength, p.s.i.	Elongation, Per Cent in 1 Inch	VHN	Minimum Bend Radius
WH160	3.5 Mn, 0.1 N	156,900	11.0	350	3/16
WH162	5.0 Mn, 0.1 N	173,500	8.5	401	<1/4*
WH145	5.0 Mn, 2.0 Cu	166,600	7.5	351	<1/4**
WH154	3.5 Mn, 2.0 Co	160,200	8.5	324	3/16
WH151	3.5 Mn, 1.0 Co	166,700	6.0	297	3/16

* Some ductility.

** Very little ductility.

In general, the other titanium-manganese base ternary alloys showed an increase in tensile strength and hardness above the value for the titanium-manganese base, but in these cases the elongation values were lowered by the additions.

The ternary titanium-manganese base alloys generally increased in hardness when quenched from temperatures of 1450 to 1750°F., as shown in Table 6. However, when some of these alloys were tested after a solution heat treatment at 1600°F., the tensile properties, as shown in Table 5, were generally inferior to those obtained in the as-hot-rolled temper.

The ternary titanium-manganese base alloys in the as-hot-rolled temper were aged for 4 hours at 750°F. and the tensile properties were then determined. As shown by the data in Table 5, the properties were generally inferior after aging to those obtained in the as-hot-rolled condition. In the following cases, increased tensile strengths were obtained by the aging treatment with little or no sacrifice in ductility.

Heat No.	Intended Composition, Per Cent
WH166	7.5 Mn, 0.1 N
WH159	2.5 Mn, 0.2 N
WH151	3.5 Mn, 1.0 Co
WH150	2.5 Mn, 2.0 Co

It should be noted, however, that neither the improvement nor the lack of improvement produced by aging is consistent. Therefore, the response to the aging treatment cannot be readily estimated.

INVESTIGATION OF REFRACTORIES FOR MELTING TITANIUM

(P. J. Maddex and L. W. Eastwood)

The investigation of unusual refractories for melting titanium was continued. Following previously established practice, 10- to 15-gram melts of titanium were made in small crucibles using a standardized melting temperature of 3100°F. The melts were held at this temperature for 1 minute and then cooled in the crucible.

In nearly all previous tests, the melts had been made in a vacuum. In many cases, gas evolution from the melt, probably hydrogen, had forced all or a portion of the melt from the test crucible. To eliminate this difficulty, a melting technique was developed using a purified argon atmosphere. The argon was purified by heating titanium chips to 2000°F. in the melting furnace just prior to melting the titanium. By this technique, hardness values comparable to those obtained with vacuum melts were obtained without having the molten metal forced from the crucible.

The results of the present experimental work on refractories are listed in Table 7. None of the crucible materials, on which tests have been completed, can be considered satisfactory.

When the melt, made in the tungsten carbide crucible, was removed for chemical analysis, it was observed that the crucible adhered to the sides but not to the bottom of the test ingot. It was felt that a difference in porosity between the bottom and the sides of the pressed crucible might account for this.

The density of the bottom of the crucible was 12.6 as compared with 13.1 for the side. Porosity of the bottom was 14.1 per cent as compared with 13.4 per cent for the side. Since there is so little difference between the values of the side and bottom of the crucible, it does not seem reasonable that the results could be accounted for on this basis. Therefore, it seems that a small temperature difference may explain the greater degree of wetting of the sides of the tungsten carbide crucible.

Negotiations have been made with the Norton Company to supply hot-pressed crucibles for these studies. It is anticipated that orders for additional crucibles can be placed in the near future.

ANALYTICAL METHODS FOR TITANIUM-BASE ALLOYS

The Determination of Oxygen in Titanium by the Cl_2 - CCl_4 Method

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

It was emphasized in the last report that the success of the Cl_2 - CCl_4 method for O_2 in titanium depends, first of all, on the

TABLE 7. DATA ON REFRACTORIES TESTED AND RESULTS OBTAINED

Crucible Material	Melting Atmosphere	Wetting of Crucible by Ti**	Chemical Analysis	Vickers Hardness* (10-Kg. Load)	Metallographic Examination	Crucible Attack at Ti-Refractory Interface
Hot-pressed TiC	Vacuum	Yes	-	244	Considerable carbide phase present***	No attack evident
Hot-pressed WC	Vacuum	Yes	0.70% C	315	Ditto	Ditto
Hot-pressed ZrC	Vacuum	Yes	-	283	"	"
Graphite, no argon purification	Argon	Yes	-	380	"	"
Graphite, purified argon	Argon	Yes	0.58%C	252	"	"
TAC lining on graphite crucible	Argon	Yes	Not complete			
WB lining on graphite crucible	Arbon	Yes	Not complete			

* 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.

** Indicated by sharp meniscus on top of ingot.

***The carbide phase appears in a dendritic pattern uniformly distributed throughout the sample.

quantitative decomposition of the sample, since all of the O_2 is to be finally measured as CO.

Reaction temperatures from 400 - 550°C. were found to be too low for complete sample decomposition. (It was anticipated that, if it were necessary to operate above 600°C., it would be impossible to obtain a low blank because of attack on the quartz tube and boat.)

Since other investigators had indicated that the Cl_2 - CCl_4 reaction should proceed quantitatively at 500 - 600°C., it was thought desirable to examine the refractory residues which remained in the boat after reaction. An X-ray diffraction analysis indicated spinels to be present rather than individual oxides. (Particular spinels cannot be positively identified on the evidence of X-ray patterns alone because the structures of many spinels are so similar in both arrangement and size of unit cell.) Since spinels are particularly refractory, it is not surprising that they resist attack at 600°C.

Semiquantitative spectrographic analysis of these same residues indicates the possibility of the following spinels being present:

Spinel	Per Cent
$Ti\ Mg_2\ O_4$	High
$Fe\ Ti\ O_4$	Medium
$Mn\ Ti\ O_4$	Low

A quartz reactor was designed for higher temperature investigation (similar to the Pyres reactor described in the previous report). A series of runs was made at higher temperatures, using both Bureau of Mines powder and DuPont sponge titanium. Results are briefly summarized below:

1. Considerable attack is obtained on the sample boat with both fused silica and "Leco HF Series" boats at temperatures from 700 - 900°C. (in one case, the attack on the boat alone was some ten times the oxide residue obtained from the sample).

2. Decomposition of the sample is sufficiently complete at 900°C. but incomplete at 700°C.

Because of the existence of spinels in the titanium residues and the high temperature and resulting high blank necessary for complete reaction, it does not appear feasible to investigate further this method for O_2.

A detailed description of the apparatus and techniques used in the Cl_2 - CCl_4 method for oxygen analysis will be included in the next bimonthly report.

The Analysis of Titanium for Oxygen by Vacuum-Fusion Methods

(M. W. Mallett, D. G. Thomas, and C. B. Griffith)

Preliminary experiments at Battelle, as well as observations in the literature, indicate that vacuum-fusion analysis of titanium for oxygen apparently yields low and erratic results. Thermodynamic calculations indicated that no extraordinary difficulty would be encountered in the reduction of titanium oxide although some interference might be expected from vaporization of titanium from the melt and its subsequent sputtering action.

Recently, several laboratories[1,2] have reported successful results with the vacuum-fusion analysis of titanium for oxygen. Therefore, a program was undertaken in which three different modifications of the vacuum-fusion method were used with the same apparatus and operator. This comparison study was made in order to evaluate the three methods in regards to one another, and possibly, to indicate interfering elements in the apparatus design.

Simultaneously, a method was developed in an attempt to circumvent the apparent short comings of the vacuum-fusion analysis. The basis of this process was the use of the isotope O^{18} together with the vacuum-fusion method. The procedure consisted of diluting a known amount of O^{18}

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(1) Private communication from Dr. G. Derge, Carnegie Institute of Technology, arranged through the courtesy of Dr. Walter A. Findlay, Remington Arms, Co., Inc.

(2) Private communication from Mr. E. J. Chapin, Metallurgy Division, Naval Research Laboratory, Washington, D. C.

tracer with the unknown amount of oxygen in the titanium. By extracting the gas from the titanium and determining the new ratio of O^{18} to O^{16} with the mass spectrometer, the unknown amount of oxygen can easily be calculated. Theoretically, this method would yield accurate results even though only a fraction of the oxygen content of a titanium sample was extracted by vacuum-fusion.

A Comparison of Oxygen Values Obtained by Three Modifications of the Vacuum-Fusion Method

Preparation of Standards. In a comparative study of modifications of the vacuum-fusion method, titanium-oxygen standard samples were prepared by two methods. In one, measured volumes of gaseous oxygen were reacted with pieces of Foote Mineral Company iodide titanium at 800°C. in a modified Sieverts apparatus. Each specimen was then held at 800-900°C. for two hours, in an attempt to obtain a homogeneous distribution of the oxygen throughout the metal.

Other titanium-oxygen standards were prepared by drilling small holes in 8-gram pieces of Foote Mineral Company iodide titanium, inserting weighed additions of TiO_2 (C.P., Baker Chemical Company), and plugging the holes with iodide titanium. The specimens were then arc melted in a water-cooled copper crucible in an argon atmosphere at 10-cm. absolute pressure, using a tungsten-tipped electrode. The ingot was then turned over and remelted to insure uniform distribution of the oxygen content.

Methods of Analysis. It was decided to analyze the two types of titanium-oxygen standards by three different modifications of the vacuum-fusion method. Following is a description of the procedures used:

Method A. This method was devised at Battelle several years ago for the determination of oxygen in thorium. The procedure consists of dropping the titanium sample into a graphite crucible containing a 1/4- to 1/2-inch-deep layer of graphite chips (1/16- 1/8-inch mesh). The crucible and chips had been previously degassed at 2150 - 2200°C. for several hours. In melting, the titanium is dispersed in thin layers between the chips, thus aiding penetration of the metal by the carbon. The most satisfactory rate of reduction was obtained at 1900°C. One to two hours are required for complete gas extraction. After completing the extraction of the first specimen, a fresh lot of degassed graphite chips is dropped before a second specimen is analyzed.

Method B. This procedure gave apparently satisfactory results for Dr. G. Derge of Carnegie Institute of Technology. It consists of degassing, at 1850°C., a bath of iron (25 grams) previously saturated with carbon to prevent erosion of the reaction crucible. Following the degassing period, the temperature is lowered to 1400°C., and 5 grams of tin is dropped and degassed for about 15 minutes. The sample is then dropped and the temperature progressively raised to 1850°C. About thirty minutes to one hour is required to complete the extraction. Additional tin is dropped between specimens.

Method C. The Naval Research Laboratory has reported[2] that satisfactory analyses were obtained by using a graphite crucible containing a quantity of graphite powder. After degassing this set-up, the temperature was lowered to 1800°C. and the sample and several grams of tin were dropped together. They report that an exothermic reaction occurs in the neighborhood of 1600°C., which causes spatter losses if tin is not used to ameliorate the reaction. Gas collection is continued for about one hour. More tin is added with each successive sample.

Although a variable number of samples that can be analyzed successively in one crucible have been reported, only two samples were run for each furnace set-up.

Analytical Results. Figure 14 is a photograph of the vacuum-fusion apparatus used in analyzing titanium for oxygen. Tables 8, 9, and 10 give the complete vacuum-fusion analysis data. A summary of the oxygen values obtained by the three methods appear in Table 11. These data show that there is no essential difference in the results of the three methods. All tend to yield low and erratic results, but, occasionally, values are obtained which are in reasonable agreement with the known oxygen additions. In general, in a series of these erratic results, the highest value obtained on the same material is likely to be the best value. Some of the highest values have been obtained in the shortest extraction time. Extending the extraction time beyond that required to return the system to the original base pressure is useless. No obvious cause of low values has been observed, except in a few cases where spatter has occurred.

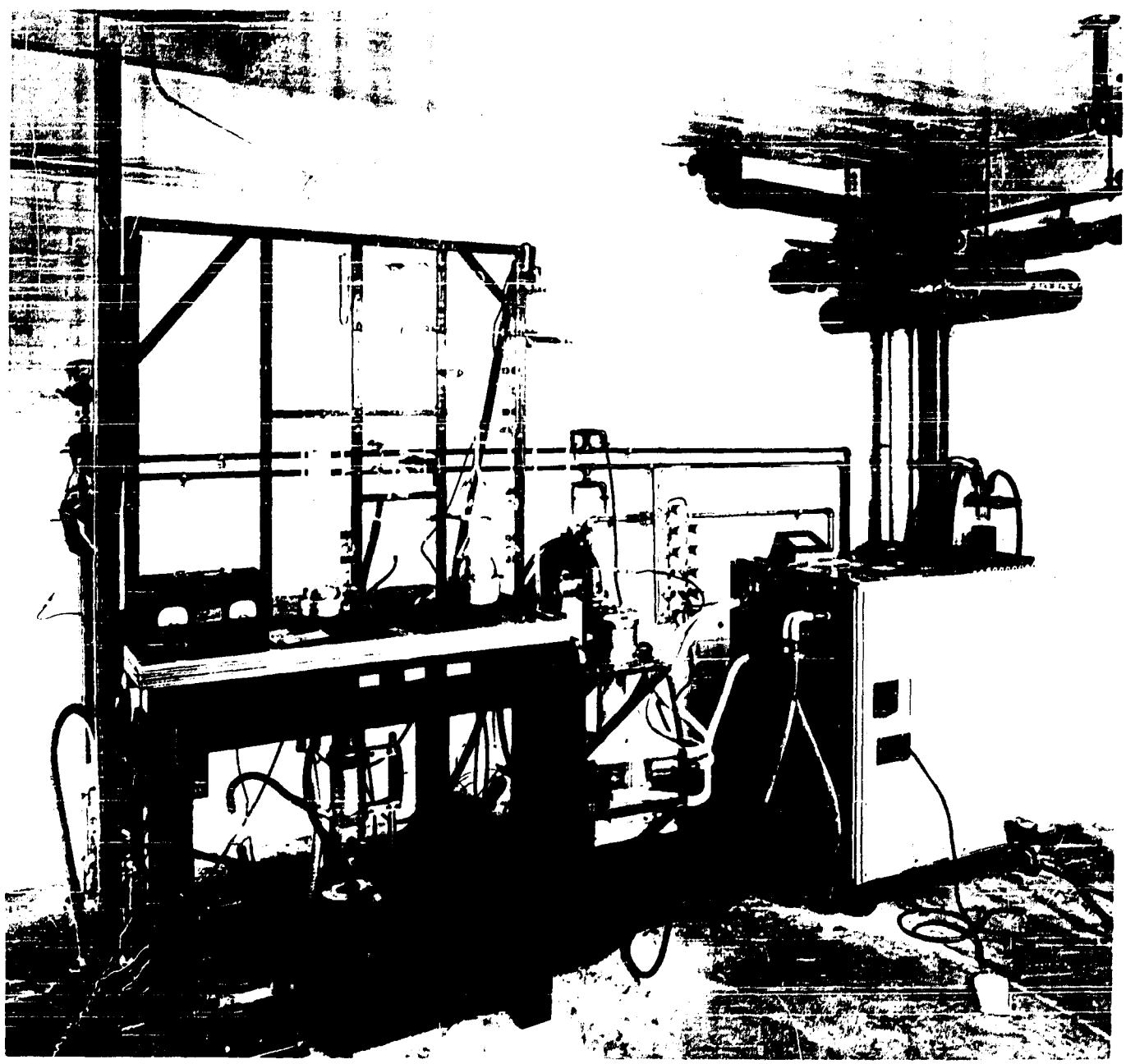

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Figure 14. Vacuum-Fusion Analysis Apparatus

TABLE 8. VACUUM-FUSION ANALYSIS RESULTS OBTAINED BY METHOD A

Sample* Code	Weight (Per Cent) Oxygen Addition	O_2	H_2	N_2	Sample Weight, Grams	Volume of Gas in ml. S.T.P. CO	H_2	N_2	Total	Run Number and Dropping Order	Extraction Time, Min.	Extraction Temperature, °C.	Sample Code
I-0-A	0.0	0.001	0.0036	0.001	8.08	0.08	3.21	0.08	3.36	13 A	20	1900	I-0-A
I-0-B	0.0	0.001	0.0034	0.003	7.54	0.08	2.88	0.16	3.12	15 A	20	1900	I-0-B
IA-0-B	0.0	<0.001	0.0058	-	7.21	Nil	4.64	-	-	7 A	30	1900	IA-0-B
IA-0-C_1	0.0	<0.001	0.0041	-	7.37	Nil	2.79	-	-	16 A	20	1900	IA-0-C_1
IA-2-B	0.02	<0.001	0.0068	<0.001	7.20	Nil	5.44	Nil	5.44	7 B	30	1900	IA-2-B
IA-2-C	0.02	<0.001	0.0039	0.004	6.12	Nil	3.20	0.25	3.45	16 B	20	1900	IA-2-C
IA-1-A_1	0.10	0.0975	0.0092	0.012	3.45	4.72	3.54	0.34	8.60	6 A	80	1900	IA-1-A_1
IA-1-C_2	0.10	0.001	0.0045	0.002	4.745	0.08	2.36	0.08	2.52	17 A	20	1900	IA-1-C_2
IA-5-A_2	0.50	0.460	0.0083	0.020	2.105	13.60	1.94	0.34	15.88	8 B	120	1900	IA-5-A_2
IA-5-B_1	0.50	0.111	0.0058	0.029	1.79	2.79	1.15	0.41	4.35	15 B	70	1900	IA-5-B_1
IA-5-B_3	0.50	0.318	0.0073	0.028	3.63	15.70	2.97	0.82	19.49	17 B	280	1900	IA-5-B_3
IS-2-A	0.021	0.003	0.0045	0.003	10.65	0.34	5.31	0.26	5.91	3 A	30	1800	IS-2-A
IS-2-C	0.019	0.001	0.0056	-	9.57	0.12	5.97	-	-	10 A	130	1900	IS-2-C
IS-1-B	0.106	0.137	0.0037	<0.006	1.73	3.34	0.72	Nil	4.06	8 A	50	1900	IS-1-B
IS-5-B	0.551	0.030	0.0061	<0.02	0.62	0.26	0.42	Nil	0.68	3 B	40	1800	IS-5-B
IS-5-D	0.484	0.007	0.0146	0.080	0.81	0.08	1.32	0.08	1.48	13 B	30	1900	IS-5-D
IS-5-E	0.521	0.016	0.0068	0.021	0.934	0.21	0.70	0.17	1.08	10 B	30	1900	IS-5-E

Note: A 1/4-1/2" layer of 1/16-inch graphite chips was placed in the crucible. A similar quantity of degassed chips was dropped just prior to dropping the second specimen in each series.

* See key at end of Table 4.

TABLE 9. VACUUM-FUSION ANALYSIS RESULTS OBTAINED BY METHOD B

Sample* Code	Weight Per Cent Oxygen Addition	Found Experimentally O_2	H_2	N_2	Sample Weight, Grams	Volume of Gas in ml. S.T.P. CO	H_2	N_2	Total	Run Number and Dropping Order	Extraction Time, Min.	Temp., °C.	Sample Code
IA-0-C_2	0%	0.0137	0.0036	0.0079	6.59	1.21	2.61	0.41	7.32	18-1	85	1850	IA-0-C_2
IA-2-D	0.02%	0.001	0.0036	0.0076	5.4	0.09	2.13	0.33	4.61	19-1	60	1850	IA-2-D
IA-1-C_1	0.1%	0.005	0.0029	0.0104	2.955	0.25	0.90	0.25	3.46	19-2	60	1850	IA-1-C_1
IA-1-C_3	0.1%	0.011	0.0019	0.1611	2.74	0.49	0.63	3.79	7.49	20-1			IA-1-C_3
IA-5-B_2	0.5%	0.088	0.0075	<0.005	1.57	1.93	1.32	nil	3.25	12-2	30		IA-5-B
IA-5-C	0.5%	0.0875	0.0098	0.0205	3.085	3.78	3.35	0.50	9.24	24-2	60	1850	IA-5-C
IS-2-F	0.02%	0.00227	0.0039	<0.001	10.21	0.34	4.38	nil	6.38	23-2	60	1850	IS-2-F
IS-1-E	0.1%	0.012	0.0057	0.004	2.49	0.41	1.56	0.08	2.05	12-1	20	1850	IS-1-E
IS-1-F	0.1%	0.015	0.0042	0.0107	1.96	0.42	0.92	0.17	3.19	24-1	60	1850	IS-1-F
IS-5-G	0.5%	0.117	0.0089	0.041	0.77	1.26	0.76	0.25	4.37	23-1	60	1850	IS-5-6

Note: Approximately 25 grams Fe and 5 grams Sn were present in the crucible when the titanium specimen was dropped. Additional tin was added just prior to dropping the second specimen in a series.

* See key, end of Table 4.

TABLE 10. VACUUM-FUSION ANALYSIS RESULTS OBTAINED BY METHOD C

Sample* Code	Weight (Per Cent)				Sample Weight, Grams	Volume of Gas in ml. S.T.P.				Run Number and Dropping Order	Extraction		
	Oxygen Addition	O_2	H_2	N_2		CO	H_2	N_2	Total		Time, Min.	Temperature, °C.	Sample Code
IA-0-A	0.0	0.005	0.0060	0.003	7.145	0.51	4.80	0.17	5.48	5 A	60	1800	IA-0-A
IA-2-A	0.02	0.024	0.0067	0.007	7.15	2.45	5.32	0.42	8.19	5 B	127	1800-1900	IA-2-A
IA-1-A_2	0.10	0.027	0.0085	0.003	3.76	1.10	3.54	0.09	4.73	9 A	60	1900	IA-1-A_2
IA-1-B	0.10	0.010	0.0118	0.028	3.83	0.51	5.02	0.85	6.38	4 A	40	1800	IA-1-B
IA-5-A_1	0.50	0.055	0.0094	<0.006	1.865	1.44	1.96	Nil	3.40	4 B	40	1800	IA-5-A_1
IA-5-A_3	0.50	0.082	0.0062	0.007	1.47	1.69	1.013	0.09	2.79	9 B	60	1900	IA-5-A_3
IA-5-A_4	0.50	0.445	0.0090	0.0294	1.40	8.74	1.40	0.33	10.47	11 B	60	1900	IA-5-A_4
IS-1-A	0.170	0.009	0.0045	0.005	2.04	0.26	1.02	0.09	1.37	1 A	40	1800	IS-1-A
IS-1-D	0.113	0.073	0.0091	<0.005	1.78	1.81	1.81	Nil	3.62	14 A	40	1900	IS-1-D
IS-5-A	0.589	0.207	0.0070	0.024	0.880	2.55	0.69	0.17	3.41	2 A	120	1800	IS-5-A
IS-5-C	0.496	0.312	0.0096	0.013	0.77	3.37	0.82	0.08	4.27	11 A	30	1900	IS-5-C
IS-5-F	0.505	0.275	0.0014	<0.010	0.810	3.13	1.23	Nil	4.36	14 B	80	1900	IS-5-F

Note: A 1/4-inch layer of -60 mesh graphite powder was present in the crucible during degassing. A similar quantity of 1/16-inch graphite chips was added between samples. This larger particle size was used to facilitate dropping. Five grams of tin was dropped simultaneously with each titanium specimen.

* See key at end of Table 4.

TABLE 11. COMPARISON OF RESULTS BY THREE MODIFICATIONS OF THE VACUUM-FUSION METHOD

Sample** Code	Oxygen Addition	Per Cent Oxygen		
		Method A	Method B	Method C
I-0-A	0	0.001		
I-0-B	0	0.001		
IA-0-A	0			0.005
IA-0-B	0	<0.001		
IA-0-C_1	0	<0.001		
IA-0-C_2	0		0.013	
IA-2-A	0.02			0.024
IA-2-B	0.02	<0.001		
IA-2-C	0.02	<0.001		
IA-2-D	0.02		0.001	
IA-1-A_1	0.10	0.098		
IA-1-A_2	0.10			0.027
IA-1-B	0.10			0.010
IA-1-C_1	0.10		0.006	
IA-1-C_2	0.10	0.001		
IA-1-C_3	0.10		0.012	
IA-5-A_1	0.50			0.055*
IA-5-A_2	0.50	0.460		
IA-5-A_3	0.50			0.082*
IA-5-A_4	0.50			0.445
IA-5-B_1	0.50	0.111		
IA-5-B_2	0.50		0.088	
IA-5-B_3	0.50	0.318		
IA-5-C	0.50		0.088	
IS-2-A	0.021	0.003		
IS-2-C	0.019	0.001		
IS-2-F	0.020		0.002	
IS-1-A	0.170			0.009
IS-1-B	0.106	0.137		
IS-1-D	0.113			0.073
IS-1-E	0.101		0.012	
IS-1-F	0.106		0.015	
IS-5-A	0.589			0.207
IS-5-B	0.551	0.030		
IS-5-C	0.486			0.312
IS-5-D	0.484	0.007		
IS-5-E	0.521	0.016		
IS-5-F	0.505			0.275
IS-5-G	0.504		0.117	

Footnotes on following page.

Footnotes for Table 11:

* Some spatter loss was observed during melt down of these specimens.

** Key to Sample Code. Sample code numbers are of the form IX-N-Yn where:

I indicates that the starting material was iodide-process titanium,

X = blank, indicates there was no further treatment of the sample,

X = A, indicates that the sample was arc melted (those samples containing a known amount of oxygen were made from a Ti-TiO_2 mixture),

X = s, indicates that the oxygen was added as gas in a Sieverts apparatus,

N = 0, indicates that no oxygen addition was made to the sample,

N = 2, indicates a 0.02 wt. % oxygen addition,

N = 1, indicates a 0.10 wt. % oxygen addition,

N = 5, indicates a 0.50 wt. % oxygen addition,

Y = A,B,C, indicates the various samples in a series, and

n = 1,2,3, indicates the various sections of sample Y.

However, two modifications on the vacuum-fusion apparatus are being made to eliminate possible sources of error. In order to increase the pumping speed, a 10-inch-long by 3/4-inch-diameter guide tube will be removed from between the vacuum-fusion crucible and the pump. Also, the furnace tube will be water cooled to keep any titanium that has been evaporated from the vacuum-fusion crucible onto the furnace tube wall below the temperature for reaction with the evolved gases. At the present point in this investigation, the trends are as follows. The arc-melted specimens tend to yield somewhat higher values than Sieverts samples of the same oxygen level. Whether this is due entirely to the analysis method or, in part, to the manner of preparing the oxygen alloy is not known. The higher the extraction temperature, the better; 1900°C. was generally used. The gas-extraction period should be continued at least one hour, and in some cases may require three to four hours. Because of the long extraction period, the system must be degassed for a long time to minimize the volume of blank gases. No more than two specimens should be analyzed successively, and, possibly, further work will show that a single run per furnace set-up is all that can be tolerated.

Vacuum-Fusion - Mass-Spectrograph
Method of Determining Oxygen in Titanium

It was suggested that by use of O^{18} tracer an absolute determination of the amount of oxygen in a sample of metal could be made.

The one major assumption involved is that the oxygen tracer added to the sample distributes itself in the same manner as the unknown amount of oxygen already present in the sample and, accordingly, that the vacuum-fusion extraction can remove the tracer oxygen in the same ratio that it exists in the titanium metal sample.

Apparatus. The O^{18}* was available as H_2O enriched to 1.5% H_2O^{18}**. Before the tracer could be reacted with the titanium, the water had to be broken down. The decomposition of the water and a separation of the hydrogen from oxygen could be affected quite easily by an electrolysis procedure. Therefore, the reaction apparatus was designed to produce the O^{18} by this method, and to enable transfer of the gas from the electrolysis cell to the reaction furnace without opening the system to the air.

Figure 15 is a photograph of this apparatus and Figure 16 a schematic diagram. The electrolysis unit located on the extreme left of the system was made up of a closed storage container, D, to hold the enriched water during evacuation of the system; an electrolysis cell, E, connected to the system by taper-joints, and with its two arms interconnected by a stopcock; a storage tube, F, of 10-mm. I.D. for the hydrogen, and storage tube, G, 7-mm. I.D., for the oxygen. Since the volume of hydrogen given off was twice that of the oxygen, the ratio of the tube diameters had to be as the square root of two is to one, if only one leveling bulb was to be used to control the pressure in the electrolysis system.

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* Natural oxygen contains 0.204% O^{18}.

** Available through AEC from Stuart Oxygen Co., 211 Bay St., San Francisco.

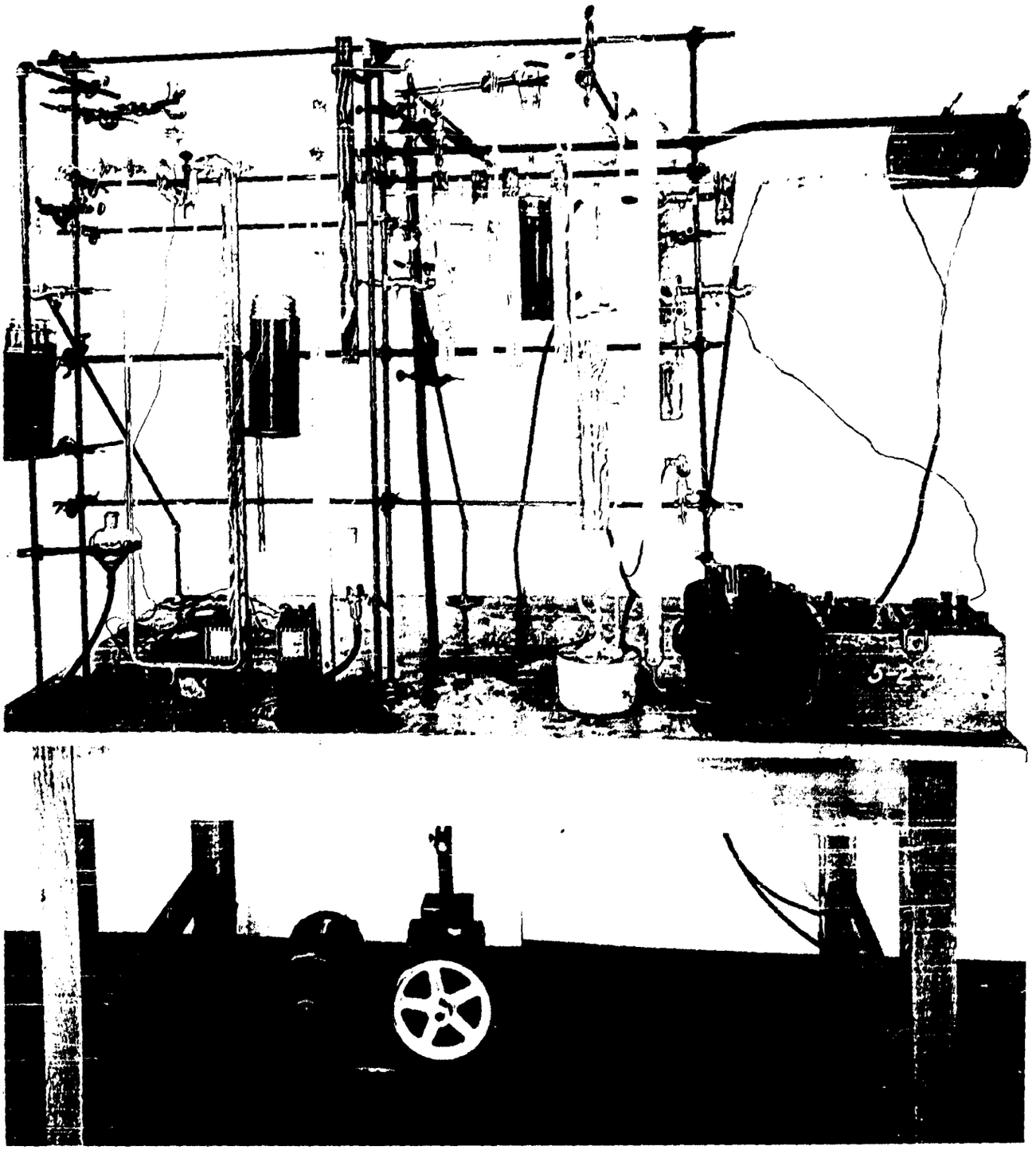

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Figure 15. O^{18} Isotopic Reaction Apparatus

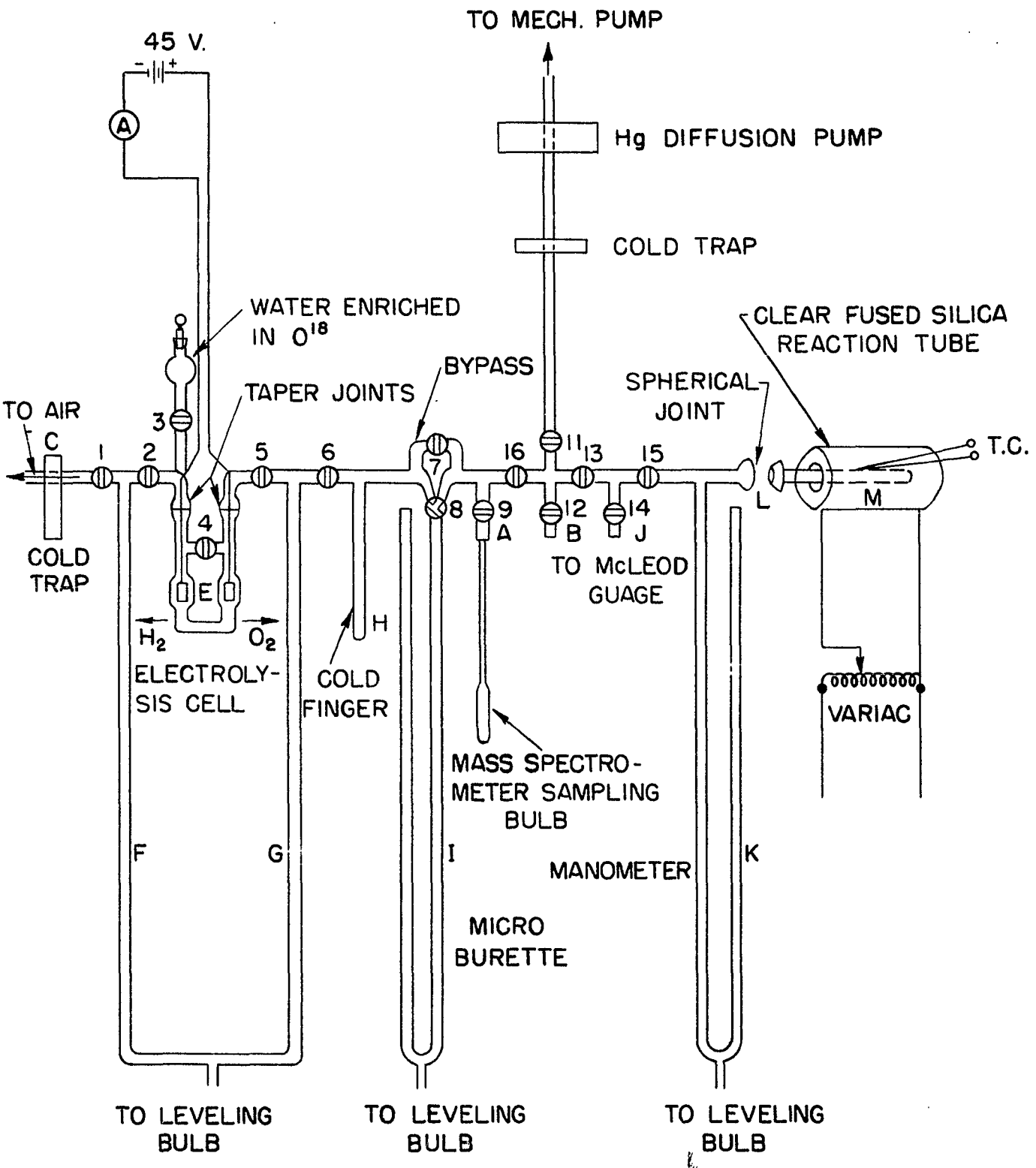

FIGURE 16. O^{18} ISOTOPIC REACTION APPARATUS

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The cold finger, H, dried the electrolyzed oxygen as it was moved over into the storage micro burette, I. The by-pass above I was used in evacuating the system before starting the electrolysis. A mass-spectrometer sampling tube was inserted in the system at Tube A, immediately after the storage burette, in order to obtain a sample of the oxygen gas as electrolyzed. The original ratio of O^{18}/O^{16} in the oxygen gas added to the titanium was determined from this sample. The manometer, K, gives qualitative information on the progress of the absorbtion of the oxygen addition by the titanium, while the McLeod gauge, J, gives a final indication of the completeness of the reaction. The reaction tube, M, of clear fused silica, connects to the system by means of a standard ball joint.

After preliminary runs, it was found that over 95 per cent of the extracted gas was hydrogen, which made it extremely difficult to run a mass-spectrometer analysis. Therefore, a combination gas-concentration and gas-loading apparatus was constructed as shown in Figure 17. The gas sample from the vacuum-fusion apparatus is transferred to the burette, B, under mercury at A. The side-arm contains a palladium tube, C, and a mass-spectrometer sampling tube at D. Suitable arrangements are provided to evacuate the system by means of a mercury-diffusion pump.

Experimental Procedure. To start a run, the system was cleaned and dried, all stopcocks were checked, and a weighed sample of titanium was placed in the reaction tube. The electrolysis cell as well as all the other parts of the system was then evacuated and checked for leaks. When no leaks were observed, Stopcocks 2 and 5 were closed and Stopcock 4 was

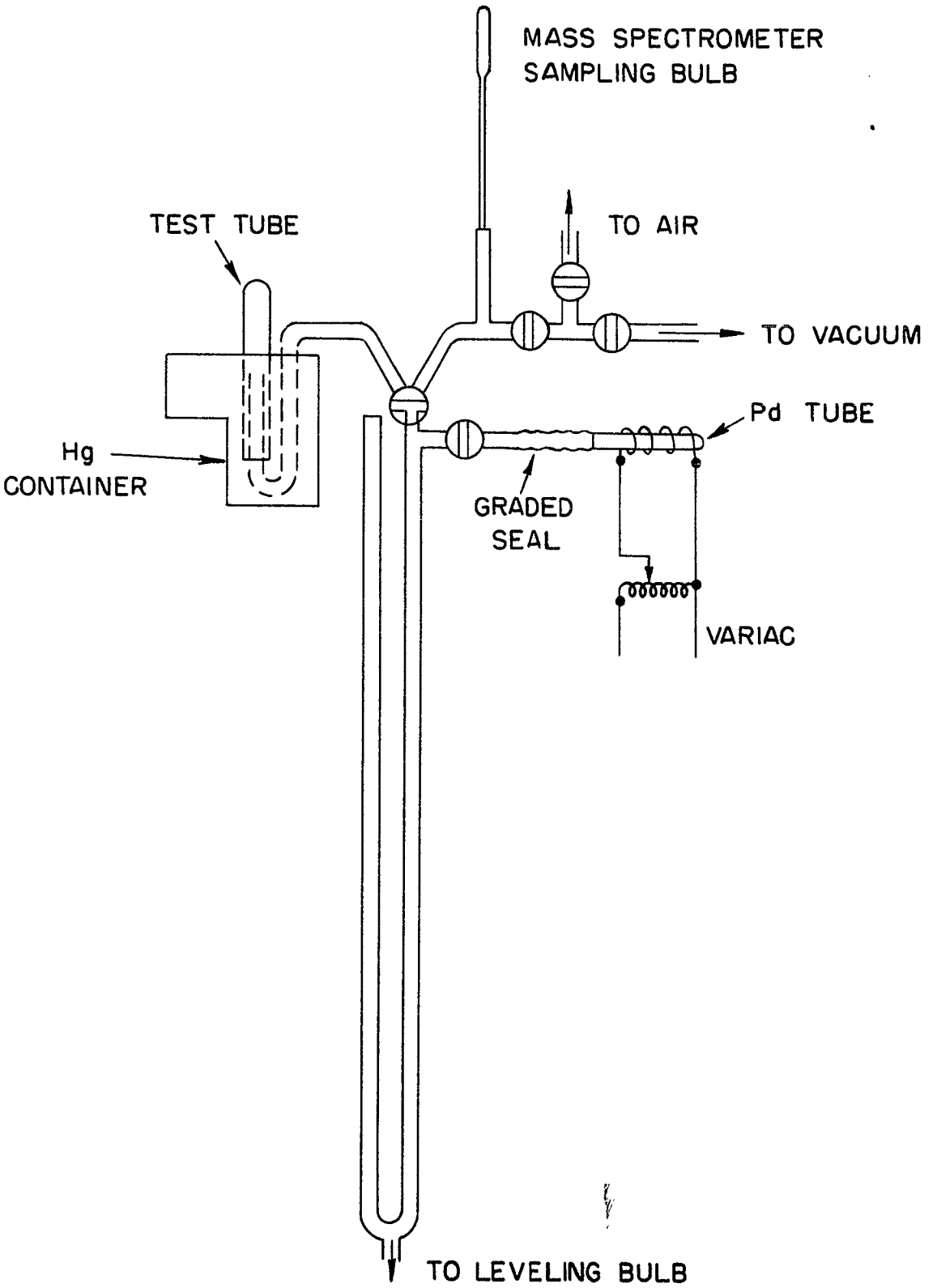

FIGURE 17. APPARATUS FOR CONCENTRATION AND LOADING OF MASS SPECTROMETER SAMPLE

opened. Water was added to the cell, E, from the reservoir, D. Stopcocks 4 and 6 were closed and Stopcocks 2 and 5 were opened. The mercury level in Tubes F and G had been as high as possible but fell when Stopcocks 2 and 5 were opened, because of the vapor pressure of the water. The leveling bulb was used to keep the level of mercury as high as possible.

Because there was a very low pressure on the system when the electrolysis was first started, the bubbles of generated gas were very large. The operation of the electrolysis cell was most difficult at this stage.

When atmospheric pressure was reached in Tubes F and G, Stopcock 1 was opened and the electrolysis was continued until Tube G was filled with oxygen. Stopcock 7 was closed and Stopcocks 6 and 8 were opened after sufficient oxygen had been produced and the gas in Tube G was transferred to the micro-burette, I.

Meanwhile the sample of titanium had been outgassing at 750°C. in the reaction tube, M. Stopcocks 11 and 15 were then closed and a known amount of oxygen was allowed to leave Burette I. Stopcock 8 was closed and Stopcock 15 was opened and the oxygen was allowed to react with the titanium at 800°C. until the system had returned to the base pressure, and for two additional hours. At the end of this time, the titanium sample was cooled quickly and removed from the reaction vessel. It was then ready for extraction in the vacuum-fusion apparatus.

After the oxygen tracer had been added to the titanium and a good vacuum had been reached once more, Stopcock 13 was closed, the microburette was filled, and Stopcock 9 was opened. The electrolyzed oxygen was transferred to the mass spectrometer sampling tube at A at a pressure of 65 to 70 cm. of mercury. The thin glass tube was then sealed off with a flame. This served as a base giving the original ratio, O^{18}/O^{16}, of the enriched oxygen addition.

Upon extraction from the titanium by vacuum-fusion, the gas was transferred to the apparatus (Figure 15) for loading mass-spectrometer sampling bulbs. All the spectrometer bulbs were carefully packed and shipped to the National Bureau of Standards where the ratio of O^{18}/O^{16} was determined through the courtesy of Dr. F. L. Mohler.

Calculations. Results obtained from the National Bureau of Standards indicated the percentage of O^{18} in the total oxygen from which data the ratio of O^{18}/O^{16} was readily calculated. If the ratio of O^{18}/O^{16} is "b", the per cent of O^{18} in the electrolyzed oxygen "a", the volume of enriched oxygen added to the sample x, and the volume of normal oxygen in the sample y, then

$$b = \frac{\text{vol. } O^{18}}{\text{vol. } O^{16}} = \frac{ax + 0.204y}{(100-a)x + 99.796y}, \qquad (1)$$

assuming that normal oxygen contains 0.204 per cent O^{18}. This equation can be rearranged to give:

$$y = \frac{ax - (100-a)\,bx}{99.796b - 0.204}. \qquad (2)$$

Preliminary calculations were made in an attempt to determine the optimum sample weight and amount of oxygen tracer to be added. Figure 18 and Table 12 show the relationship of the weight of titanium sample, the volume of enriched (O^{18} = 7.7 x normal) oxygen added to the titanium, the total volume of enriched and normal oxygen contained in the metal after addition, the ratio b of O^{18} to O^{16} present in the treated sample, and the weight per cent of normal oxygen originally present in the titanium sample.

Because perhaps only 50 to 60% of the oxygen is extracted by vacuum fusion, a small error (3%) may result due to mass difference effects of O^{18} and O^{16}.

It will be noted (see Figure 18) that the addition of 1 cc. of enriched oxygen to a 1-gram sample of titanium is the most versatile combination of conditions. The entire range of analyses from 0.1 to 1.0 weight per cent oxygen is obtainable with reasonable precision. The use of large additions, such as 10 cc. of enriched oxygen, shows no advantage and, in fact, may cause trouble because of the large volumes of evolved gas to be handled.

On the graph, the total oxygen of a sample is recorded as O_2. However, in the vacuum-fusion extraction the oxygen will be evolved as CO, and so the potential total volume of CO is twice the volume of O_2. On the other hand, only about 50% of the potential gas will be recovered,so the volume of O_2 indicated may be considered as the volume of CO likely to be collected during vacuum fusion of the sample.

Examination of the curves shows that greater precision is obtained at the higher values of the ratio b. In the sections where the curves approach the vertical, precision is rather poor.

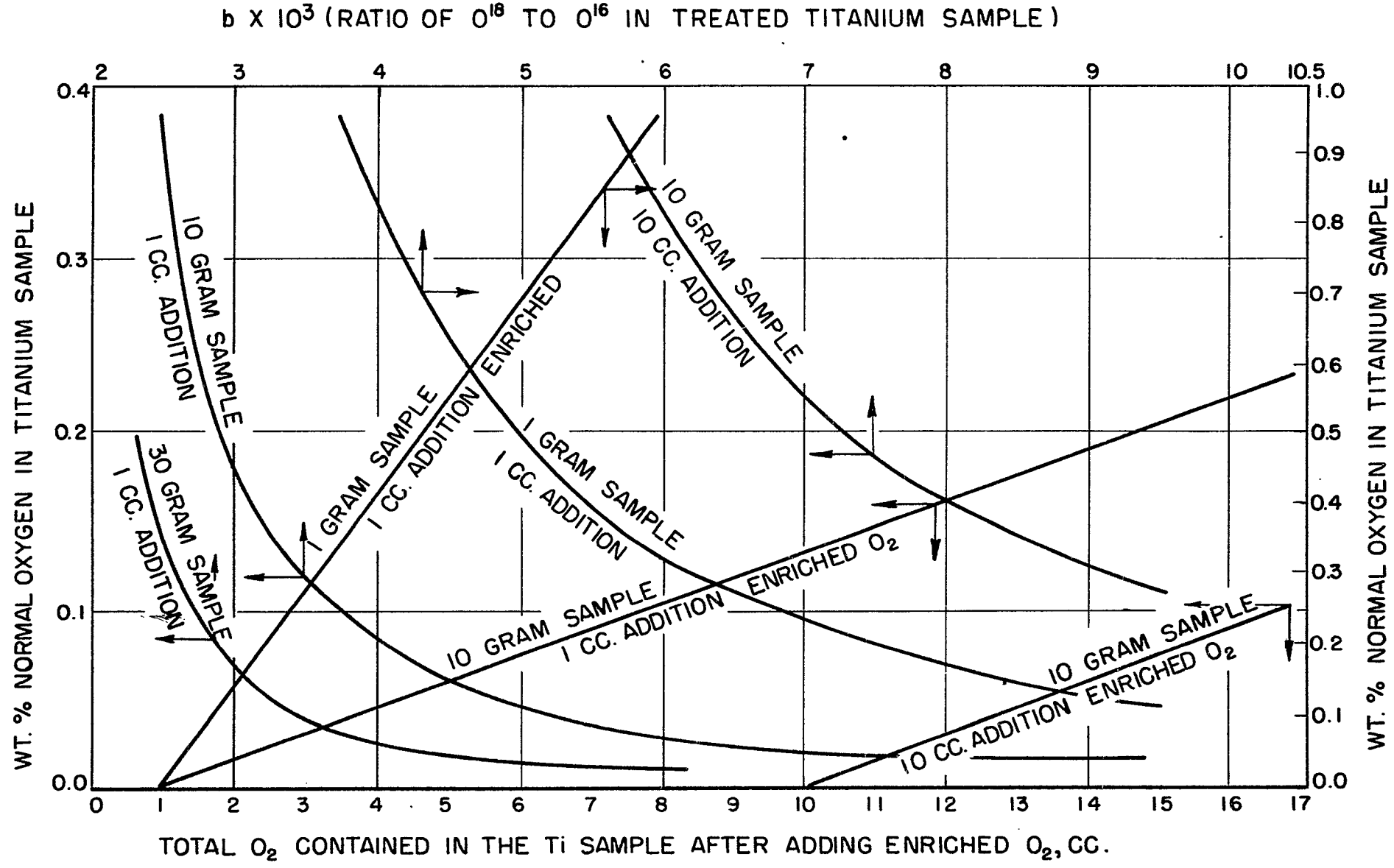

FIGURE 18. DATA FOR USE IN THE ANALYSIS OF TITANIUM FOR OXYGEN BY TREATMENT WITH O^{18}-ENRICHED (7.7 NORMAL) OXYGEN.

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TABLE 12. CALCULATED DATA ON THE ANALYSIS OF TITANIUM FOR OXYGEN BY THE ADDITION OF O^{18} ENRICHED OXYGEN*

Ratio b	Volume O^{18} Added	Volume O^{16} Found, cc.	Normal Oxygen in Titanium, Wt, %
	Calculations Based on a One-Gram Sample		
0.0030	1	12.00	1.710
0.0035	1	7.67	1.095
0.0040	1	5.50	0.785
0.0050	1	3.34	0.476
0.0060	1	2.25	0.321
0.0070	1	1.60	0.228
0.0095	1	0.735	0.105
0.00249	10	5.47	0.0780
0.00250	10	5.44	0.0778
0.00251	10	5.42	0.0773
	Calculations Based on a Ten-Gram Sample		
0.0024	1	31.5	0.45
0.0025	1	25.5	0.357
0.0029	1	13.45	0.192
0.0036	1	7.12	0.105
0.0095	1	0.735	0.0105
0.00350	1	7.67	0.1095
0.00360	1	7.12	0.1015
0.00370	1	6.65	0.0950
0.0025	10	250.0	-
0.0030	10	120.0	1.700
0.00514	10	31.50	0.450
0.0060	10	22.40	0.320
0.00800	10	11.65	0.166
0.00900	10	8.57	0.122
0.00965	10	7.00	0.100
0.010	10	6.25	0.089
	Calculations Based on a Thirty-Gram Sample		
0.0023	1	42.3	0.201
0.0024	1	31.5	0.150
0.0025	1	25.5	0.119
0.0029	1	13.45	0.064
0.0030	1	12.00	0.057
0.0035	1	7.67	0.036
0.0045	1	4.2	0.02
0.0062	1	2.1	0.01

Footnote on following page.

Footnote for Table 12:

* The calculations were made using the approximation

$$b = \frac{1.5x + 0.2y}{100x + 100y}$$

where x is the volume of enriched oxygen added to the sample of titanium and y is the unknown volume of normal oxygen present in the titanium.

Where the oxygen content is known to be low ($<0.1\%$), it is advantageous to increase the sample weight to 10-30 grams.

Data in the Segré chart indicate that the mass spectrograph can measure the ratio, b, to $\pm 0.01\%$. If this is true, the precision of the method is about $\pm 7\%$ at the worst and becomes much better at higher b values.

In the interest of conserving time, the calculations were based on the approximation

$$b = \frac{1.5x + 0.2y}{100x + 100y} \qquad (3)$$

rather than the exact

$$b = \frac{1.57x + 0.204y}{98.43x + 99.796y} \; . \qquad (4)$$

Experimental Results. Oxygen -18 tracer was added to four specimens of titanium. Samples of electrolyzed oxygen were taken after treatment of each of the first and second titanium specimens. Table 13 summarizes the physical properties of the titanium samples before and after the oxygen addition and homogenization. The oxygen was removed from the specimens by the vacuum-fusion method and the gas was transferred, as extracted, to the mass-spectrometer sampling bulbs.

The mass-spectrometer analysis indicated that there was about 1% N_2 and less CO_2 in the gas samples. However, there was 95% or more hydrogen in the sample, so that it was impossible to obtain enough CO in the spectrometer for good sensitivity. Apparently, as much as 50% H_2

TABLE 13. SUMMARY OF PHYSICAL PROPERTIES OF TITANIUM SAMPLES TO WHICH OXYGEN-18 TRACER WAS ADDED

Sample Number	Weight, Grams	Volume O^{18} Tracer Added, cc, S.T.P.	Description of Sample	
			Before Oxygen Addition	After Oxygen Tracer Addition
R-1	5.15	2.30	Iodide titanium; massive, bright, and shiny.	Two dark spots on side next to reaction tube.
R-2	0.80	2.30	Bur. of Mines; -10+20 mesh, 0.1% normal oxygen previously added.	Very little change.
R-3	2.36	2.30	Same as R-2.	Silica tube coated with black, brown, and mirror stains, sample unchanged.
R-4	4.10	2.30	Iodide titanium; massive, bright, and shiny.	Dull grey coating on outside.

would not cut down the precision of the analysis, and it was suggested that the amount of hydrogen be reduced to at least that level in succeeding samples. Accordingly, the gas-concentration and gas-loading apparatus shown in Figure 15 was constructed.

Gas samples from the vacuum-fusion apparatus will be transferred to the storage burette and there the hydrogen diffused out through the palladium tube. In preliminary tests, 5 cc. of pure hydrogen were admitted to the palladium tube and in 7 minutes at 300°C. the system was back to a vacuum.

The tabulated results from the Bureau of Standards are given in Table 14. The results given in the column headed "Total" are the results that are used in the calculations. The average value of the per cent O^{18} of Samples B-1 and B-2 corresponds to the term "a" in Equation 1, x = 2.30 cc., and "b" for each sample is obtained by dividing the figure in the "Total" column by 100 minus the value. For example, Sample R-4 may be calculated as follows:

$$b = \frac{0.568}{100-0.568} = \frac{(0.0151)(2.3) + (0.00219y)}{(0.99432)(2.3) + 0.99780y} \qquad (5a)$$

$$0.00573 = \frac{(0.0347) + (0.00219y)}{(2.29) + (0.99780y)} \qquad (5b)$$

$$y = \frac{0.0216}{0.00351} = 6.15 \text{ cc.} \qquad (5c)$$

The results of these calculations are given in Table 15. It can be seen that the results are high by a factor of about 20. Whether the hydrogen alone was responsible for this, or whether there was a leak in the system due to faulty technique in the preliminary runs, has not been determined.

TABLE 14. MASS-SPECTROMETER RESULTS FROM NATIONAL BUREAU OF STANDARDS

Sample Number	Volume, Per Cent O^{18}*		Atom Per Cent O^{18} Excess over Standard
	Total	Standard(a)	
B-1	1.51_7	0.21_9	$1.29_8 \pm 0.005$(b)
B-2	1.50_8	0.21_9	$1.28_9 \pm 0.004$
R-1	Sample practically all hydrogen		
R-2	0.38_1	0.21_9	$0.16_2 \pm 0.003$
R-3	0.24_1	0.21_9	$0.02_2 \pm 0.002$
R-4(c)	0.56_8	0.21_9	$0.34_9 \pm 0.004$

* Based on total oxygen content of gas sample.

Note: (a) Oxygen from air.
(b) Average deviation from the mean. Mean values are based on at least ten repeated measurements.
(c) High per cent of hydrogen in sample would alter calculations.

TABLE 15. PRELIMINARY RESULTS OF ANALYSIS OF TITANIUM SAMPLE FOR OXYGEN BY THE O^{18} ISOTOPIC METHOD

Sample Number	Titanium Weight, Grams	Description of Sample	Volume Normal O_2 Originally in Sample (Mass Spectrometer), cc, S.T.P.	Weight % O_2 in Sample	Duration of Homogenizing Treatment
R-1	5.15	Iodide titanium	Too much hydrogen	-	750°C. 13 hours
R-2	0.80	Bureau of Mines, 0.1% normal oxygen added	16.0	2.85%	800°C. 2 hours
R-3	2.36	Same as R-2	146.0	-	750°C. 2 hours
R-4	4.10	Iodide titanium	6.15	0.214%	750°C. 2-1/2 hours

The gas-concentration and gas-loading apparatus having been completed, samples can now be prepared in which there is little or no hydrogen and so more favorable results are to be expected in future experiments.

FUTURE WORK

The work on the preparation and evaluation of alloys of titanium will be continued. At present, binary alloys of titanium with zirconium, columbium, tantalum, beryllium, and silver, and ternary titanium-manganese-carbon, titanium-manganese-vanadium, and titanium-molybdenum-tungsten alloys are being investigated.

The more promising compositions which have been made to date will shortly be again investigated in order to study further these compositions and select those alloys warranting a more extensive study as outlined in the proposal.

The study of refractories for melting titanium will be continued.

With reference to the Cl_2 - CCl_4 method for determining oxygen, no further tests are contemplated. A detailed description of the apparatus and techniques, however, will be included in the next bi-monthly report.

At the present time, arc-melted samples of iodide titanium with known amounts of oxygen added as TiO_2 are being prepared. These samples will be forwarded to Dr. G. Derge for vacuum-fusion analysis in his laboratory.

Upon the completion of certain modifications of the vacuum-fusion apparatus, spot checks will be run on Methods A, B, and C, in hopes that the yield of extracted gas will be improved.

The gas-concentrating and loading apparatus has been completed and tested. The gas extracted by vacuum-fusion analysis from samples processed in the O^{18} apparatus will contain large amounts of hydrogen. This hydrogen will be removed from future samples in the gas-concentration and loading apparatus before sending to the Bureau of Standards. However, all work on the O^{18} analysis method is being held in abeyance until the results of Dr. Derge's analysis of the Ti-TiO_2 samples, prepared at Battelle, are obtained.

The data on which this report is based are listed in the following BMI Notebooks:

No. 3911, pp. 66-78

No. 3912, pp. 5-11

No. 4519, pp. 39-98

No. 4728, pp. 1-5

No. 4112, pp. 90-91

No. 4736, pp. 2-10
pp. 35-43

No. 4461, pp. 18-22
pp. 32-34
pp. 39-60
pp. 63-84
pp. 90-100

No. 4505, pp. 2-61

No. 733

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September 14, 1949

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Attn: Ms. Kelly Akers (DTIC-R)
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Dear Ms. Akers

This concerns the following Technical Report:

Technical Report number: ADB816506
Technical Report Title: Research and Development on Titanium Alloys
Technical Report Date: 31 Aug 49
Previous classification/distribution code: UNCLAS

Subsequent to WPAFB FOIA Control Number 2010-01928-F, the above record has been cleared for public release.

The review was performed by the following Air Force organization: AFRL/RX and 88 ABW/IPI.

Therefore, the above record is now fully releasable to the public. Please let my point of contact know when the record is available to the public. Email: darrin.booher@wpafb.af.mil If you have any questions, my point of contact is Darrin Booher, phone DSN 787-2719.

Sincerely,

KAREN COOK
Freedom of Information Act Manager
Base Information Management Section
Knowledge Operations

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