WADC TECHNICAL REPORT 54-205
PART II

DEVELOPMENT OF IMPROVED TITANIUM-BASE ALLOYS

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FOREWORD

This report was prepared at Battelle Memorial Institute under USAF Contract No. AF 33(616)-384. The contract was initiated under Project No. 7351, "Metallic Materials", Task No. 73510, "Titanium Metal and Alloys", formerly RDO No. 615-11, "Titanium Metal and Alloys" and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with First Lieutenant E.F. Erbin acting as project engineer.

The research was conducted at Battelle by the persons whose names and activities on the project are shown below:

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This report covers period of work from December 1953 to December 1954.

ABSTRACT

The heat-treated properties of 24 alpha-beta titanium alloys have been evaluated. Manganese, molybdenum, and vanadium were the principal alloying additions. The strength-ductility relationships of these alloys increased with increasing alloy content up to about 7 per cent alloy addition. The Ti-3Mn-complex alloy had room-temperature properties slightly better than those of any of the other alloys tested. However, a Ti-5Mn-2V alloy exhibited excellent elevated-temperature stability at 650 F. A binary Ti-8V alloy was very interesting in that it could be quenched from the beta-phase region and aged to give very good properties. Most alloys are embrittled as a result of being heated into the beta-phase region.

An evaluation of elevated-temperature properties of the Ti-3Mn-complex alloy indicated that the short-time tensile and 100-hour stress-rupture strengths were improved by heat treating initially to a tensile strength of 180,000 psi, instead of annealing to a level of 135,000 psi. However, there was no difference in the 1000-hour stress-rupture strengths for specimens initially heat treated to 135,000- or 180,000-psi strengths.

It was found that pickling to remove the air-contaminated surface of alloy sheet added large amounts of hydrogen. A process was worked out for descaling and pickling titanium-alloy sheet without appreciable hydrogen pickup. Using this process, the Ti-3Mn-complex alloy sheet has been heat treated to 180,000-psi tensile strength with over 10 per cent elongation in 1 inch.

Increasing the hydrogen content of the Ti-3Mn-complex alloy to 260 ppm decreased its tensile ductility and caused premature stress-rupture failures of notched specimens at room temperature. The hydrogen level that causes embrittlement in this alloy is a function of the heat-treated strength of the alloy. As the strength is increased through heat treatment, the hydrogen content required to cause embrittlement is decreased.

PUBLICATION REVIEW

This report has been reviewed and is approved for publication.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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H. A. Robinson, W. M. Parris, and P. D. Frost

INTRODUCTION

This summary report on Contract No AF 33(616)-384 covers research done during the period from December 8, 1953, to December 31, 1954. This research was a continuation of that carried out under Contracts Nos. W 33-038 ac-21229, AF 33(038)-3736, and AF 33(616)-384. The results of the work on these contracts are contained in the following six reports:

Summary Report, Part III, July 30, 1949
AF Technical Report 6218, Part II, June 30, 1950
AF Technical Report 6623, June 18, 1951
WADC Technical Report 52-249, June 18, 1952
WADC Technical Report, 52-334, December 31, 1952
WADC Technical Report 54-205, August 1954

The present extension of this contract provided for the development of titanium-base alloys with improved strength-ductility relationships at room and elevated temperatures or heat treatability, or a combination of these properties.

SUMMARY

The objectives of the research during the past year were as follows:

(1) To investigate the properties of new alloys having compositions different from that of the Ti-3Mn-complex alloy

(2) To investigate fully the effects of hydrogen on the tensile properties, especially ductility, of the Ti-3Mn-complex alloy

(3) To evaluate in further detail the properties of the Ti-3Mn-complex alloy, particularly with respect to elevated-temperature strength and stability.

The results obtained on these three phases of the research are summarized in the following pages.

New Alloys

In research conducted in previous years for the Air Force, the Ti-3Mn-complex alloy was found to have a better combination of tensile properties than other experimental alloys. In the early work, successful heat-treating procedures had not been developed and, therefore, most evaluation tests were made on alloys in the hot-rolled condition. Later, a few of the most promising alloys were compared in the heat-treated condition and the Ti-3Mn-complex was still superior, particularly with respect to stability at elevated temperatures. Since the other alloys tested had substantial quantities of active eutectoid formers, iron or chromium, it was thought that these elements might not be beneficial even in the relatively small amounts present in the Ti-3Mn-complex. Therefore, most new alloy compositions studied this year were based on manganese, molybdenum, and vanadium. With the exception of several binary titanium-vanadium alloys, the total alloy contents ranged from 2 to 10 per cent. Several of the new compositions varied only slightly from that of the Ti-3Mn-complex alloy.

The new alloys were evaluated by tensile tests conducted on heat-treated specimens taken from hot-rolled bar stock and sheet. All of the alloy compositions, with the exception of binary Ti-20V and Ti-35V alloys, developed good strength and ductility after heat treatment. Strengths as high as 200,000 psi, with elongations ranging from 6 to 10 per cent in 1 inch, were produced in several compositions. Generally, however, the ductility at a given strength level was not so good as that obtained in the Ti-3Mn-complex alloy. This was particularly true of compositions having less than 6 per cent total alloy content.

With the exception of a Ti-8V and a Ti-16V alloy, all of the alloys tested were subject to beta embrittlement. That is, they showed decreased ductility when fabricated or heat treated in the beta-phase region. The two binary titanium-vanadium alloys exhibited unusual behavior in that they attained essentially the same properties when fabricated in either the beta- or the alpha-beta-phase regions. The Ti-8V alloy was particularly outstanding in that it developed very good ductility at strengths up to 180,000 psi after quenching from the beta-phase region and aging. This or similar alloys might make excellent forging materials.

The principal cause of beta embrittlement seems to be grain-boundary alpha. Several heat-treatment cycles were tried on various alloys to eliminate the grain-boundary alpha, but none were successful. However, the shape and size of the alpha within the grain could be altered to some extent by heat treatment. Nearly equiaxed alpha was formed by quenching from the beta-phase region and then re-solution treating in the alpha-beta-phase region. Very large, elongated alpha grains were formed by furnace cooling from the beta- to the alpha-beta-phase region. Beta grain size, on the other hand, was not altered much by the heat treatments, but seemed to be characteristic of the alloy composition. A Ti-5Mn-2.5Cr-1Th alloy had the smallest grain size, whereas a Ti-5Mn-2.5Cr alloy had the largest grain size of the alloys investigated. These results indicate that thorium may be useful as a beta-grain-refining element.

Stability tests at 650 F indicated that several of the alloys were equal to or better than the Ti-3Mn-complex alloy in this respect. In particular, a Ti-5Mn-2V composition exhibited excellent stability characteristics. This alloy, heat treated to 200,000-psi strength, maintained this strength with no loss of ductility after 1000 hours of unstressed exposure at 650 F. Under similar conditions, the Ti-3Mn-complex alloy lost some strength and ductility.

Early in the current work, it was found that pickling of sheet to descale and remove contaminated surface layers introduced large amounts of hydrogen into alpha-beta alloys. Consequently, a large part of the alloy development was concerned with developing methods to descale and pickle sheet without adversely affecting its properties through hydrogen pickup. Mechanical descaling by vapor blasting or descaling chemically in a molten "Virgo" salt bath was found to be satisfactory. Sodium hydride descaling introduced considerable quantities of hydrogen. Pickling in various nitric acid-hydrofluoric acid-water solutions removed contaminated metal without appreciably affecting hydrogen content, provided the HNO_3:HF ratio was at least 7:1. During this work, it was confirmed further that very thin heat-treatment scale can affect the ductility of sheet material adversely.

After satisfactory processing procedures were established, several of the new alloy compositions were tested as 0.065-inch-thick sheet after various heat treatments. Again, the Ti-3Mn-complex alloy had slightly better combinations of strength and ductility than any of the new compositions. The results indicated, however, that most of the compositions could be heat treated to about 180,000 psi, with elongations of 8 to 10 per cent in 1 inch. All of the alloys could be heat treated to give bend radii of about 1 T or less.

Effects of Hydrogen on the Ti-3Mn-Complex Alloy

Early this year, bar stock from the same heat of the Ti-3Mn-complex was shown to have widely varying properties in tests conducted at Battelle, the WADC Materials Laboratory, and the laboratory of a titanium producer. It was found that the bars tested had a high hydrogen content and that the properties varied because different strain rates had been used. This discovery was responsible, in part, for the initiation of an extensive evaluation of the effects of hydrogen in titanium alloys, conducted by the Materials Laboratory. The research at Battelle supplements this work.

The object of the Battelle program was to correlate the effects of hydrogen content, fabrication temperature, heat treatment, and strain rate on the properties of the Ti-3Mn-complex alloy. Specimens swaged in the alpha-beta region, fully annealed, then tested at a low strain rate (0.005 inch per minute) decreased in tensile ductility as the hydrogen content was increased from 40 to 690 ppm. Reduction-of-area values for the low and high hydrogen levels were 67 and 17 per cent, respectively. During these tests, it was also observed that hydrogen appreciably lowered the beta-transus temperature of the alloy.

Material for the major part of the investigation was tensile tested under various combinations of the following conditions:

(1) Fabricated at temperatures in either the alpha-beta or the beta-phase regions

(2) Heat treated (solution treated and overaged) to strength levels of 120,000 and 180,000 psi; also tested in the solution-treated condition (150,000 psi)

(3) Hydrogenated or vacuum annealed to levels of 30, 90, 140, and 260 parts per million (ppm) of hydrogen

(4) Tested at strain rates of 0.002 and 0.005 inch per inch per minute and 0.10 inch per minute.*

The results indicated that the effects of hydrogen were dependent upon both heat treatment and strain rate. The specimens heat treated to a high strength level showed a serious reduction in ductility with 140 ppm of hydrogen, whereas the ductility of the lower strength specimens was not appreciably affected until a level of 260 ppm was reached.

The ductility of the alloy at the higher hydrogen levels also varied with strain rate, particularly at the lower strength levels. Generally, the ductility increased with increasing strain rate.

Notched stress-rupture tests at room temperature confirmed the effects of heat treatment and strain rate on the tolerance of the alloy for hydrogen. Embrittlement, as indicated by short times to rupture, occurred at the high strength level with as little as 90 ppm, whereas the lower strength level showed no embrittlement at 140 ppm.

Apparently, hydrogen, per se, is not a major factor in beta embrittlement. Material rolled in the beta-phase region exhibited about the same degree of embrittlement with increasing hydrogen as the material rolled in the alpha-beta phase region.

* The two lower strain rates were established by a strain pacer; the higher strain rate represents the platen speed.

Elevated-Temperature Properties of the Ti-3Mn-Complex Alloy

Elevated-temperature tensile strengths of the Ti-3Mn-complex alloy heat treated to the 180,000-psi strength level were higher up to about 800 F than those of the C-130AM alloy in the annealed condition. Above 800 F, C-130AM had higher strengths. At the 180,000-psi strength level, the Ti-3Mn-complex alloy had 100-hour stress-rupture strengths of 47,000 psi at 800 F and about 116,000 psi at 650 F. At 800 F, the 100-hour stress-rupture strength was about 55 per cent of the yield strength at this temperature. However, at 650 F, the 100-hour stress-rupture strength was the same as the short-time tensile strength. The latter behavior indicates that some strengthening reaction, possibly strain aging, occurs during testing at 650 F. The same alloy, overaged to a strength of 135,000 psi, had considerably lower tensile and 100-hour stress-rupture strength at 650 F.

ALLOY SELECTION, MELTING, AND FABRICATION

As stated in the "Summary", earlier research had shown some evidence that alloys containing iron or chromium were somewhat less stable than the Ti-3Mn-complex alloy when exposed to elevated temperatures. Loss of ductility during such exposure could be due to eutectoid decomposition of the retained-beta phase. For this reason, many of the alloys prepared this year contained only manganese, molybdenum, vanadium, either singly or in combination. Manganese is a very sluggish eutectoid former, and the other two elements are of the terminal solid-solution type that form no compound. The few alloys that did contain iron or chromium were variations of the Ti-3Mn-complex alloy. Iron and chromium were included in these alloys to evaluate the role of these elements in establishing the properties of the complex alloy. The nominal compositions of the new alloys are given in Table 1, along with the experimentally determined beta-transus temperatures and the rolling temperatures employed for each alloy.

All alloys were double arc melted in a water-cooled copper crucible using a tungsten electrode. The procedures used, with one exception, are described in WADC Technical Report 52-249.* The exception is in the processing of the single-melted material into remelt stock. The single-melted ingot was rolled to sheet and grit blasted, instead of being pickled to remove the hot-rolled scale. Pickling between melts was shown (WADC Technical Report 54-205, pp D-1 through D-4) to add hydrogen to the alloys. High-quality sponge, having a hardness of 110 BHN, was used for all alloys. All of the alloys, except three, forged satisfactorily at 1700-1750 F. The 20 and 35 per cent vanadium alloys were difficult to forge at 1850 F, and the 49 per cent vanadium alloy was hot-short at this temperature. Alloy

*Summary Report to Wright Air Development Center, "Development of Titanium-Base Alloys", June 18, 1952 Contract No. AF 33(038)-3736.

TABLE 1. APPROXIMATE BETA-TRANSUS AND ROLLING TEMPERATURES FOR EXPERIMENTAL ALLOYS

Alloy	Nominal Composition, weight per cent						Beta-Transus Temperature, F	Rolling Temperature, F	
	Mn	Mo	V	Fe	Cr	Other		$\alpha+\beta$	β
WU50A		1	1				>1600	1425	
WU51A		1	3.5				>1575	1450	
WU59A		1	5.0				>1550	1450	
WU56A		3.5	1				>1575	1450	
WU55A		3.5	3.5				>1500	1450	
WU54A		3.5	5.0				>1500	1450	
WU58A		5.0	1				>1550	1450	
WU52A		5.0	3.5				>1500	1450	
WU53A		5.0	5.0				>1475	1450	
WV12			8				>1550	1450	1600
WV13			16				>1400	1375	1600
WV14			20				--	--	1500
WV15			35				--	--	1500
WV16			49				Broke up during forging		
WV1	3	1	1	1	1		1550	1450	1650
WV2	3	1	1				1600	1500	1700
WV3	3			1	1		1550	1450	1650
WV4	2	1	1	1	1		1575	1450	1700
WV5	1	1	1	1	1		1600	1500	1700
WV6	2	1	1				1625	1500	1700
WV7	2			1	1		1575	1450	1700
WW20	3	1	1	1	1	0.1Th	--	1425	1625
WW21	3	1	1	1	1	0.25Th	--	1425	1625
WW8	5	2					1500	1400	1650
WW9	2	5					1550	1450	1650
WW10	5		2				1500	1400	1650
WW11	2		5				1550	1450	1650

material to be rolled to 1/2-inch-diameter bar stock was forged to about 3/4-inch-square bars. Material to be rolled to sheet was forged to about 1/2-inch-thick-sheet bars.

An approximate beta-transus determination was made for all alloys before final rolling. This was necessary because, in some cases, in the past, estimates of the transus from phase diagrams were in error and improper rolling temperatures were used. Small 0.10-inch-thick sections of the forged bars were heated for 1/2 hour at 25 F intervals near the estimated transus and quenched in cold water. A metallographic examination indicated that the alloys were all beta at the temperatures given in Table 1. The beta transus for the molybdenum-vanadium and vanadium alloys was above the highest solution temperature used. At the temperatures indicated in the table for these alloys, about 5 to 10 per cent of alpha remained in the microstructure. However, these determinations were accurate enough to indicate a temperature for rolling that would be well below the beta transus. Table 1 includes the rolling temperatures used for finish rolling both bar stock and sheet in either the alpha-beta- or the beta-phase region.

The 3/4-inch-square forged bars from each heat were rolled to 1/2-inch-diameter bar stock at the temperatures shown. A number of rolling passes were required, and the bars were reheated to the rolling temperature between each pass.

The 1/2-inch-thick-sheet bars were rolled to 0.065-inch sheet at the alpha-beta temperatures shown in Table 1. Reduction per pass was about 20 per cent, and the material was reheated between passes. In order to minimize anisotropy, the sheet bar was rolled in one direction to a thickness of about 1/4 inch and then turned 90 degrees to the original direction and rolled down to the finished size.

EVALUATION OF NEW ALLOYS AS 1/2-INCH-DIAMETER BAR STOCK

Initially, each of the alloys was tensile tested in a number of conditions of heat treatment in order to determine whether any of them possessed superior room-temperature strength-ductility relationships. The range of properties of three heats of the Ti-3Mn-complex alloy was used as a basis for comparison, since this material had the best combinations of strength and ductility of any alloy tested to date. A few of the alloys that showed promise were then tested for unstressed elevated-temperature stability at 650 F. Some of the alloys that had good strength and ductility were also evaluated as sheet materials, as described in a later section of this report.

In general, none of the alloys tested had better strength-ductility relationships than the Ti-3Mn-complex alloy. However, two findings of considerable importance resulted from the tensile and stability tests. They were:

(1) A Ti-8V alloy quenched from the beta-phase region and overaged developed very good high-strength properties. This implies that the alloy was much less susceptible to the beta-embrittlement phenomenon than any alloys previously tested under Air Force contracts. Thus, this alloy or similar compositions may represent a new approach to the beta embrittlement encountered in current forging alloys.

(2) A Ti-5Mn-2V alloy was completely stable for 1000 hours at 650 F at a strength level of 200,000 psi. At this strength level, the ductility of the Ti-3Mn-complex alloy was lower after exposure. Thus, superior elevated-temperature stability is indicated for the Ti-5Mn-2V alloy.

The details of test procedures and properties of the various alloys are contained in the following sections of this report.

Heat-Treatment and Testing Procedures

Since the compositions of the alloys listed in Table 1 were considerably different from those of alloys studied previously, certain compositions were evaluated initially for heat-treatment response in terms of hardness. The heat treatments were of the solution-treating and aging type developed under this contract and Contract No. AF 33(038)-3736. The age-hardening curves were similar to those for the Ti-3Mn-complex as regards time to reach peak hardness and overage, but the absolute hardness values were dependent upon the composition. Lower total-alloy materials hardened on quenching and usually had lower maximum hardness. Because of the similarity in aging responses of the various alloys, the times and temperatures applied to the bar-stock tensile blanks were usually the same. The solution-treating temperatures were varied to give approximately the same volume of alpha phase in the microstructure.

The combination of solution and aging temperatures applied to the tensile specimens gave a range of tensile strengths and ductility. Three-inch lengths of the 1/2-inch-diameter bar stock were heat treated in air. These bars were then machined to standard 0.250-inch by 1-inch-gage-section tensile bars. Tensile testing was done on a Baldwin-Southwark Universal Testing Machine, using a head speed of 0.02 inch per minute to fracture. Past experience had shown that good agreement was obtained in duplicate specimens of bar stock; therefore, in the interest of conserving time and expense, single specimens were tested. This permitted yield strengths to be obtained only for specimens believed to have enough ductility to allow an extensometer to be used. Cross-sectional specimens were cut near the shoulders of the test bars for hardness measurements. Vickers hardness values given in the tables are the average of three impressions made with a 10-kilogram load. These hardness specimens were used later for microstructural studies. The tensile and hardness data are presented in the following sections devoted to the various alloy series.

Properties of Binary Titanium-Vanadium Alloys

In order to investigate the effects of vanadium in titanium-base alloys, alloys containing 8, 16, 20, 35, and 49 per cent commercial (93 per cent pure) vanadium were made as 5-pound ingots for testing. The Ti-49V composition was hot short and cracked severely during forging of the first-melt ingot. It also had a liquid oxide at forging temperatures, making it difficult to handle; therefore, no further work was done on this alloy. The Ti-8V alloy forged very easily at 1700 F. As the vanadium content was increased, the alloys were increasingly difficult to forge. The Ti-35V alloy did not forge as easily at 1850 F as did the Ti-8V alloy at 1700 F.

The beta transus was determined roughly for the Ti-8V and Ti-16V alloys, as described in the section "Alloy Selection, Melting, and Fabrication". No beta-transus determination was made for the Ti-20V and Ti-35V alloys, since the phase diagram indicates a transus below 1200 F (WADC Technical Report 53-41, pp 111-114). Because of the difficulty in forging these two compositions, it was deemed impractical to roll either one in the alpha-beta temperature range. Both, therefore, were rolled in the beta-phase region at 1500 and 1600 F.

The heat treatments, tensile properties, and hardnesses of the titanium-vanadium alloys are given in Table 2. For easy comparison, the values of tensile elongation versus tensile strength of the Ti-8V and Ti-16V alloys are given in Figure 1. In Figure 2, the properties of these two alloys after various fabrication and heat treatments are compared with the average properties of the Ti-3Mn-complex alloy in the same conditions (WADC Technical Report 54-205, p 22). The Ti-20V and Ti-35V alloys developed high strengths after heat treatment, but their ductilities were generally low. No further work on these two alloys is contemplated.

The Ti-8V and Ti-16V alloys exhibit some remarkable properties. As shown in Figure 1, they develop essentially the same tensile properties after rolling in either the alpha-beta- or the beta-phase regions and heat treating. After either rolling procedure, the properties of the Ti-8V alloy fell somewhat below those of the Ti-3Mn-complex alloy rolled in the alpha-beta-phase region (Figure 2). When compared with the Ti-3Mn-complex alloy rolled in

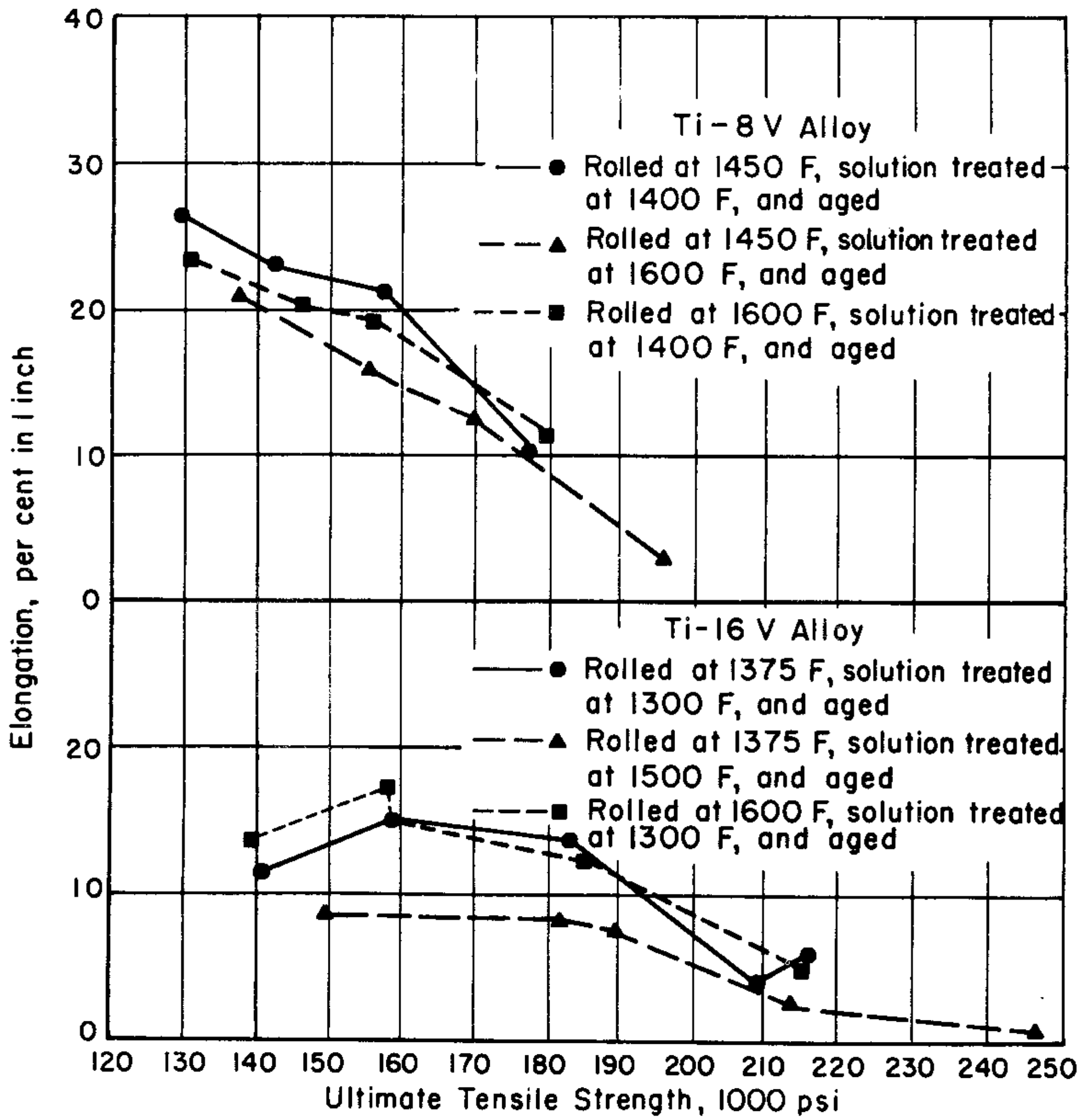

FIGURE 1. TENSILE STRENGTH VERSUS ELONGATION OF HEAT-TREATED $\frac{1}{2}$-INCH-DIAMETER BAR STOCK OF TITANIUM-VANADIUM ALLOYS

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TABLE 2. TENSILE PROPERTIES AND HARDNESSES OF HEAT-TREATED 1/2-INCH-DIAMETER BARS OF TITANIUM-VANADIUM ALLOYS

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F	Elongation, per cent in 1 inch[b]	Reduction of Area, per cent[b]	Yield Strength, 0.2 Per Cent Offset, psi[c]	Ultimate Tensile Strength, psi[b]	VHN[d] (10-Kg Load)
WV12A	1450	1400	48	800	10.5	27.0	170,500	177,500	363
8.0V	1450	1400	24	900	21.0	53.0	153,500	158,000	336
	1450	1400	1	1000	21.5	53.0	150,500	157,500	339
	1450	1400	8	1000	23.0	55.0	138,000	142,500	317
	1450	1400	8	1100	26.5	56.0	125,000	129,500	299
	1450	1600	8	800	(e)	(e)	-	190,500[e]	401
	1450	1600	48	800	3.0[c]	2.0[c]	-	195,500[c]	401
	1450	1600	24	900	12.5	13.5	160,000	170,000	357
	1450	1600	8	1000	16.0	27.0	146,500	155,500	345
	1450	1600	8	1100	21.0[c]	38.0[c]	128,500	137,500[c]	327
	1600	1400	48	800	11.5	22.0	176,000	180,000	373
	1600	1400	24	900	19.5	44.0	151,000	156,000	360
	1600	1400	8	1000	20.5	37.5	140,500	146,500	322
	1600	1400	8	1100	23.5	45.0	124,500	131,000	312
WV13A	1375	1300	8	800	4.0	9.5	201,000	209,500	397
16.0V	1375	1300	48	800	6.0	16.5	210,000	216,000	421
	1375	1300	24	900	13.5	30.0	175,500	183,500	376
	1375	1300	8	1000	15.0	33.0	147,500	158,500	336
	1375	1300	8	1100	11.5	15.5	123,500	141,000	304
	1375	1500	48	800	0.5	2.0	-	247,500	483
	1375	1500	24	900	2.5	5.5	-	213,500	401
	1375	1500	2	1000	7.5	11.5	178,500	189,500	373
	1375	1500	8	1000	8.0	11.0	166,500	182,000	374
	1375	1500	8	1100	8.5	14.5	134,500	149,500	325
	1600	1300	48	800	5.0	8.5	213,500	215,500	429
	1600	1300	24	900	12.5	17.5	177,500	185,500	383
	1600	1300	8	1000	15.0	24.0	145,000	159,000	327
	1600	1300	8	1100	11.5	13.5	122,000	141,000	312

TABLE 2. (Continued)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F	Elongation, Per Cent in 1 Inch[b]	Reduction Of Area, per cent[b]	Yield Strength, 0.2 Per Cent Offset, psi[c]	Ultimate Tensile Strength, psi[b]	VHN[d] (10-Kg Load)
WV14A	1500	1500	48	800	1.0	2.5	-	248,500	459
20.0V	1500	1500	1	1000	6.5	8.0	163,500	185,000	351
	1500	1500	8	1000	6.0	9.0	159,500	180,500	354
	1500	1500	8	1100	7.5	11.5	127,000	150,500	312
	1600	1500	48	800	(f)	(f)	-	240,000[f]	455
	1600	1500	8	1000	5.5	9.5	156,000	179,000	360
	1600	1500	8	1100	6.5	11.5	123,500	150,500	312
WV15A	1500	1500	-	-	7.5	12.0	166,500	174,500	351
35.0V	1500	1500	48	800	0.0	0.5	-	148,500	357
	1500	1500	24	900	0.5	0.5	-	190,000	366
	1500	1500	8	1000	3.0[c]	3.5[c]	-	181,500[c]	357
	1600	1500	48	800	5.5	7.0	179,500	188,000	363
	1600	1500	24	900	3.0[c]	6.5[c]	-	191,000[c]	366
	1600	1500	8	1000	(g)	(g)	-	176,000[g]	327

(a) Cold-water quenched after 1 hour at temperature.
(b) Average of two values.
(c) Single values.
(d) Average of three impressions.
(e) Specimens broke outside of gage marks.
(f) Specimens broke in gage marks at 247,000 and 233,500 psi.
(g) Specimens broke in gage marks.

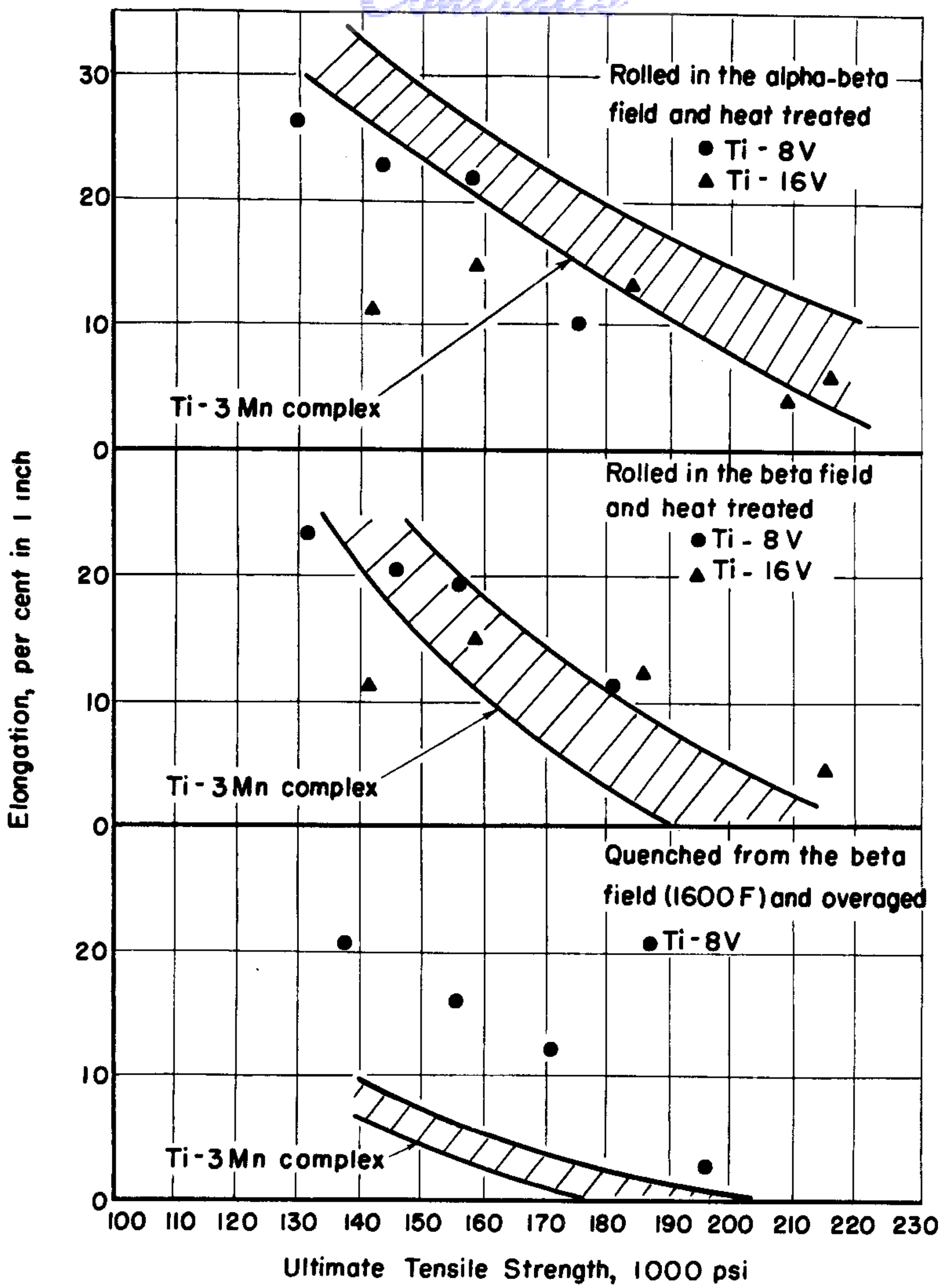

FIGURE 2. COMPARISON OF THE TENSILE PROPERTIES OF THE Ti - 8V AND Ti - 16V ALLOYS WITH THOSE OF THE Ti - 3 Mn - COMPLEX ALLOY AFTER VARIOUS FABRICATION AND HEAT - TREATMENT CYCLES

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the beta-phase region, however, the properties of the Ti-8V and Ti-16V alloys were essentially the same.

The Ti-8V alloy is unusual in that it develops reasonably good high-strength properties after quenching from the beta-phase region and over-aging. As may be seen in Figure 2, this alloy had much better properties than did the Ti-3Mn-complex alloy after quenching from the beta-phase region and aging. The aging behavior of the Ti-8V alloy quenched from 1600 F is also unusual, because the microstructure in the quenched condition was alpha prime, rather than retained beta. Presumably, retained beta is necessary in order for age hardening to occur. It is possible that some retained beta that was not visible microscopically existed in the alloy in conjunction with the alpha prime. It is also possible, however, that the hardening in this alloy was due to some unknown reaction.

Thus, the Ti-8V and Ti-16V alloys appear to be much less susceptible to beta embrittlement than do any alpha-beta-type alloys tested to date. For this reason, vanadium alloys containing up to 16 per cent vanadium look very promising for use as high-strength forging alloys.

Properties of Titanium-Molybdenum-Vanadium Alloys

The titanium-molybdenum-vanadium alloys containing from 2 to 10 per cent total alloy additions were made as 2-pound ingots. There was not enough material to roll at temperatures both below and above the beta transus. However, the extent to which the alloys were susceptible to beta embrittlement was ascertained by solution treating at various temperatures above and below the beta transus. Tensile blanks were solution treated at 1300, 1400, 1500, and 1700 F and aged at temperatures from 800 to 1100 F. The tensile and hardness data for these alloys are given in Table 3. Also shown in the table are the rolling temperatures and heat treatments for each test bar.

All of the alloys responded to age hardening, as is shown by the variations of tensile strength and elongation in Figure 3. The Ti-3Mn-complex alloy property range has been included in these figures for comparison. The Ti-1Mo-1V alloy showed a property range from about 95,000-psi ultimate strength with 33 per cent elongation to 115,000-psi ultimate strength with 21 per cent elongation. In the compositional range of 6 to 8.5 per cent total alloy, the properties varied from 110,000-psi ultimate strength with 31 per cent elongation to about 190,000-psi ultimate strength with 10 per cent elongation. These properties are lower than those of the Ti-3Mn-complex alloy, which ranged from 135,000-psi to 200,000-psi ultimate strength for the same elongation values.

The values plotted in Figure 3 are for solution temperatures of 1300, 1400, and 1500 F. Over this range of solution temperatures, the ductility

TABLE 3. TENSILE PROPERTIES AND HARDNESSES OF HEAT-TREATED 1/2-INCH-DIAMETER BARS OF TITANIUM-MOLYBDENUM-VANADIUM ALLOYS

Heat No. and Nominal Composition, weight per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging: Time, hours	Aging: Temperature, F	Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
WU50A	1425	1300	48	800	33.0	62.5	-	100,500	268
1.0Mo-1.0V	1425	1300	8	1100	30.0	45.5	78,000	96,000	235
	1425	1400	8	900	24 0	35.5	85,500	102,500	285
	1425	1400	24	900	28.0	31.5	85,500	98,500	265
	1425	1400	8	1100	33.0	50.5	79,500	94,500	264
	1425	1500	24	900	22 0	36.0	87,000	114,000	310
	1425	1500	8	1100	30.0	31.5	78,500	97,500	285
WU51A	1450	1300	48	800	29.0	50.0	108,000	118,500	266
1.0Mo-3.5V	1450	1300	8	1100	33.0	57.0	96,000	104,500	237
	1450	1400	8	900	29.5	58.5	107,000	120,000	229
	1450	1400	24	900	30.0	55.0	107,500	117,000	238
	1450	1400	8	1100	32.0	55.0	96,000	107,500	236
	1450	1500	24	900	21.0	60.5	-	148,500	260
	1450	1500	8	1100	30.0	58.5	-	116,000	228
WU59A	1450	1300	48	800	30.0	59.5	-	128,500	317
1.0Mo-5.0V	1450	1300	8	1100	30.0	60.5	108,000	113,000	285
	1450	1400	8	900	21.0	61.0	132,500	141,000	322
	1450	1400	24	900	26.0	58.5	123,000	126,500	297
	1450	1400	8	1100	31.0	60.5	113,000	114,000	276
	1450	1500	48	800	5 0	8.0	171,500	184,000	363
	1450	1500	24	900	18.0	45.0	142,500	152,500	327
	1450	1500	8	1100	28.0	64.0	118,000	122,000	300
	1450	1700	48	800	2 5	4.0	-	175,000	355
	1450	1700	24	900	6.0	9.5	-	162,000	336
WU56A	1450	1300	48	800	30.0	54.5	105,000	119,000	274
3.5Mo-1V	1450	1300	8	1100	33.0	63.0	90,000	104,500	249
	1450	1400	8	900	31.0	56.5	102,500	120,000	276
	1450	1400	24	900	32.0	55.0	102,000	117,500	276
	1450	1400	8	1100	33.0	47.0	95,500	106,000	257
	1450	1500	24	900	16 0	38.5	128,000	154,500	330
	1450	1500	8	1100	28.0	62 0	106,000	117,000	266

TABLE 3. (Continued)

Heat No. and Nominal Composition, weight per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging		Elongation, per cent in 1 Inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
			Time, hours	Temperature, F					
WU55A	1450	1300	48	800	21.0	57.0	127,500	139,500	311
3.5Mo-3.5V	1450	1300	8	1100	31.0	57.0	110,000	116,000	279
	1450	1400	8	900	28.0	59.5	131,000	145,000	317
	1450	1400	24	900	28.0	61.0	-	138,500	306
	1450	1400	8	1100	30.0	63.0	112,500	119,000	289
	1450	1500	48	800	3.0	6.0	199,500	210,500	405
	1450	1500	24	900	10.0	18.0	166,000	178,000	366
	1450	1500	8	1100	29.0	63.0	124,000	132,500	309
	1450	1700	48	800	0.0	1.5	-	202,000	429
	1450	1700	24	900	0.0	1.5	-	183,000	383
WU54A	1450	1300	48	800	25.0	61.5	141,500	152,500	333
3.5Mo-5.0V	1450	1300	8	1100	29.0	64.5	124,000	124,000	294
	1450	1400	8	900	23.0	61.5	146,500	153,500	345
	1450	1400	24	900	27.0	61.0	144,500	149,000	329
	1450	1400	8	1100	30.0	62.0	126,500	129,000	304
	1450	1500	48	800	1.0	2.0	207,000	216,000	437
	1450	1500	24	900	14.0	24.5	172,500	182,500	380
	1450	1500	8	1100	19.0	60.0	133,500	140,500	297
	1450	1700	48	800	0.0	0.5	-	200,000	437
	1450	1700	24	900	3.0	1.5	-	188,500	390
WU58A	1450	1300	48	800	25.0	59.0	123,500	134,000	309
5.0Mo-1.0V	1450	1300	8	1100	32.0	64.0	106,500	108,000	267
	1450	1400	8	900	29.0	64.0	115,000	131,000	306
	1450	1400	24	900	32.0	63.5	116,000	128,000	302
	1450	1400	8	1100	31.0	63.0	109,000	113,000	268
	1450	1500	48	800	10.0	11.0	-	190,000	405
	1450	1500	24	900	17.0	43.0	158,500	179,000	372
	1450	1500	8	1100	25.0	68.0	115,500	124,500	283
	1450	1700	48	800	1.0	1.5	-	198,000	442
	1450	1700	24	900	1.0	0.0	-	187,500	409

TABLE 3. (Continued)

Heat No. and Nominal Composition, weight per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging: Time, hours	Aging: Temperature, F	Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
WU52A	1450	1300	48	800	20.0	59.0	149,000	162,500	349
5.0Mo-3.5V	1450	1300	8	1100	25.0	58.5	123,000	123,000	283
	1450	1400	8	900	19 0	31.5	151,500	159,000	351
	1450	1400	24	900	25.0	54.0	151,000	157,000	342
	1450	1400	8	1100	28.0	55.0	129,500	129,500	309
	1450	1500	48	800	(d)	(d)	-	(d)	433
	1450	1500	24	900	3.0	6.0	177,500	195,500	405
	1450	1500	8	1100	23.0	53.5	133,500	141,500	322
	1450	1700	48	800	0.5	1.5	-	197,000	442
	1450	1700	24	900	1.5	1.5	-	192,500	401
WU53A	1425	1300	48	800	17.0	29.5	160,500	168,500	373
5.0Mo-5.0V	1425	1300	8	1100	25.0	63.5	126,000	126,500	289
	1425	1400	8	900	16.0	38.5	160,000	170,500	380
	1425	1400	24	900	21.0	56.5	158,000	165,500	360
	1425	1400	8	1100	25.0	56.5	133,000	135,500	328
	1425	1500	48	800	(e)	(e)	-	(e)	519
	1425	1500	24	900	10.0	16.5	-	184,000	387
	1425	1500	8	1100	16.0	27.0	132,500	142,000	328
	1425	1700	48	800	(e)	(e)	-	(e)	519
	1425	1700	24	900	2.0	5.8	-	187,000	390

(a) Cold-water quenched after 1 hour at temperature.
(b) Single values.
(c) Average of three impressions.
(d) Specimen broke in shoulder.
(e) Specimens broke in gage marks.

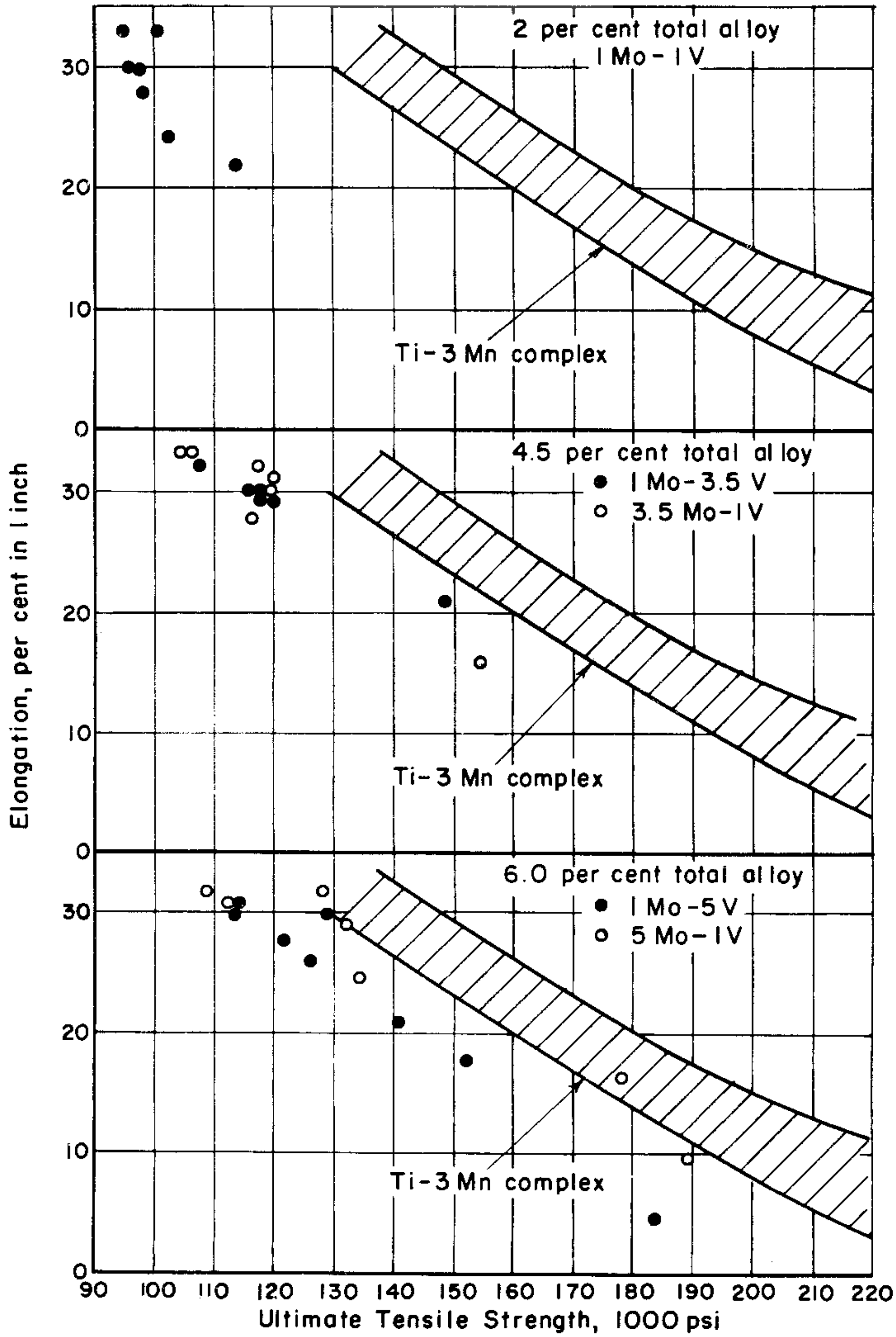

FIGURE 3. TENSILE STRENGTH VERSUS ELONGATION OF HEAT-TREATED $\frac{1}{2}$-INCH-DIAMETER BAR STOCK OF TITANIUM-MOLYBDENUM-VANADIUM ALLOYS ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION A-13486

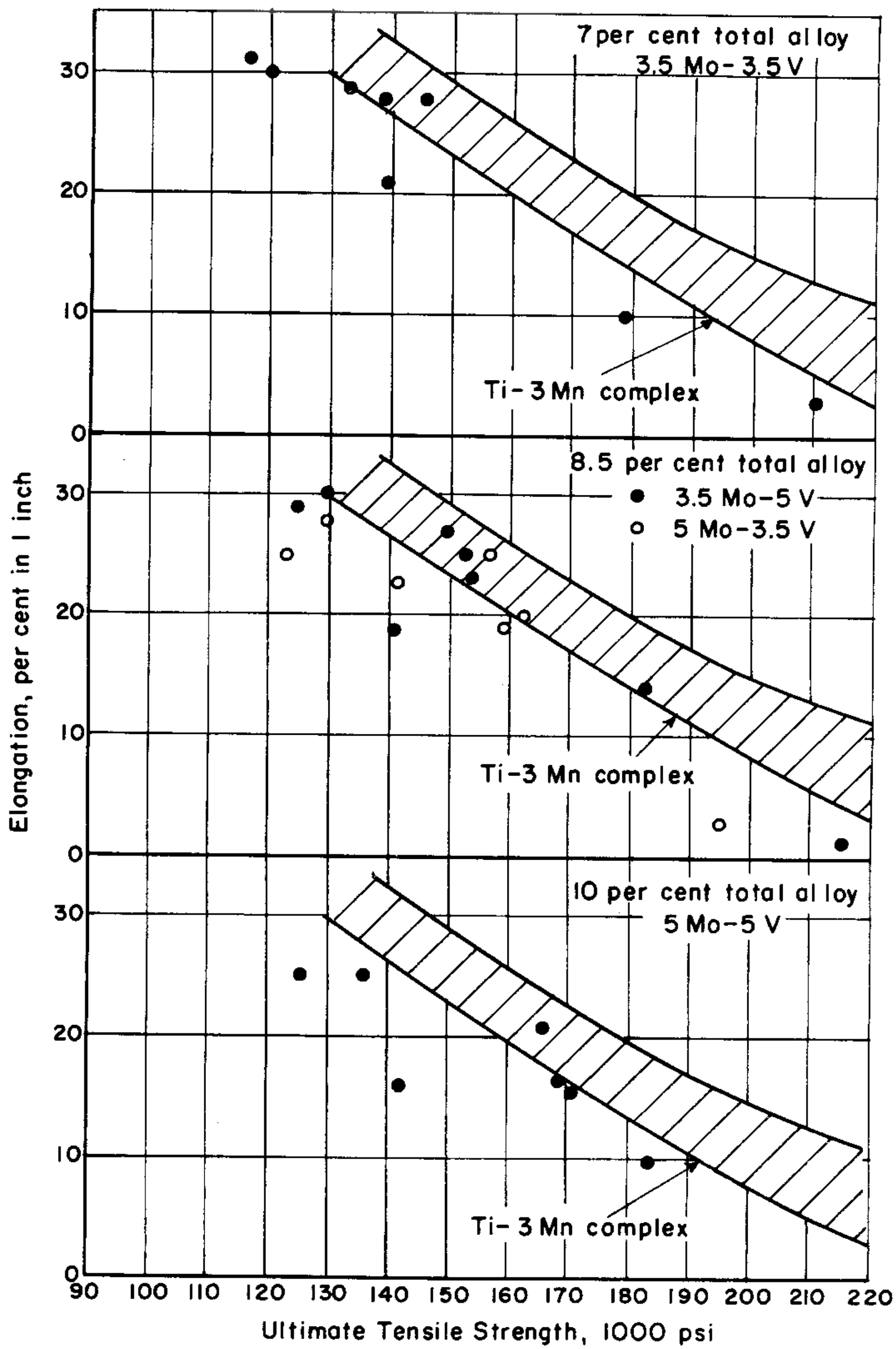

FIGURE 3. (CONTINUED)

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after aging was good. Solution treating at 1700 F, however, decreased the ductility to nearly zero. It would seem, therefore, that these alloys are subject to beta embrittlement to the same degree as are the majority of other beta-stabilized alloys tested to date.

The molybdenum-vanadium alloys probably can be forged at higher temperatures than can the Ti-3Mn-complex alloy and retain good ductility. This is indicated by the good ductility obtained in the aged molybdenum-vanadium alloys after a 1500 F solution treatment. In earlier work, the Ti-3Mn-complex alloy aged after a 1500 F solution treatment showed poor ductility (WADC Technical Report 54-205, p 66). Microstructures of these alloys after solution treating in the beta-phase region showed semicontinuous grain-boundary alpha, which is believed to be one of the basic causes of beta embrittlement.

Properties of Titanium-Manganese-Molybdenum and Titanium-Manganese-Vanadium Alloys

The titanium-manganese-molybdenum and titanium-manganese-vanadium alloys were made as 10-pound ingots, fabricated and heat treated as described in a previous section, and tested at room temperature. The results of tensile tests on heat-treated specimens of these alloys are presented in Table 4. As is shown in Figure 4, the tensile strength-ductility relationships of the alloys rolled and solution treated in the alpha-beta-phase region were somewhat poorer than those of the Ti-3Mn-complex alloy. The alloys containing vanadium had slightly higher ductility than the alloys containing molybdenum. All alloys showed susceptibility to embrittlement when fabricated or heat treated in the beta-phase region. This has been characteristic of most of the beta-stablized alloys evaluated under this program. One important thing, however, may be noted from Figure 4 — the Ti-2Mn-5V and the Ti-5Mn-2V alloys appeared to be somewhat less susceptible to this effect than the titanium-manganese-molybdenum alloys. This seems to be characteristic of many vanadium-containing alloys, as was shown for the Ti-8V and Ti-16V alloys discussed previously.

The elevated-temperature stability of the titanium-manganese-molybdenum and titanium-manganese-vanadium alloys will be discussed later.

Properties of Alloys Produced by Varying the Composition of the Ti-3Mn-Complex

The Ti-3Mn-complex alloy evolved when the effects of various elements were being evaluated with the alloys in the hot-rolled condition. Since the alloy is now heat treated to produce optimum properties, it was felt that the

TABLE 4. TENSILE PROPERTIES AND HARDNESSES OF HEAT-TREATED 1/2-INCH-DIAMETER BARS OF TITANIUM-MANGANESE-MOLYBDENUM AND TITANIUM-MANGANESE-VANADIUM ALLOYS

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature[a], F	Aging: Time, hours	Aging: Temperature, F	Elongation, per cent in 1 inch[b]	Reduction of Area, per cent[b]	Yield Strength, 0.2 Per Cent Offset, psi[b]	Ultimate Tensile Strength, psi[b]	VHN (10-Kg Load)[c]
WW8	1400	1300	24	900	23.0	54.5	135,000	143,000	317
5Mn-2Mo	1400	1300	1	1000	21.0	48.0	138,500	148,000	311
	1400	1300	4	1000	21.0	43.0	131,000	140,500	309
	1400	1300	8	1000	23.0	40.5	128,500	137,500	314
	1400	1300	8	1100	27.0	45.5	119,500	126,000	286
	1400	1400	48	800	9.0	22.0	197,000	199,000	405
	1400	1400	1	1000	16.0	43.0	159,000	170,500	390
	1400	1400	4	1000	17.5	40.5	145,500	156,500	362
	1400	1400	8	1000	23.0	53.5	143,000	152,500	336
	1400	1550	24	900	1.0	3.2	--	193,500	421
	1400	1550	8	1100	9.0	11.8	130,000	137,500	319
	1650	1300	24	900	10.0	15.0	--	145,500	322
	1650	1300	8	1000	13.0	17.8	127,500	139,000	312
	1650	1300	8	1100	19.0	21.3	121,000	131,000	306
WW9	1450	1350	24	900	21.0	43.5	134,500	143,500	317
2Mn-5Mo	1450	1350	8	1100	29.0	58.5	118,000	120,000	274
	1450	1450	48	800	1.0	2.5	195,500	210,000	433
	1450	1450	8	1000	18.5	47.5	137,500	149,500	327
	1450	1600	24	900	0.0	1.4	--	184,500	384
	1450	1600	8	1100	8.0	12.6	127,000	139,000	317
	1650	1350	24	900	14.0	21.1	--	141,000	314
	1650	1350	8	1000	18.0	30.2	117,000	132,000	306
	1650	1350	8	1100	23.0	34.8	111,500	123,500	292

TABLE 4 (Continued)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging		Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
			Time, hours	Temperature, F					
WW10	1400	1300	24	900	21.0	44.0	142,500	151,500	330
5Mn-2V	1400	1300	8	1100	29.0	52.5	123,000	130,000	299
	1400	1400	48	800	9.0	21.0	189,000	199,500	401
	1400	1400	8	1000	23.0	50.5	142,500	150,500	336
	1400	1550	24	900	4.5	12.6	--	186,000	397
	1400	1550	8	1100	18.0	33.4	126,500	134,500	317
	1650	1300	24	900	11.0	13.2	--	154,000	345
	1650	1300	8	1000	17.0	20.7	135,500	143,500	330
	1650	1300	8	1100	19.0	19.3	127,000	135,000	317
WW11	1450	1350	24	900	24.0	59.5	135,000	143,500	314
2Mn-5V	1450	1350	1	1000	25.0	61.0	139,500	147,500	333
	1450	1350	8	1100	29.0	59.0	119,000	125,500	281
	1450	1450	48	800	20.0	48.5	176,000	182,000	401
	1450	1450	1	1000	17.0	58.0	152,500	161,500	351
	1450	1450	8	1000	22.0	62.0	137,500	143,500	319
	1450	1600	24	900	9.0	17.5	--	166,500	352
	1450	1600	8	1000	21.0	39.5	124,000	134,000	329
	1650	1350	24	900	23.0	37.0	125,500	140,500	319
	1650	1350	8	1000	23.5	39.5	121,000	133,500	312
	1650	1350	8	1100	27.0	47.0	115,000	124,000	292

(a) Cold-water quenched after 1 hour at temperature.
(b) Single values.
(c) Average of three impressions.

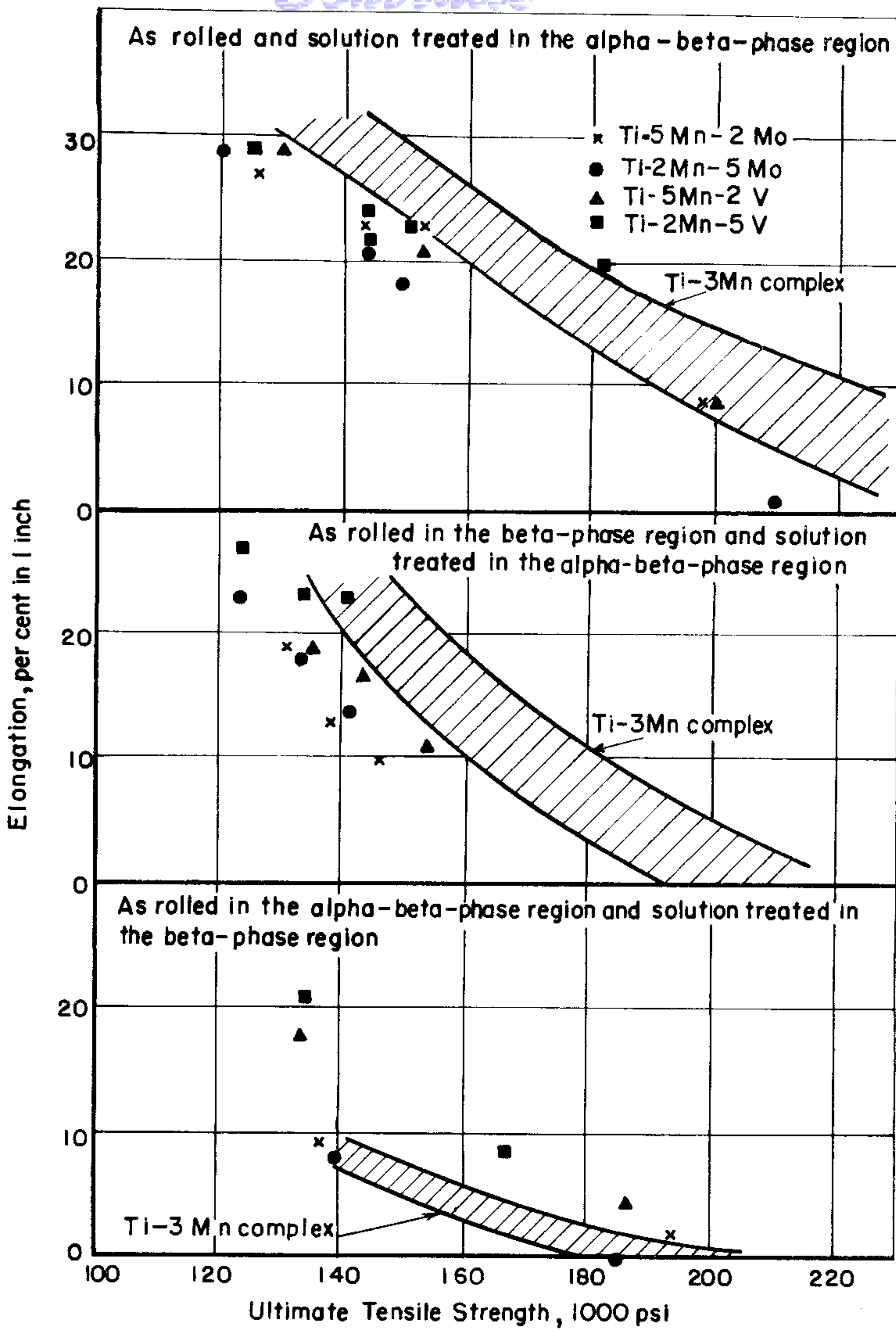

FIGURE 4. EFFECTS OF ROLLING AND SOLUTION TEMPERATURE ON TENSILE PROPERTIES OF TITANIUM-MANGANESE-MOLYBDENUM AND TITANIUM-MANGANESE-VANADIUM ALLOYS

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effects of various alloy components should be evaluated anew. A series of alloys was made to evaluate the following general factors:

(1) Effect of eliminating iron and chromium on tensile properties

(2) Effect of eliminating molybdenum and vanadium on tensile properties

(3) Effect of reducing manganese content on tensile properties

(4) Effect of reducing total alloy content on tensile properties.

These alloys were made as 10-pound ingots. After fabrication, they were solution treated in both the alpha-beta- and the beta-phase regions and aged between 800 and 1100 F for various periods of time. The tensile results for the heat-treated bars are given in Table 5.

As would be expected, the lower alloy-content materials allowed somewhat higher hot-working temperatures and slightly higher alpha-beta-solution temperatures than could be used for the Ti-3Mn-complex alloy.

In general, the strength-ductility relationships of the alloys were somewhat lower than that of the Ti-3Mn-complex alloy shown in Figure 5. This is particularly true of the material that was rolled in the beta-phase region and subsequently heat treated. There are not enough data for a given composition to allow any conclusions to be drawn regarding the relative effects on properties of the eutectoid-forming elements, iron and chromium, and the solid-solution formers, vanadium and molybdenum. However, examination of the data in Table 5 indicates that the former elements, in combination with manganese, give higher strengths after a given solution and aging treatment. Whether this higher strength is the result of greater solid-solution hardening or of an effect on the kinetics of the aging reaction is not known.

The one trend that is shown fairly conclusively by these data is the effect on properties of reducing the total alloy content below the nominal 7 per cent figure. All of the alloys tested contained, nominally, less total alloy than the Ti-3Mn-complex. It is apparent from Figure 5 that several of the alloys, particularly those containing 4 or 5 per cent total alloy, had somewhat lower ductility at a given strength level than did the Ti-3Mn-complex alloy. This apparent loss of ductility is probably associated with the relative amount of the retained beta and the relative strength of this beta-precipitated-alpha mixture present in the alloys after aging. As the total alloy content decreases, the relative volume occupied by the beta phase containing precipitated alpha must decrease. In order to maintain a given strength, then, these beta areas must become stronger and, therefore, less

TABLE 5. EFFECTS OF COMPOSITIONAL CHANGES ON PROPERTIES OF Ti-3Mn-COMPLEX ALLOY (HEAT-TREATED 1/2-INCH-DIAMETER BAR STOCK)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging Time, hours	Aging Temperature, F	Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
WV1	1450	1300	48	800	20.0	52.0	169,000	182,500	389
3Mn-1Fe-1Cr-	1450	1300	8	900	19.0	40.0	161,500	169,000	335
1Mo-1V	1450	1300	24	900	24.5	57.5	155,000	159,000	333
	1450	1300	2	1000	25.0	59.0	154,500	155,000	309
	1450	1300	8	1000	26.5	62.0	146,500	148,000	309
	1450	1300	8	1100	29.0	60.5	134,500	138,000	299
	1450	1375	48	800	8.5	13.0	186,500	201,000	413
	1450	1375	8	900	15.0	30.0	169,500	180,000	360
	1450	1375	24	900	21.0	58.0	164,000	170,500	370
	1450	1375	2	1000	23.0	59.0	161,000	168,000	354
	1450	1375	8	1000	25.0	60.0	156,000	158,000	354
	1450	1375	8	1100	27.0	60.5	141,000	143,500	317
	1450	1450	48	800	12.5	42.5	206,000	215,000	442
	1450	1450	8	900	15.0	50.0	185,000	192,000	390
	1450	1450	24	900	14.0	32.0	172,500	181,000	394
	1450	1450	2	1000	18.0	56.0	172,500	178,000	380
	1450	1450	8	1000	20.0	39.5	157,500	168,000	366
	1450	1450	8	1100	21.0	41.0	142,500	148,500	333
	1450	1600	24	900	0.0	0.0	--	202,000	409
	1450	1600	8	1000	1.0	3.5	--	181,500	382
	1450	1600	24	1000	1.5	6.5	--	167,000	370
	1450	1600	8	1100	7.5	15.0	141,500	143,500	333
	1650	1300	48	800	3.5	4.0	157,500	179,000	-
	1650	1300	24	900	12.0	15.5	146,500	159,000	-
	1650	1300	8	1000	15.5	19.5	142,000	148,500	317
	1650	1300	8	1100	19.0	22.5	135,500	141,000	319

TABLE 5. (Continued)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging		Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
			Time, hours	Temperature, F					
WV2	1500	1400	24	900	22.0	48.0	133,000	144,500	317
3Mn-1Mo-1V	1500	1400	8	1100	36.0	58.0	118,000	126,000	292
	1500	1500	48	800	18.0	47.0	168,000	180,000	370
	1500	1500	8	1000	28.0	63.0	134,000	146,000	319
	1500	1650	24	900	2.5	6.3	--	169,500	366
	1500	1650	8	1100	9.0	13.8	125,500	138,000	312
	1700	1400	24	900	18.0	18.9	122,500	145,500	322
	1700	1400	8	1000	21.0	30.0	118,000	136,000	312
	1700	1400	8	1100	24.5	32.5	113,500	126,000	299
WV3	1450	1350	24	900	20.0	39.0	138,000	154,000	327
3Mn-1Cr-1Fe	1450	1350	8	1100	28.0	47.0	122,000	127,500	283
	1450	1450	48	800	11.0	24.0	171,000	192,500	380
	1450	1450	8	1000	25.0	47.5	133,000	152,500	333
	1450	1600	24	900	0.0	0.0	--	166,500	394
	1450	1600	8	1100	6.0	7.9	124,500	137,500	329
	1650	1350	24	900	9.0	11.2	124,000	147,500	330
	1650	1350	8	1000	15.0	18.9	119,500	139,000	317
	1650	1350	8	1100	23.0	29.3	122,500	129,000	292
WV4	1450	1375	24	900	23.0	48.5	152,000	160,500	336
2Mn-1Fe-1Cr-	1450	1375	8	1100	27.0	61.0	130,000	134,500	304
1Mo-1V	1450	1475	48	800	9.5	25.5	180,000	201,500	401
	1450	1475	8	1000	22.0	52.5	143,500	157,500	345
	1450	1625	24	900	1.0	2.4	--	191,000	394
	1450	1625	8	1100	9.0	15.4	135,500	145,000	336
	1700	1375	24	900	12.5	15.4	136,500	158,000	345
	1700	1375	8	1000	18.0	24.3	129,500	146,500	339
	1700	1375	8	1100	20.0	36.2	124,000	134,000	322

TABLE 5. (Continued)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging: Time, hours	Aging: Temperature, F	Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
WV6	1500	1425	24	900	24.5	51.0	116,500	134,000	294
2Mn-1Mo-1V	1500	1425	8	1100	30.0	53.5	105,500	117,500	215
	1500	1525	48	800	14.0	37.0	152,000	175,500	366
	1500	1525	8	1000	25.0	55.0	126,000	141,500	304
	1500	1675	24	900	3.0	40.7	--	153,000	333
	1500	1675	8	1100	11.5	12.7	112,500	128,500	299
	1700	1425	24	900	23.0	30.9	112,000	122,500	306
	1700	1425	8	1000	26.5	32.7	108,000	127,000	297
	1700	1425	8	1100	28.0	44.6	103,000	118,000	289
WV7	1450	1375	24	900	20.0	34.0	118,500	140,500	297
2Mn-1Fe-1Cr	1450	1375	8	1100	30.0	49.0	108,000	122,000	270
	1450	1475	48	800	16.0	38.0	150,500	171,500	353
	1450	1475	8	1000	25.0	43.0	120,000	137,500	314
	1450	1625	24	900	1.0	2.4	--	160,000	370
	1450	1625	8	1100	9.0	11.8	113,500	130,000	302
	1700	1375	24	900	15.0	20.6	112,500	138,000	312
	1700	1375	8	1000	20.0	36.8	110,500	130,500	292
	1700	1375	8	1100	29.0	42.0	107,000	121,500	279
WV5	1500	1400	24	900	20.0	47.0	134,500	149,000	325
1Mn-1Fe-1Cr-	1500	1400	1	1000	22.0	48.5	133,500	152,500	327
1Mo-1V	1500	1400	8	1100	28.0	50.5	117,500	128,000	294
	1500	1500	48	800	16.5	49.0	165,000	184,500	383
	1500	1500	1	1000	17.5	57.5	156,500	171,000	370
	1500	1500	8	1000	23.0	57.0	138,500	151,500	333
	1500	1650	24	900	2.0	3.2	--	173,000	373
	1500	1650	8	1100	10.0	11.8	122,500	138,000	322
	1700	1400	24	900	14.0	19.1	123,500	146,500	342
	1700	1400	8	1000	17.0	21.9	116,000	136,500	319
	1700	1400	8	1100	23.5	36.0	110,500	126,000	297

TABLE 5. (Continued)

Heat No. and Nominal Composition, per cent	Rolling Temperature, F	Solution Temperature(a), F	Aging Time, hours	Aging Temperature, F	Elongation, per cent in 1 inch(b)	Reduction of Area, per cent(b)	Yield Strength, 0.2 Per Cent Offset, psi(b)	Ultimate Tensile Strength, psi(b)	VHN (10-Kg Load)(c)
WW20	1450	1325	24	900	20.0	39.5	147,000	155,500	339
3Mn-1Fe-1Cr-1Mo-	1450	1325	8	1100	30.5	54.0	125,000	130,500	312
1V-0.1Th	1425	1425	48	800	8.0	18.0	199,500	209,000	421
	1425	1425	8	1000	19.0	44.0	153,500	163,500	354
	1425	1575	24	900	0.0	0.6	--	181,000	-
	1425	1575	8	1100	11.0	15.9	130,000	136,500	-
	1625	1325	24	900	12.0	17.6	129,500	151,000	339
	1625	1325	8	1000	17.0	28.2	125,000	142,500	325
	1625	1325	8	1100	23.5	41.6	116,500	128,000	302
WW21	1425	1325	24	900	18.0	40.5	142,500	155,500	345
3Mn-1Fe-1Cr-	1425	1325	8	1100	28.0	50.0	121,000	129,500	285
1Mo-1V-0.25Th	1425	1425	48	800	7.0	17.0	199,500	209,500	413
	1425	1425	8	1000	21.0	44.5	144,500	157,000	348
	1425	1575	24	900	0.0	0.2	--	176,500	401
	1425	1575	8	1100	9.0	15.3	125,500	135,500	314
	1625	1325	24	900	12.0	14.8	122,000	148,000	325
	1625	1325	8	1000	19.0	25.8	117,500	137,000	322
	1625	1325	8	1100	23.0	34.0	113,000	126,500	314

(a) Cold-water quenched after 1 hour at temperature.
(b) Single values.
(c) Average of three impressions.

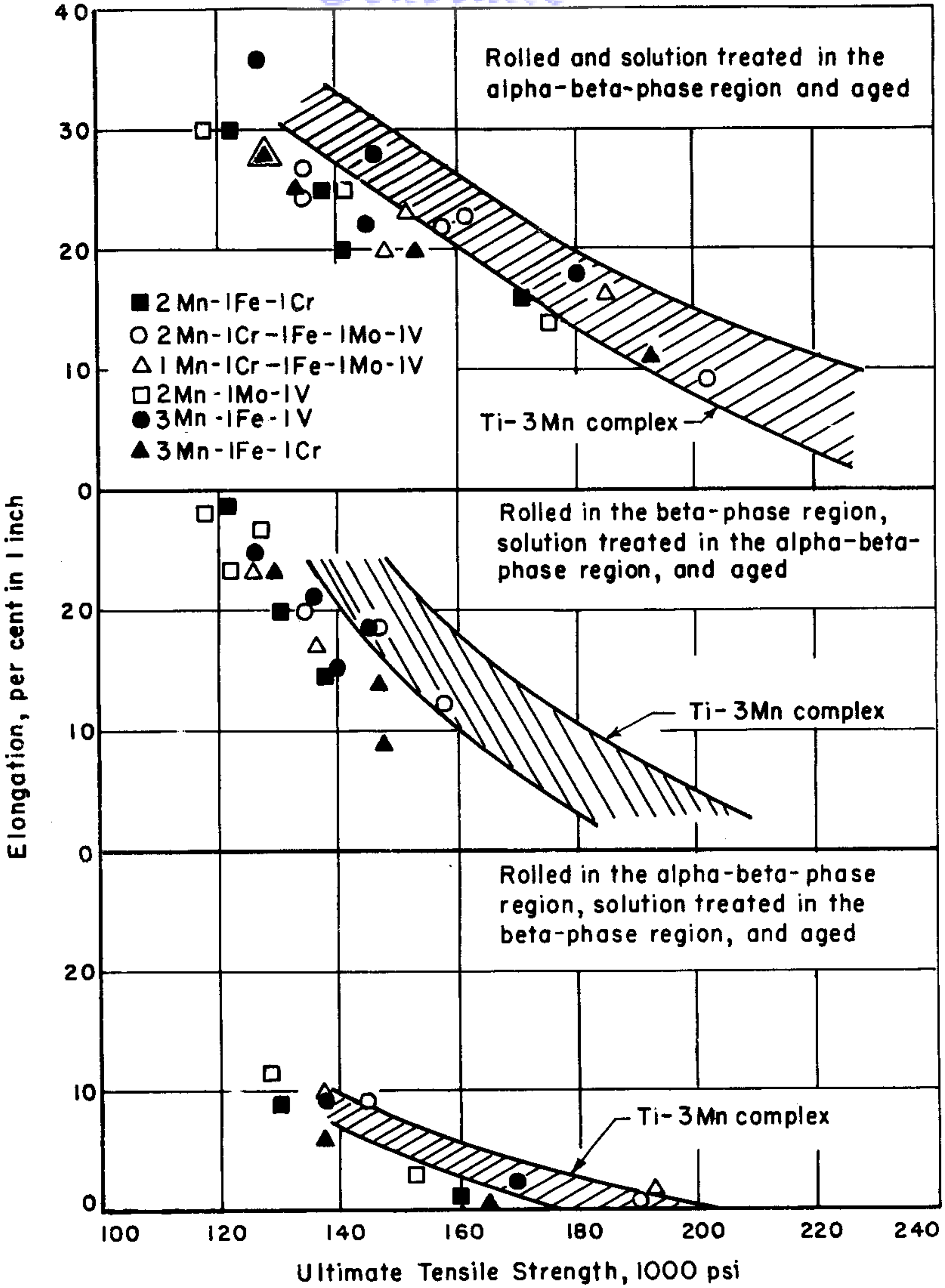


FIGURE 5. COMPARISON OF THE PROPERTIES OF VARIOUS COMPLEX ALLOYS WITH THOSE OF THE Ti-3Mn-COMPLEX ALLOY AFTER DIFFERENT TREATMENTS

A-13488

ductile. It is reasonable to assume that the lower ductility of the beta regions reduces the over-all ductility of the alloy.

These considerations suggest that, for any alloy system, there should be an optimum total alloy content, which will give the best strength-ductility ratio over a range of strength levels after heat treatment. In the complex system just described, the optimum content appears to be 7 per cent or higher.

Microstructure

The properties of the experimental alloys discussed in the preceding sections were correlated with their microstructures. Specimens for micro-examination were usually cut at the shoulder area of the test bar and the cross section examined. All specimens were mounted in Bakelite and hand ground through 600-grit, wet silicon carbide paper. Final polishing was done with rouge on a small-diameter, high-speed (1725 rpm) polishing wheel covered with a cloth like "Mira cloth". Etching was usually done by swabbing with a solution containing 1.5 per cent hydrofluoric acid and 3.5 per cent nitric acid in water. This etchant produced a dark stain on certain alloys containing relatively large amounts of molybdenum and/or vanadium. The alloys that stained with the above solution were etched by swabbing with a solution of 20 per cent hydrofluoric acid and 20 per cent nitric acid in glycerine.

The alloys all had the same basic structural constituents, namely, alpha and beta phases, which have a characteristic distribution for each condition of fabrication and solution treatment. Aging had little effect on the basic appearance, as usually the alpha precipitate produced was too fine to be resolved microscopically, but merely caused the beta matrix to etch darker.

The relationship between the fabrication and solution treatments and the characteristic microstructures may be summarized as follows:

(1) Rolling and solution treating in the alpha-beta-phase region produced equiaxed alpha uniformly distributed in the beta matrix, as shown for the Ti-3Mn-complex alloy in Figure 6. Alloys with this microstructure had excellent strength-ductility relationships.

(2) Rolling in the beta-phase region and solution treating in the alpha-beta-phase region produced a large-grained structure having continuous alpha at the prior beta grain boundaries and a Widmanstätten alpha within the grains, as shown for the Ti-3Mn-complex alloy in Figure 7. Alloys with this microstructure had intermediate ductility at low strength levels and low ductility at high strength levels.

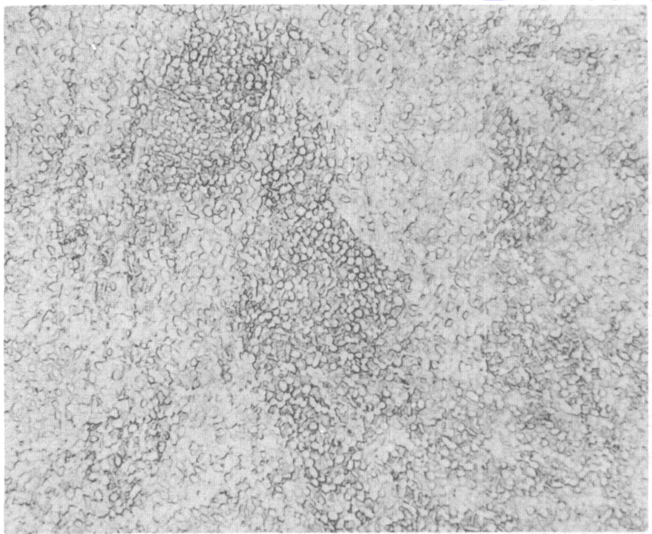
(3) Rolling in either the alpha-beta or the beta-phase region and solution treating in the beta-phase region produced a large-grained structure. When sufficient beta-stabilizing elements were present, a fully retained-beta structure was obtained which, upon aging, showed a fine precipitate of alpha within the grains and continuous grain-boundary alpha around the beta grains. In addition to the alpha at the grain boundaries, there appeared to be an unidentified grain-boundary phase in both the aged and the as-quenched conditions. The structure of the Ti-3Mn-complex alloy, quenched from the beta-phase region and aged, is shown in Figure 8. Specimens having this structure had low ductility at all strength levels.

The Ti-8V alloy had a structure that appeared to be fully transformed to alpha prime, as shown in Figure 9. There was no apparent change in the structure during aging, but the alloy did show age hardening. The Ti-8V alloy specimens with this structure had good ductility when aged to strength levels up to 180,000 psi.

The basic structures may be altered somewhat by alloying elements. The main alteration is in the size of the various microconstituents. Molybdenum and vanadium showed a strong tendency to reduce the alpha-grain size for specimens rolled and solution treated in the alpha-beta-phase region. The microstructures in Figures 10 and 11 show fine primary alpha structures in the Ti-5Mo-2Mn alloy and the Ti-16V alloy, respectively. The glycerine-base etchant caused much lighter staining than did the standard hydrofluoric acid-nitric acid etch.

Elevated-Temperature Stability

One primary aim of the research this year was to obtain improved stability at elevated temperatures. The Ti-3Mn-complex alloy has proved to be stable for 1000 hours at 650 F unstressed at strength levels up to 170,000 psi. An improvement in stability at higher temperatures or at higher strength levels was desired. Only the alloy compositions that could be heat treated to strengths above 170,000 psi were evaluated. Tensile blanks of the heat-treated bar stock of each alloy were held unstressed in air at 650 F for 200 and 1000 hours. After these exposures, the blanks were machined to tensile specimens and tested at room temperature. The tensile data for these tests are presented in Table 6. Included in the table are the data previously obtained (WADC Technical Report 54-205, p 38) for the Ti-3Mn-complex alloy heat treated to two strength levels.



500X N86

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 6. Ti-3Mn-COMPLEX ALLOY, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION, AND AGED

Structure: Random spheroidal primary alpha and unresolved alpha precipitate in a matrix of retained beta.

Characteristics: Excellent strength-ductility relationships at all strength levels.

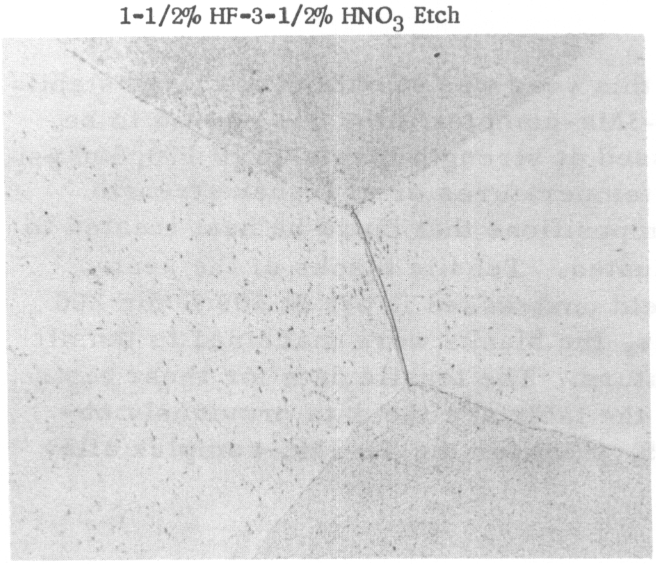
500X N81

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 7. Ti-3Mn-COMPLEX ALLOY, ROLLED IN THE BETA-PHASE REGION, SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION, AND AGED

Structure: Grain boundary and Widmanstätten primary alpha and unresolved alpha precipitate in a beta matrix.

Characteristics: Fair ductility at low strength levels; poor ductility at high strengths.



500X N83

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 8. Ti-3Mn-COMPLEX ALLOY, ROLLED IN THE ALPHA-BETA-PHASE REGION, SOLUTION TREATED IN THE BETA-PHASE REGION, AND AGED

Structure: Beta matrix with fine alpha precipitate within beta grains. Alpha and possibly an unidentified phase at the beta grain boundaries.

Characteristics: Low ductility at all strength levels.

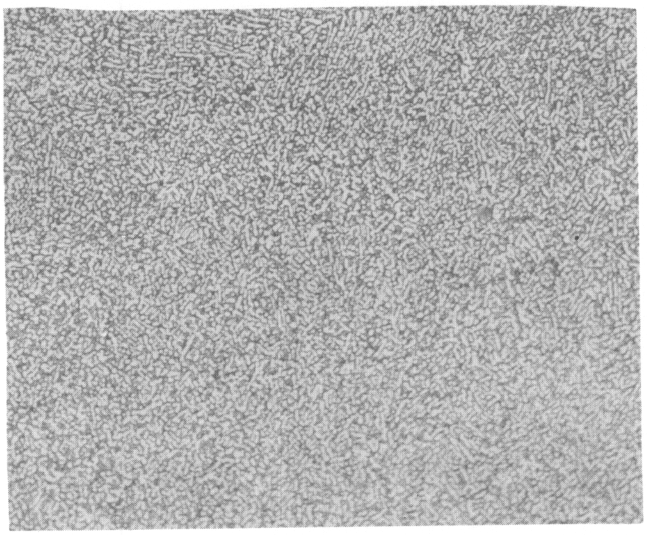
500X N19249

20% HF-20% HNO_3-60% Glycerine Etch

FIGURE 9. Ti-8V ALLOY, ROLLED IN THE ALPHA-BETA-PHASE REGION, SOLUTION TREATED IN THE BETA-PHASE REGION, AND AGED

Structure: Aged alpha prime, possibly some precipitated alpha and retained beta.

Characteristics: Good ductility at strength levels up to 180,000 psi.

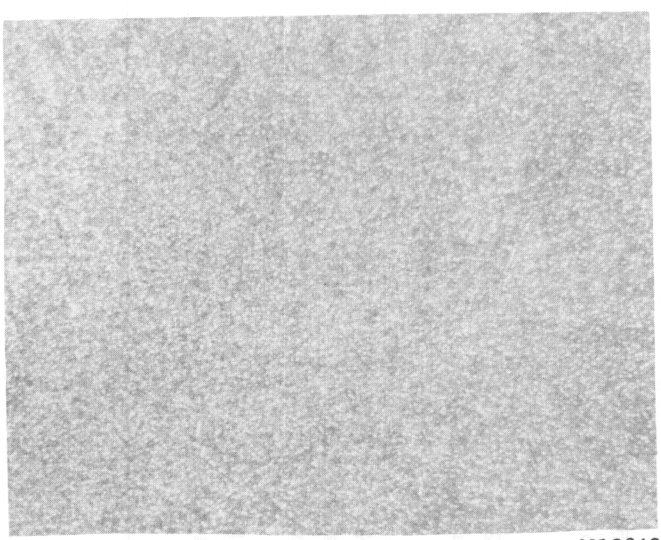


500X N18363

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 10. Ti-5Mo-2Mn ALLOY, SOLUTION TREATED AT 1350 F AND AGED 8 HOURS AT 1100 F

Structure: Fine primary alpha (white) and very dark beta matrix that contains an unresolved pre-cipitate of alpha.



500X N18648

20% HF-20% HNO_3-60% Glycerine Etch

FIGURE 11. Ti-16V ALLOY, SOLUTION TREATED AT 1300 F AND AGED 48 HOURS AT 800 F

Structure: Very fine primary alpha (white) and dark beta matrix that contains an un-resolved precipitate of alpha.

TABLE 6. EFFECT OF UP TO 1000 HOURS' EXPOSURE AT 650 F ON ROOM-TEMPERATURE TENSILE PROPERTIES OF SEVERAL HEAT-TREATED TITANIUM ALLOYS

Heat No. and Nominal Composition, per cent	Heat Treatment: Solution Temperature[a], F	Heat Treatment: Aging Time, hours	Heat Treatment: Aging Temperature, F	Time at 650 F, hours	Elongation, per cent in 1 inch[b]	Reduction of Area, per cent[c]	Yield Strength, 0.2 Per Cent Offset, psi[b]	Ultimate Tensile Strength, psi[b]
WT136A and WU24A	1300	24	800	–	12.5	27.0	167,500	190,500
3Mn-1Fe-1Cr-	1300	24	800	200	6.0	–	–	201,500
1Mo-1V	1300	24	800	1000	7.0	14.0	175,000	197,500
WT136A	1400	2	1000	–	23.5	52.0	160,500	166,000
WU24A	1400	2	1000	200	17.0	29.5	161,500	171,500
	1400	2	1000	1000	17.5	46.5	162,000	172,000
WU58	1500	48	800	–	10.0	11.0	–	190,000
5Mo-1V	1500	48	800	200	6.0	9.5	168,500	191,500
	1500	48	800	1000	8.5	11.0	155,000	186,500
WV2	1500	48	800	–	18.0	47.0	168,000	180,000
3Mn-1Mo-1V	1500	48	800	200	20.0	51.5	159,500	173,500
	1500	48	800	1000	19.0	42.0	157,000	173,000
WV4	1475	48	800	–	9.5	25.5	180,000	201,500
2Mn-1Fe-1Cr-	1475	48	800	200	12.5	23.0	189,500	197,500
1Mo-1V	1475	48	800	1000	11.0	17.5	185,500	195,000
WW8	1400	48	800	–	9.0	22.0	197,000	199,000
5Mn-2Mo	1400	48	800	200	15.0	43.0	172,000	188,500
	1400	48	800	1000	15.5	42.0	171,000	187,500

TABLE 6. (Continued)

Heat No. and Nominal Composition, per cent	Heat Treatment			Time at 650 F, hours	Elongation, per cent in 1 inch[b]	Reduction of Area, per cent[c]	Yield Strength, 0.2 Per Cent Offset, psi[b]	Ultimate Tensile Strength, psi[b]
	Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F					
WW10	1400	48	800	-	9.0	21.0	189,000	199,500
5Mn-2V	1400	48	800	200	14.0	39.0	189,500	199,500
	1400	48	800	1000	14.0	32.5	185,500	198,000
WW21	1425	48	800	-	7.0	17.0	199,500	209,500
3Mn-1Fe-1Cr-	1425	48	800	200	6.0	12.0	196,000	207,000
1Mo-1V-0.25Th	1425	48	800	1000	5.0	6.0	200,000	206,500

(a) Cold-water quenched after 1 hour at temperature.
(b) Average of two values.
(c) Single values.

Exposure at 650 F had little effect on the ultimate strengths of all but two of the alloys. The Ti-3Mn-1Mo-1V and Ti-5Mn-2Mo alloys showed a decrease in strength after the 200-hour exposure but essentially no further change. This may have been caused by initial unintentional variations in heat treatment, because the control specimens were heat treated at a different time than those that were exposed to the elevated temperature. Since the decrease in strength was accompanied by an increase in ductility, it may be that the alloys were insufficiently overaged to produce a stable structure. The loss of strength was not great, but may indicate that a transformation was taking place that could cause an increased creep rate in these particular alloys. It may also be noted that, for the Ti-5Mn-2Mo alloy, the yield-tensile ratio decreased after long-time exposure at 650 F.

Of the remaining alloys, the Ti-5Mn-2V and the Ti-2Mn-1Cr-1Fe-1Mo-1V showed the best properties after exposure. The Ti-5Mn-2V alloy retained a strength of 198,000 psi, with an increase from 9 to 14 per cent elongation, and the Ti-2Mn-complex alloy showed a slight decrease in strength to 195,000 psi and an increase in elongation to 11.5 per cent after the 1000-hour exposure at 650 F. The Ti-3Mn-complex alloy, while retaining the above strength level, showed a decrease in elongation to about 7 per cent for the same exposure.

It is apparent that none of the alloys tested became completely embrittled during the exposure. With one or two exceptions, the changes in strength between the 200-hour and the 1000-hour exposures were insignificant. It is impossible to conclude, from these data, that any one of the several more promising alloys is the most stable. However, it would appear that further tests should be made on the Ti-5Mn-2V and Ti-2Mn-complex alloys in preference to the others. The stability of these alloys when exposed to elevated temperatures under stress may differ from their stability when not stressed.

EVALUATION OF SELECTED ALLOYS AS SHEET

Another phase of this year's work was to develop a high-strength, heat-treatable sheet alloy with adequate formability. Actual evaluation of sheet materials was rather limited during the period covered by this report for two reasons: (1) a great deal of effort was devoted to evaluating the effects of hydrogen on the Ti-3Mn-complex alloy, and (2) it was necessary to develop a method of processing alloy sheet to give consistently good ductility.

As far as strength is concerned, the same alloy compositions that have shown excellent strength and ductility as bar stock should have essentially the same properties in sheet form. However, this has not been the case in the past. Various explanations have been advanced for the poor ductility of the heat-treated sheet, among them:

(1) Presence of hydrogen

(2) Sharp corners on the test section

(3) Surface contamination.

Work under the present contract indicated that excessive amounts of hydrogen, much of it introduced during the pickling, was probably the chief cause of low ductility. Therefore, a major part of the effort on sheet alloys was directed toward developing a method of processing sheet that would produce material free from surface contamination and having low hydrogen content.

Development of a Method of Processing Alloy Sheet

The deleterious effect of hydrogen was indicated in some earlier work under this contract (WADC Technical Report 54-205, p 51). However, conclusive proof of its effects and the discovery that large amounts of hydrogen could be introduced into sheet during pickling occurred under the present contract extension.

The first evidence that hydrogen was absorbed by sheet during pickling was found during some experiments on sheet rolled from a large ingot of the Ti-3Mn-complex alloy. Two series of test specimens were apparently treated in the same manner; i.e., after similar heat treatments, they were pickled in an ammonium bifluoride-sulfuric acid solution to remove the scale and contaminated surface metal. When the tensile results were obtained, showing large differences in tensile elongation between the two groups of specimens, a hydrogen analysis was made of one specimen from each series. The hydrogen contents of the two specimens and the tensile-test results were as follows:

Hydrogen Content, ppm by weight		Elongation, per cent in 1 inch	Ultimate Tensile Strength, psi	VHN (10-Kg Load)
As Rolled	Pickled and Heat Treated			
80	200	10.0	157,000	354
80	660	0.5	151,000	335

The large difference in hydrogen contents appeared to be the cause of the variation in tensile ductility. Of greater concern was the high level of hydrogen in both series of specimens, as compared with the as-rolled material. This excess hydrogen was apparently picked up during the pickling operation.

With the finding of excessive hydrogen pickup from the pickling bath, the descaling and pickling procedures were evaluated.

Investigation of Descaling and Pickling Methods

The work just described indicated strongly that the ammonium bifluoride-sulfuric acid solution usually used for pickling introduced hydrogen into the titanium-alloy sheet. There was some question as to the mechanism of the introduction of hydrogen, i.e., whether it formed a surface film of hydride that later diffused into the sheet or whether it diffused directly into the material. There had been some indication in research under Contract No. AF 33(616)-445 that the first mechanism was operative. Some specimens prepared for X-ray diffraction examination by pickling showed a strong TiH pattern when examined immediately after etching. If the specimens were allowed to stand a few days, however, no hydride pattern was detected. Experiments were made to investigate the hydrogenating tendencies of the pickling bath normally used and to determine whether hydrogenation during pickling was connected with the formation of a surface hydride film.

The following two etchants were used for these experiments:

A - 2 grams of ammonium bifluoride per 100 cc of a 33 per cent by volume sulfuric acid solution (used at 170 F)

B - 70 per cent nitric acid and 5 per cent hydrofluoric acid by volume in water (used at 190 F).

Etchant A had been used in the past to remove hot-rolled scale. Etchant B would not remove as-rolled scale. However, metal could be removed uniformly with the latter etchant after the scale had been removed by grit blasting.

Hydrogen analyses were made on Ti-3Mn-complex sheet material treated as follows after being pickled in the above etchants:

(1) As pickled

(2) As pickled, with about 0.002 inch filed off the surface

(3) As aged 1 week at room temperature

(4) As aged 1 week at room temperature, and with 0.002 inch filed off the surface

(5) As solution treated 1/2 hour at 1300 F and water quenched, with 0.002 inch filed off the surface.

All of the conditions were applied to 0.06-inch-thick sheet and a few tests were made on 0.020-inch-thick sheet to determine the effect of surface-to-volume ratio on hydrogen pickup. The results of these tests are given in Table 7.

The following observations may be made from the data in Table 7:

(1) The sulfuric acid-ammonium bifluoride etchant excessively increased the hydrogen content of this sheet material. The hydrogen pickup was roughly proportional to the volume-to-surface ratio.

(2) The nitric acid-hydrofluoric acid etchant produced no significant increase in hydrogen content.

(3) The hydrogen apparently diffused rapidly throughout the sheet (no difference in hydrogen content was noted after filing off the surface).

(4) Aging at room temperature had no effect on hydrogen content.

(5) Solution heat treating at 1300 F decreased the hydrogen content from 380 to 280 ppm by weight, but had no effect at a hydrogen level of 90 ppm.

Thus, the tendency of the ammonium bifluoride-sulfuric acid solution to introduce hydrogen into alloy sheet was firmly established. These results also indicated that the hydrogen diffuses rapidly into the material, rather than forming a surface film of hydride.

Although the 70 per cent nitric acid-5 per cent hydrofluoric acid pickle appeared to add no hydrogen during pickling, it has several disadvantages:

(1) It may become explosive. It has been reported that 65 per cent boiling nitric acid used in corrosion tests of titanium has exploded.

(2) It does not effectively remove the scale formed during hot rolling or solution treating.

(3) It fumes excessively during use.

Consideration of these factors led to the initiation of a rather extensive program aimed at developing a method of descaling and pickling sheet that would produce a smooth, contamination-free surface without appreciably increasing the hydrogen content. Two methods of descaling were used: (1) treatment in a molten sodium hydride bath, and (2) treatment in a molten Virgo salt bath. Various pickling solutions were used in conjunction with

TABLE 7. EFFECT OF ETCHANTS ON HYDROGEN CONTENT OF Ti-3Mn-COMPLEX-ALLOY SHEET MATERIAL

Nominal Sheet Thickness, mils	Pickled in Etchant[a]	Further Treatment	Hydrogen Content, ppm by weight
20	--	Filed[b]	96
60	--	Filed[b]	80
20	A	--	890
20	B	--	92
60	A	--	370
60	B	--	85
60	A	Filed[b]	350
60	B	Filed[b]	100
20	A	Aged 1 week[c]	890
20	B	Aged 1 week[c]	97
60	A	Aged 1 week[c]	360
60	B	Aged 1 week[c]	79
60	A	Aged 1 week[c] and filed[b]	380
60	B	Aged 1 week[c] and filed[b]	79
60	A	1/2 hr at 1300 F, CWQ, and filed[b] [d]	280
60	B	1/2 hr at 1300 F, CWQ, and filed[b] [d]	90

(a) All specimens grit blasted and 0.001 inch etched off surface in: Etchant A, 33 per cent H_2SO_4 in water with 2 grams $NH_4F \cdot HF$ per 100 cc of solution at about 170 F, or Etchant B, 70 per cent HNO_3, 5 per cent HF, and 25 per cent H_2O at 190 F.

(b) About 0.002 inch filed off all surfaces just before analysis.

(c) At room temperature.

(d) Solution treatment started within 5 minutes after etching.

these descaling methods in an attempt to find a solution more suitable for general use. The pickling solutions contained a strong oxidizing agent (HNO_3 or H_2O_2 with hydrofluoric acid). In addition, some of the solutions contained an inhibitor that has reduced hydrogen pickup in steel during pickling. Small samples of Ti-3Mn-complex-alloy sheet were treated, using various combinations of the descaling and pickling methods. Each sample was then analyzed for hydrogen. Details of these tests and the results of the hydrogen analyses are given in Table 8.

Test 1 indicated that considerable hydrogen was absorbed by the sheet during descaling in the sodium hydride bath. Comparison of Tests 2 and 4 and Tests 3 and 6 confirmed this observation. Hydrogen absorption in the Virgo descaling bath was apparently very slight, several specimens descaled by this method having hydrogen contents between 90 and 100 ppm. These values were very close to the original hydrogen content, namely, 90 ppm.

Of the pickling solutions tested, three showed very low hydrogenation tendencies. They were as follows:

(1) 77 per cent acetic acid, 8 per cent HNO_3, 15 per cent HF

(2) 70 per cent HNO_3, 10 per cent HF, used at 100 F

(3) 35 per cent HNO_3, 5 per cent HF.

The acetic acid solution fumed excessively. Of the two HNO_3-HF solutions, the one containing 70 per cent HNO_3 gave a smoother surface. Some of the other solutions given in Table 8 showed some promise, but further investigation would be required before they could be used generally.

On the basis of this work, it seems that the best procedure for preparing sheet specimens consists of descaling in Virgo salt, followed by pickling in a 70 per cent nitric acid-10 per cent hydrofluoric acid-water solution to remove the contaminated surface layer. The Virgo salt bath has one drawback — it will not remove scale below about 875 F. Thus, the descaling period would have to be considered as part of the aging treatment for sheet age hardened at 800 to 900 F.

Effect of Specimen Geometry and Contaminated Metal Removal on Tensile Properties of the Ti-3Mn-Complex Alloy Sheet

Having established a procedure for removing the scale and the contaminated surface layer of sheet without appreciably increasing its hydrogen content, a check of other variables affecting sheet properties was made. A series of tests was made to determine the effect of a radius on the corners of the gage section of sheet tensile specimens. Earlier work had indicated that

TABLE 8. EFFECTS OF DESCALING AND PICKLING TREATMENTS ON HYDROGEN CONTENT OF Ti-3Mn-COMPLEX-ALLOY SHEET[a]

Test	Descaling Method[b]	Composition of Pickling Agent, volume per cent: HNO_3	CH_3COOH	H_2O_2	HF	Water	Pickling-Bath Temperature, F	Pickling Time, minutes	Amount of Metal Removed, 0.001 inch	Hydrogen Content of Sheet, ppm by weight[c]
1	Sodium hydride	--	--	--	--	--	--	--	--	180
2	Sodium hydride	8	77	--	15	--[d]	135	4	4.5	250
3	Sodium hydride	6	--	--	4	90[d]	140	12	4.0	280
4	Virgo	8	77	--	15	--[d]	150	4	7.0	100[e]
5	Virgo	8	77	--	15	--	130	4	6.0	93[e]
6	Virgo	6	--	--	4	90[d]	135	20	5.5	170
7	Virgo	6	--	--	4	90	130	5	6.0	150
8	Virgo	70	--	--	10	20	100	20	5.5	90
9	Virgo	70	--	--	10	20	120	8	4.5	102
10	Virgo	70	--	--	10	20	120	12	5.5	104
11	Virgo	70	--	--	10	20	160	3	5.0	108[e]
12	Virgo	35	--	--	5	60	170	7.5	5.0	98
13	Virgo	20	--	--	5	75	170	10	5.5	107
14	Virgo	--	--	70	10	20	100	2.4	5.5	119
15	Virgo	--	--	70	10	20	140	0.67	5.0	117
16	Virgo	--	--	35	5	60	120	7	5.0	113
17	Virgo	--	--	20	5	75	120	7	5.0	129
18	Virgo	--	--	6	4	90	160	4	6.0	235

(a) Initial hydrogen content — 90 ppm.
(b) Specimens held for 10 minutes at 750 F in the sodium hydride bath or 30 minutes at 885 F in the Virgo bath and water quenched.
(c) By vacuum-fusion analyses.
(d) Also contained about 2 grams of Rochelle salts per 100 cc of solution.
(e) These pickling baths fumed excessively.

this variable might affect properties considerably. Various radii were obtained on duplicate heat-treated specimens of the Ti-3Mn-complex alloy by filing, belt sanding, and pickling. Radii of 0.003, 0.015, and 0.030 inch were obtained by the first two methods. The radius obtained by normal pickling was about 0.003 inch. The tensile results obtained on these specimens are given in Table 9. The strengths given were not corrected for the reduced area caused by increasing the radii. There was no significant difference in the ductilities of specimens having radii produced by the various methods.

TABLE 9. EFFECT OF GAGE-SECTION RADIUS ON TENSILE DUCTILITY OF SHEET SPECIMENS OF Ti-3Mn-COMPLEX ALLOY(a)

Gage-Section Radius (b)	Elongation(c), per cent in 1 inch(d)	Ultimate(c) Tensile Strength, psi (d)
Deburred by filing	11.0	180,000
0.015-inch radius by filing	11.5	174,500
0.030-inch radius by filing	10.0	168,000
Deburred by belt sanding	11.5	177,500
0.015-inch radius by belt sanding	11.0	174,500
0.030-inch radius by belt sanding	9.0	169,000
As pickled after machining	12.5	180,000

(a) Heat C1 rolled at 1350 F.
(b) All filed and belt-sanded radii were finished with 240-grit paper in the longitudinal direction.
(c) Solution treated 1/2 hour at 1300 F, cold-water quenched, and aged 8 hours at 900 F.
(d) Average of two values.

Several other variables of specimen design and condition were evaluated in the next experiment:

(1) Section size
    (a) Sheet thickness
    (b) Width and length of gage section

(2) Directionality

(3) Surface condition (presence of rolling or heat-treating scale).

These tests were made on single heat-treated specimens of the Ti-3Mn-complex alloy. The data for the effects of section size and directionality are given in Table 10. The results of the tests for the effect of surface condition are given in Table 11.

TABLE 10. EFFECTS OF SECTION SIZE AND DIRECTIONALITY ON TENSILE DUCTILITY OF SHEET SPECIMENS OF THE Ti-3Mn-COMPLEX ALLOY[a]

Specimen Size: Nominal Thickness, 0.001 inch	Specimen Size: Nominal Size of Gage Section, inches	Orientation of Testing Direction[b]	Elongation, per cent in nominal gage length	Ultimate Tensile Strength, psi
Solution Treated 1 Hour at 1300 F, Cold-Water Quenched, and Aged 8 Hours at 900 F[c]				
40	1/4 x 1	L	13.0	186,000
40	1/4 x 1	T	13.0	182,000
40	1/2 x 2	L	12.0	178,000
40	1/2 x 2	T	9.5	185,500
65	1/4 x 1	L	13.5	181,000
65	1/4 x 1	T	13.0	186,000
65	1/2 x 2	L	16.5	178,500
65	1/2 x 2	T	15.5	184,000
Solution Treated 1 Hour at 1300 F, Cold-Water Quenched, and Aged 8 Hours at 1100 F[c]				
40	1/4 x 1	L	23.0	154,500
40	1/4 x 1	T	20.0	153,500
40	1/2 x 2	L	20.5	152,500
40	1/2 x 2	T	23.0	151,000
65	1/4 x 1	L	21.5	149,000
65	1/4 x 1	T	24.0	152,500
65	1/2 x 2	L	20.5	149,500
65	1/2 x 2	T	22.0	152,500

(a) Heat C1 rolled at 1350 F.
(b) L - Parallel to rolling direction
T - Transverse to rolling direction
(c) Descaled in a Virgo salt bath and pickled in 70 per cent HNO_3-10 per cent HF-20 per cent water solution to remove 0.002 inch from each surface before and after heat treating.

TABLE 11. EFFECT OF SCALE ON TENSILE DUCTILITY OF SHEET SPECIMENS OF THE Ti-3Mn-COMPLEX ALLOY[a]

Surface Treatment After Heat Treatment[c]	Specimen Size[d]	Heat Treated With Hot-Rolling Scale Removed[b]: Elongation, per cent in nominal gage length	Heat Treated With Hot-Rolling Scale Removed[b]: Ultimate Tensile Strength, psi	Heat Treated With Hot-Rolling Scale on Specimens: Elongation, per cent in nominal gage length	Heat Treated With Hot-Rolling Scale on Specimens: Ultimate Tensile Strength, psi
Solution Treated 1 Hour at 1300 F, Cold-Water Quenched, and Aged 8 Hours at 900 F					
None	A	9.0	203,000	10.0	184,500
S	A	13.0	186,000	13.0	171,000
M	A	13.0	174,000		
None	B	15.0	184,000	9.5	182,000
S	B	12.0	178,000	15.0	183,000
M	B	10.5	175,500		
None	C	11.5	189,000	7.0	179,500
S	C	13.5	181,000	12.5	180,000
M	C	14.5	179,000		
None	D	15.0	184,000	7.5	209,000
S	D	16.5	178,500	15.0	180,500
M	D	12.0	176,000		
Solution Treated 1 Hour at 1300 F, Cold-Water Quenched, and Aged 8 Hours at 1100 F					
None	A	13.0	156,000	12.5	153,000
S	A	23.0	154,500	18.0	159,000
M	A	20.0	152,500		
None	B	21.5	154,000	12.0	150,500
S	B	20.5	152,500	20.0	152,500
M	B	18.5	148,500		
None	C	19.0	151,500	19.0	149,500
S	C	21.5	149,000	23.0	150,500
M	C	24.0	148,500		
None	D	20.0	150,500	17.5	147,500
S	D	20.5	149,500	23.0	154,500
M	D	20.5	147,500		

(a) Heat C1 rolled at 1350 F.

(b) Descaled in Virgo salt bath and pickled in 70 per cent HNO_3-10 per cent HF-20 per cent water solution.

(c) S - Descaled and pickled as in (b)
M - Descaled mechanically by vapor blasting and pickled as in (b)
None - Sharp corner of gage section removed by hand with 240-grit sandpaper.

(d) A - 0.040-inch-thick sheet with 1/4 x 1-inch gage section
B - 0.040-inch-thick sheet with 1/2 x 2-inch gage section
C - 0.065-inch-thick sheet with 1/4 x 1-inch gage section
D - 0.065-inch-thick sheet with 1/2 x 2-inch gage section.

Section size, that is, the width and length of the gage section of the test specimen, had essentially no effect on tensile elongation. However, elongation did vary somewhat with sheet thickness. The thicker (0.065 inch) sheet had, generally, slightly more elongation than the 0.040-inch sheet. Thus, it appears that sheet thickness, rather than the size of the specimen used, was the controlling factor in determining ductility in these tensile tests. Likewise, there was no conclusive evidence of directionality in the sheet.

The necessity for removing the scale and the contaminated surface metal to obtain maximum tensile elongation is confirmed by the data in Table 11. A comparison of specimens salt-bath descaled (S) or mechanically descaled (M) showed no significant change in tensile elongation. Likewise, specimens descaled and pickled before and after heat treatment or just after showed no difference in ductility, but specimens tested with scale intact from heat treatment or hot rolling had lower ductility than specimens descaled and pickled.

The results of the sheet-preparation evaluation may be summarized as follows:

(1) Excessive hydrogen pickup may be prevented during descaling of titanium sheet by descaling mechanically (vapor blasting) or chemically (Virgo salt bath), followed by pickling in a solution containing a high percentage of nitric acid. A nitric acid-hydrofluoric acid ratio of 7:1 is satisfactory. The scale and contaminated layer of metal incurred during rolling and heat treating must be removed.

(2) Specimen geometry in low-hydrogen sheet has little effect on strength or ductility.

With this information in mind, the sheet-evaluation program described in the following section was undertaken.

Properties of Heat-Treated Sheet Alloys

Several of the alloys evaluated as bar stock in the previous sections of this report were evaluated as 0.06-inch-thick sheet. Where a similar range of properties was obtained on bar stock for two or more alloys, only one alloy was tested as sheet. The forging of the alloy to sheet bar was discussed in the section on "Alloy Selection, Melting, and Fabrication". These sheet bars were finish rolled in the alpha-beta-phase region to about 14-gage sheet. Longitudinal 1/2-inch-wide tensile blanks and 1-1/8-inch-wide bend blanks were heat treated to various strength levels. After heat treatment and machining, the specimens were treated in a Virgo descaling bath for about 40 minutes at about 880 F. This treatment was followed by etching in a 70 per cent concentrated nitric acid-10 per cent concentrated hydrofluoric acid-20 per cent water solution. A minimum of 0.004 inch of thickness was removed by the pickling solution. Tensile tests were made on a Baldwin-Southwark Universal Testing Machine, using a head speed of 0.02 inch per minute to fracture. The guided-bend tests were made by bending a 1-inch-wide specimen into a 105-degree sharp-cornered V block with various mating V punches having specified nose radii. The nose radii are in 1/64-inch increments up to 1/16 inch and then in various increments up to a 1-inch radius. A specimen is bent over successively smaller radii until a crack is visible in the tension side of the bend. The T value is equal to the smallest "no crack" radius divided by the thickness of the sheet specimen being tested.

The tensile and bend properties of the alloy sheet as heat treated are given in Table 12. The tensile elongation of each alloy is plotted as a function of strength in Figure 12. In general, the strength-ductility relationship for all of the alloys was good, even though the elongation values were 6 to 8 per cent lower than for the same alloy bar stock. However, it is evident from Figure 12 that the strength-ductility relationship of the manganese-complex alloys was somewhat better than that of the other alloys tested. Although the sheet material was not heat treated to the highest strength condition, extrapolation of the curve for the two manganese-complex alloys in Figure 12 gives a strength of 180,000 psi with over 10 per cent elongation in 1 inch. These complex alloys were heat treated to a low strength of about 130,000 psi with over 20 per cent elongation.

The bend tests were made to provide an order-of-merit evaluation of these alloys for formability. The alloys with the smaller bend radii should be more formable, but a satisfactory bend-test result does not insure that an alloy will form readily into an actual part. All of the alloys could be heat treated to give a minimum bend radius of about 1 T. One thing in particular should be noted about the bend-radii values: in certain cases, there was an indication that the lowest strength condition did not produce the smallest T value. This may be important when the formability of the alloys is evaluated, as the softest condition generally allows for the greatest deformation.

Weldability

No attempt was made in this year's work to develop a weldable sheet alloy. However, it had been reported previously that a Ti-5V alloy made with high-purity vanadium was weldable, i.e., it had good ductility as welded. Therefore, a few welds were made on the Ti-8V alloy sheet. The welding was done parallel to the direction of rolling. Welds were made

TABLE 12. TENSILE PROPERTIES AND MINIMUM BEND RADII OF 14-GAGE TITANIUM SHEET ALLOYS CONTAINING 4 TO 7 PER CENT BETA STABILIZERS

Heat No. and Nominal Composition, weight per cent	Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F	Minimum Bend Radius, T [b,c]	Elongation, per cent in 1 inch[c]	Ultimate Tensile Strength, psi[c]
WV1 3Mn-1Fe-1V-1Cr-1Mo	1300	24	900	0.7	15.5	153,500
		8	1100	0.7	20.0	133,000
	1375	24	900	0.9	15.5	165,000
		8	1100	1.0	21.0	137,500
	1450	24	900	1.6	13.0	174,000
		8	1100	1.0	22.5	143,500
WV2 3Mn-1Mo-1V	1400	24	900	1.1	15.5[d]	137,500
		8	1100	1.1	19.0	120,000
	1500	24	900	1.4	14.5	147,000
		8	1100	1.4	20.5	127,000
WV4 2Mn-1Fe-1V-1Cr-1Mo	1375	24	900	1.1	17.5[d]	152,000
		8	1100	1.3	23.0	133,000
	1475	24	900	2.4	13.0	166,000
		8	1100	0.8	18.5	139,500
WV6 2Mn-1Mo-1V	1425	24	900	1.1	16.0	128,500
		8	1100	0.9	22.5	117,500
	1525	24	900	2.1	11.5	149,500
		8	1100	1.2	20.0	123,000
WW8 5Mn-2Mo	1300	24	900	1.4	13.0	141,000
		8	1100	1.0	22.5	125,000
	1400	24	900	1.4	14.0	158,500
		8	1100	0.8	19.5	130,500
WW10 5Mn-2V	1300	24	900	1.0	14.0	142,500
		8	1100	0.8	22.5[d]	127,000
	1400	24	900	1.2	14.5	154,500
		8	1100	0.7	22.0	131,000
WW11 2Mn-5V	1350	24	900	0.7	19.5	140,000
		8	1100	0.9	23.0	125,500
	1450	24	900	0.4	15.5	148,500
		8	1100	0.4	19.0	128,000
WV12 8V	1400	24	900	1.0	14.0	147,500
		8	1100	0.6[d]	19.5	122,500
	1600	24	900	13.4[d]	6.0	171,500
		8	1100	8.8[d]	11.5	142,500

TABLE 12. (Continued)

Heat No. and Nominal Composition, weight per cent	Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F	Minimum Bend Radius, T[b,c]	Elongation, per cent in 1 inch[c]	Ultimate Tensile Strength, psi[c]
WW20	1325	24	900	3.2	13.0	149,500
3Mn-1Fe-1V-1Cr-		8	1100	0.7	23.5	128,000
1Mo-0.1Th	1425	24	900	5.0	8.5	172,000
		8	1100	0.7	20.5	136,500
WW21	1325	24	900	1.5	16.0	149,500
3Mn-1Fe-1V-1Cr-		8	1100	1.3	24.0	127,500
1Mo-0.25Th	1425	24	900	4.8	14.0[d]	170,500
		8	1100	1.0	20.0	133,500

(a) Cold-water quenched after 1/2 hour in a purified argon atmosphere.

(b) T = smallest successful bend radius divided by the thickness of the sheet.

(c) Average of two values, except where noted.

(d) Single values.

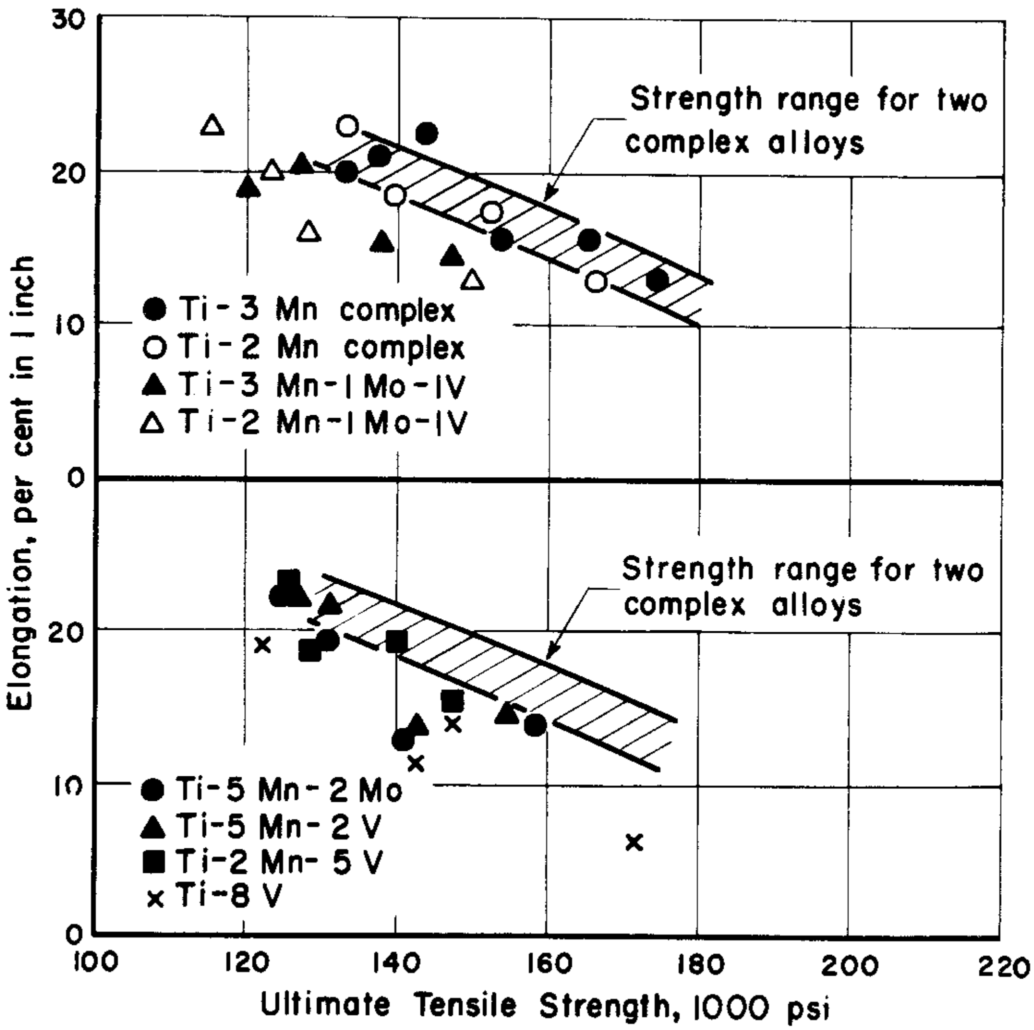


FIGURE 12. TENSILE STRENGTH VERSUS ELONGATION OF HEAT-TREATED BETA-STABILIZED TITANIUM SHEET ALLOYS

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manually in an inert-atmosphere welding chamber at a speed of approximately 4 inches per minute at 60 amperes.

Guided-bend tests were made on the specimens in the as-welded and in three heat-treated conditions, as indicated in Table 13. Solution treating at 1400 F and aging at 1000 F produced the best ductility. All other conditions were extremely brittle. The minimum bend radius of Specimen 3 given the above treatment was 8.3 T, whereas, for the other three specimens, the minimum was greater than 25 T. The poor response of this alloy to welding may have been due to the fact that commercial vanadium was used in its preparation. This grade of vanadium contains 6 to 7 per cent of oxygen, which did not seem to affect the tensile ductility adversely, but may have contributed to the low ductility of the weld specimens.

TABLE 13. BEND DUCTILITY OF WELDED Ti-8V ALLOY SHEET

Specimen	Heat Treatment	Minimum Bend Radius, T[a]
1	As welded	> 25 T
2	Aged 8 hours at 1000 F	> 25 T
3	Solution treated at 1400 F and aged 8 hours at 1000 F	8.3 T
4	Solution treated at 1600 F and aged 8 hours at 1000 F	> 25 T

(a) T = smallest successful bend radius divided by the thickness of the sheet.

EFFECTS OF HYDROGEN IN THE Ti-3Mn-COMPLEX ALLOY

During the past year, considerable emphasis was placed on the evaluation of the effects of hydrogen in an alpha-beta titanium alloy. The Ti-3Mn-complex alloy is representative of this type of alloy and, therefore, the effect of normal hydrogen contents on its tensile properties was evaluated. This evaluation supplements the work reported by WADC in the past year.

Prior to the program to be discussed here, two effects of hydrogen had been noted in routine testing work. The first was with Ti-3Mn-complex alloy sheet material and was fully described in the section on "Evaluation of Selected Alloys as Sheet".

The second problem traced to hydrogen involved tensile data for bar-stock material of the Ti-3Mn-complex alloy from a large ingot melted by the United States Bureau of Mines. A part of this ingot was rolled by a commercial mill to 7/8-inch-diameter bar stock. The commercial fabricator obtained good tensile ductility in tests of this material, whereas tests at Battelle showed low tensile elongation. A check of testing procedures at the two laboratories indicated the only difference to be in the rates of straining during testing. To evaluate the effect of the strain rate on properties, several bars cut from adjacent locations were heat treated and tested at three laboratories. The results of these tests are given in Table 14. It may be seen that the three groups of specimens varied considerably in tensile strength, probably because of unintentional variations in the heat-treatment procedure. The most significant feature of these data, however, is the wide variation in ductility with changing strain rates in the first group of specimens shown in the table. The strain rate was not varied on the second specimen group, and the results of the third group showed a maximum ductility at the intermediate strain rate. The same behavior occurred in later tests and will be discussed in another section in this report.

In the initial evaluation of the cause of the differences in ductility, examination of the microstructure indicated that this 7/8-inch-diameter bar was rolled in the beta-phase region. That is, it consisted of coarse, equiaxed-beta grains with grain-boundary and Widmanstätten alpha. Therefore, lengths of this material were rerolled to 1/2-inch-diameter bars at 1375 or 1450 F. A comparison of the tensile properties of 1/2-inch bar stock rerolled from the 7/8-inch-diameter bar stock and 1/2-inch bar stock rolled at Battelle from a top section of the same original ingot is given in Table 15. The Battelle-fabricated bar from the top of the ingot had much better ductility than the rerolled bar stock at intermediate and high strength levels. The better ductility was not a direct result of the fabrication itself, but was due to certain processing steps used during the commercial rolling, namely, pickling used to improve the surface quality of the bars. The effect of this cleanup method was found when hydrogen analyses were made of the two materials. The commercially rolled material had about 200 ppm by weight of hydrogen, whereas the material rolled at Battelle from the top section of the ingot had only about 50 ppm of hydrogen. The results of these tests indicated that the hydrogen content of the Ti-3Mn-complex alloy could affect the tensile ductility adversely, at least in some conditions of heat treatment. Therefore, the research described in this section of the report was initiated to determine the relationships between hydrogen content, strain rate, heat treatment, and properties of this alloy.

TABLE 14. EFFECT OF STRAIN RATE ON TENSILE PROPERTIES OF HEAT C1[a] OF THE Ti-3Mn-COMPLEX ALLOY ROLLED IN THE BETA FIELD, SOLUTION TREATED AT 1300 F, COLD-WATER QUENCHED, AND AGED 8 HOURS AT 900 F

Test	Hot-Rolled Bar	Strain Rates, inch/minute[b] To Yield	Strain Rates, inch/minute[b] Beyond Yield	Elongation, per cent in 2 inches	Reduction of Area, per cent	Yield Strength, 0.2 Per Cent Offset, psi	Ultimate Tensile Strength, psi
1[c]	2	0.01	0.01	5.0	8.5	148,500	155,500
2	3	0.01	0.02	12.5	17.0	146,000	153,000
3	4	0.01	0.1	15.0	44.0	146,500	155,500
4	5	0.01	0.1	14.0	42.5	149,500	158,000
5[d]	2	0.01[e]	0.1	12.5	--	157,500	170,500
6	3	0.01[e]	0.1	12.5	--	159,000	172,000
7	4	0.01[e]	0.1	11.5	--	162,000	174,500
8	5	0.01[e]	0.1	9.0	--	161,000	174,500
9[f]	3	0.02	0.02	3.5	--	--	183,000
10	5	0.02	0.02	4.0	--	--	178,000
11	2	0.02	0.1	7.0	9.0	165,000	180,500
12	3	0.02	0.1	6.5	10.5	167,500	182,500
13	4	0.02	0.2	5.5	10.0	170,000	186,500
14	5	0.02	0.2	2.5	4.3	175,000	190,000

(a) Melted by the United States Bureau of Mines.
(b) Crosshead speed for 2-inch gage lengths, unless otherwise noted.
(c) Tests 1 to 4 made by Materials Laboratory, Wright Air Development Center.
(d) Tests 5 to 8 made by Mallory-Sharon Titanium Company.
(e) True strain rate, in inch per inch per minute, obtained from strain pacer.
(f) Tests 9 to 14 made by Battelle.

TABLE 15. TENSILE PROPERTIES AND HARDNESSES OF HEAT C1[a] OF THE Ti-3Mn-COMPLEX ALLOY

Rolling Temperature, F	Solution Temperature[b], F	Aging Time, hours	Aging Temperature, F	Elongation, per cent in 1 inch	Reduction of Area, per cent	Yield Strength, 0.2 Per Cent Offset, psi	Ultimate Tensile Strength, psi	VHN, (10-Kg Load)
Rerolled[c] to 1/2-Inch-Diameter Bar Stock From Commercially Rolled 7/8-Inch-Diameter Stock								
1375	1300	8	1100	32	59	--	144,000	--
1375	1300	8	1100	30	57	144,000	144,500	327
1375	1300	8	900	3	9	--	177,000	--
1375	1300	8	900	4	7	173,500	176,000	389
1375	1400	24	800	0	0	--	222,000	--
1375	1400	24	800	0	0	--	210,500	447
1450	1300	8	1100	30	61	--	144,500	--
1450	1300	8	1100	28	62	143,000	144,000	336
1450	1300	8	900	4	7.5	--	178,000	--
1450	1300	8	900	7	8.5	176,500	179,500	387
1450	1400	24	800	0	1	--	214,500	--
1450	1400	24	800	0	1	--	226,500	464
Fabricated[d] at Battelle From Section of Top of Ingot								
1450	1300	8	1100	31	54	--	145,000	--
1450	1300	8	1100	30	56	136,500	146,500	331
1450	1300	8	900	20	40	--	170,500	--
1450	1300	8	900	21	45	145,000	168,000	369
1450	1400	4	1000	21	42	--	168,000	--
1450	1400	4	1000	21	45	--	169,500	360
1450	1400	24	800	5	10	--	218,500	--
1450	1400	24	800	2	2.5	220,000	220,100	424

(a) Melted by United States Bureau of Mines.
(b) Cold-water quenched after 1 hour at temperature.
(c) Rerolled from 7/8-inch to 1/2-inch round at Battelle.
(d) Forged to 3/4-inch square and then rolled to 1/2-inch round.

Effect of Hydrogen on Annealed Bar Stock

The contemplated program to evaluate the effect of hydrogen required that a new, larger Sieverts apparatus be constructed. While this work was in progress, a limited program to evaluate the effect of hydrogen on the properties of annealed Ti-3Mn-complex-alloy bar stock was initiated. This work utilized a smaller Sieverts apparatus already available. The range of hydrogen contents used in this investigation was somewhat greater than that employed in the main part of the program.

Hydrogenation and Fabrication

The material for this investigation was 5/8-inch-diameter hot-rolled bar from the commercial heat used throughout this investigation. This rod was descaled by machining and cut into 4-inch lengths for hydrogenation or vacuum annealing. Vacuum annealing was done at 1470 F for 5-1/2 hours. Hydrogenation was done at 1470 F for 30 minutes, followed by furnace cooling to 1250 F for 1 hour to equilibrate, and then cooling in vacuum. Most of the vacuum-annealed or hydrogenated 5/8-inch rods were swaged at 1450 or 1600 F to 1/4-inch diameter. As the work progressed, it was found that the higher hydrogen-content material had a beta transus below 1450 F. Therefore, some additional high-hydrogen material was worked at 1300 F to 3/8-inch-diameter rod and finished to 1/4-inch-diameter rod at 1200 F. The rods swaged at 1450 and 1600 F were then annealed 1 hour at 1300 F, furnace cooled to 1100 F, held 15 minutes, and then air cooled. The rods swaged at 1200 F were stabilized by holding at 1200 F for 5-1/2 hours, furnace cooling to 1100 F, holding for 1/2 hour, and air cooling.

Hydrogen levels for the various rods were:

	Hydrogen Content, ppm by weight				
Rods swaged at 1200 F		40[a],	390,	690	
Rods swaged at 1450 F	36[a],	160,	260,	380,	690
Rods swaged at 1600 F	78[a],	170,	260,	370,	690

(a) Vacuum-fusion analyses; all other values based on residual plus Sieverts addition.

Mechanical Properties and Microstructures

Substandard tensile specimens having a 0.125-inch diameter by 1/2-inch gage section were machined from the fabricated and annealed rods. The specimens were tensile tested at a head speed of 0.005 inch per minute to fracture. The results of these tests are given in Table 16. The tensile

TABLE 16. EFFECT OF HYDROGEN ON TENSILE PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY AS STABILIZED AFTER VARIOUS HOT-WORKING TEMPERATURES

Hydrogen Content, ppm by weight	Elongation, per cent in 1/2 inch[(a)]	Reduction of Area, per cent[(a)]	Yield Strength, psi[(a)]	Ultimate Tensile Strength, psi[(a)]	VHN (10-Kg Load)
		Swaged at 1200 F			
40[(b)]	36[(c)]	67[(c)]	82,000[(c)]	115,000[(c)]	--
390	19	38	103,000	116,000	--
690	15	17	114,000	122,000	--
		Swaged at 1450 F			
36[(b)]	28	56	113,000	124,000	276
160	27	45	119,000	128,000	299
260	24	34	124,000	131,000	297
380	20	22	122,000	128,000	297
690	7	11	121,000[(d)]	122,000	294
		Swaged at 1600 F			
78[(b)]	19	32	112,000	121,000	292
170	16	34	111,000	120,000	285
260	10	21	118,000[(d)]	123,000	297
370	11	18	117,000	121,000	285
690	8	12	115,000	122,000	302

(a) Average of two values, except where noted.

(b) Determined by vacuum-fusion analyses; other values nominal additions.

(c) Single values.

(d) Yield strength at 0.2 per cent offset.

strength, elongation, and reduction of area are also plotted as functions of the hydrogen content in Figure 13. It is readily seen that the tensile ductility for all conditions of fabrication markedly decreased with increasing hydrogen content.

The fabrication temperature also had an effect on the properties at any given hydrogen level. Swaging at 1200 F produced lower strength and greater ductility than swaging at higher temperatures. It may be noted that the strength and ductility of specimens containing 690 ppm of hydrogen were the same after swaging at 1450 F or at 1600 F. An examination of the microstructures for both conditions showed a transformed-beta structure typical of material fabricated in the beta-phase region, as shown in Figure 14. This structure in material hot worked at 1450 F indicates a marked lowering of the beta transus by hydrogen, since the vacuum-annealed structure of the material swaged at 1450 F was equiaxed alpha-beta (Figure 16). The structure of the material swaged at 1450 F ranged from the equiaxed structure to the Widmanstätten type as the hydrogen content increased from low to high levels. The mixed structure found at intermediate hydrogen levels is shown in Figure 15.

Thus, hydrogen in this alloy decreased the ductility by two separate mechanisms. An increase in hydrogen content decreased the ductility in specimens having the same microstructures. The hydrogen had the additional effect of lowering the beta transus, so that a transformed-beta structure was obtained at some hydrogen level, even when specimens were hot worked at as low as 1450 F. A large-grained transformed-beta structure of the type shown in Figure 14 is known to have intrinsically lower ductility than a fine-grained equiaxed alpha-beta structure.

This work conclusively demonstrated that hydrogen in amounts as low as 260 ppm reduces the ductility of the Ti-3Mn-complex alloy in the annealed condition. The next section of the report describes a more extensive investigation of the interrelations between hydrogen content, heat treatment, strain rate, and properties of the alloy.

Effects of Hydrogen Content, Heat Treatment, and Strain Rate on Properties

Preparation of Material

This phase of the program to determine the effects of hydrogen on the properties of the Ti-3Mn-complex alloy was quite extensive. To evaluate completely all of the selected effects required about 100 pounds of titanium alloy. To meet the large materials requirement of this phase of the hydrogen investigation, part of a single commercial heat of the Ti-3Mn-complex alloy was purchased. One large heat would be more homogeneous than would

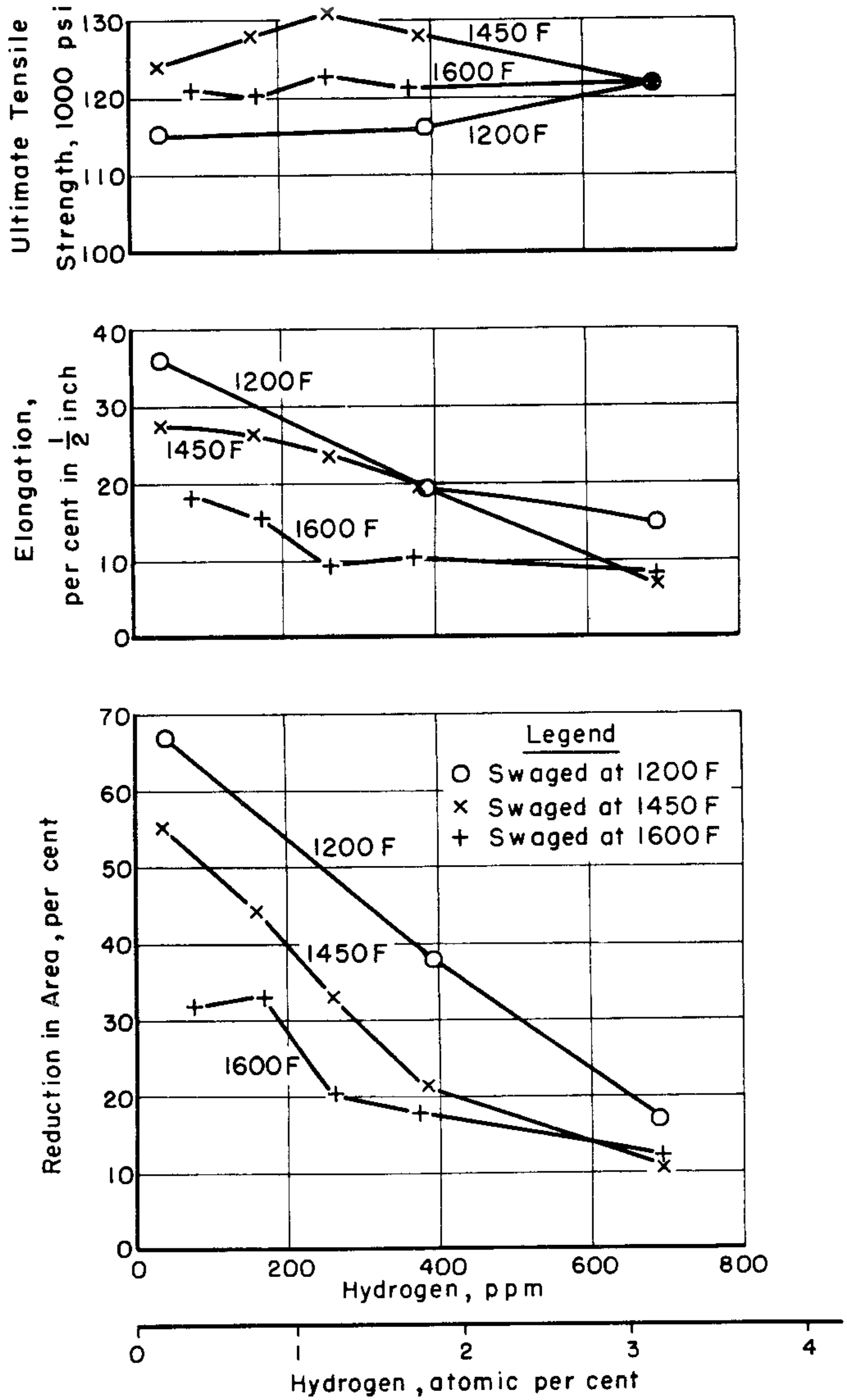
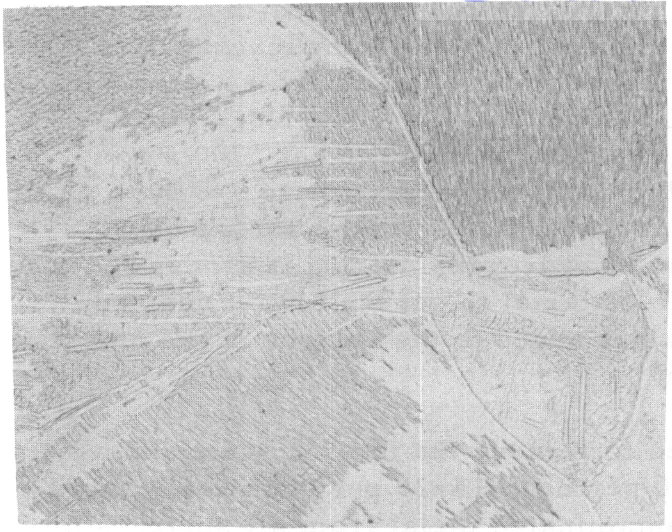


FIGURE 13. SLOW-SPEED ROOM-TEMPERATURE TENSILE PROPERTIES OF ANNEALED Ti-3Mn-COMPLEX-ALLOY SWAGED RODS AT VARIOUS HYDROGEN LEVELS

A-13489

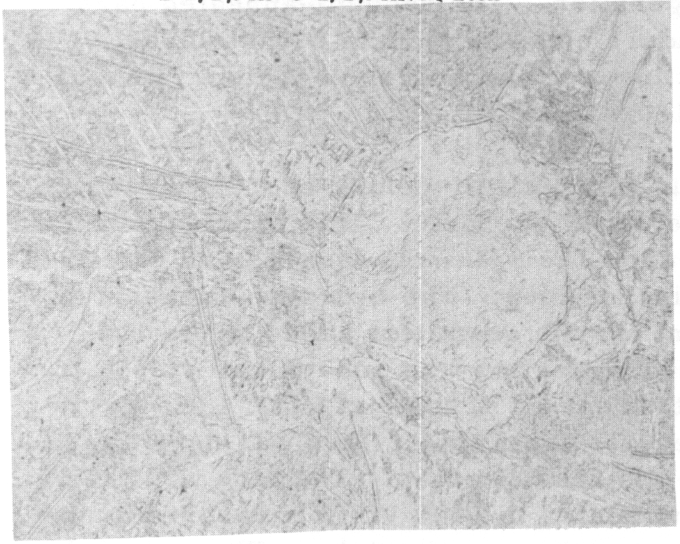


500X N17016

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 14. Ti-3Mn-COMPLEX ALLOY WITH 690 PPM OF HYDROGEN, SWAGED AT 1450 F AND STABILIZED

Structure: Grain-boundary and Widmanstätten alpha, typical of alloy cooled slowly from beta field.

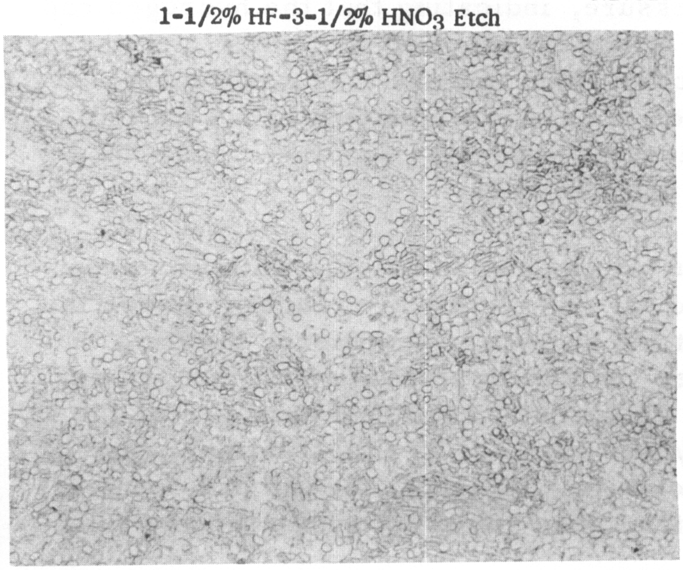


500X N17014

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 15. Ti-3Mn-COMPLEX ALLOY WITH 260 PPM OF HYDROGEN, SWAGED AT 1450 F AND STABILIZED

Structure: Mixed structure containing some Widmanstätten alpha and grain-boundary alpha, plus some spheroidal primary alpha in beta matrix, typical of alloy hot worked very close to the beta transus.



500X N17012

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 16. Ti-3Mn-COMPLEX ALLOY WITH 36 PPM OF HYDROGEN, SWAGED AT 1450 F AND STABILIZED

Structure: Random equiaxed alpha in beta matrix, typical of alloy hot worked in the alpha-beta field.

several small heats. The alloy was purchased as 4-1/2-inch-diameter forged and machined bar stock. A composite sample from several bars was analyzed for alloy additions and interstitials. The analyses, in weight per cent, were: Mn-3.36, Fe-0.91, Cr-1.18, Mo-1.08, V-0.98, C-0.02, N_2-0.008, O_2-0.09, and H_2-0.005. The bars were forged at 1700 F to 3/4-inch-square bars for rolling to 1/2-inch-round bars or to 5/8 by 1-1/2-inch-sheet bars for rolling to 14-gage sheet. These forged bars, as noted above, contained about 50 ppm of hydrogen, making it necessary to vacuum anneal about one-fourth of the material to get the lowest desired hydrogen level of 30 ppm. Hydrogen was added to the balance of the stock to get levels of 90, 140, and 260 ppm.

The forged bars were grit blasted to remove the forging scale and were then pickled in a 70 per cent HNO_3-10 per cent HF-20 per cent water solution to remove about 0.002 inch of contaminated metal from all surfaces before vacuum annealing or hydrogenation. This treatment also produced a clean surface satisfactory for hydrogenation. The vacuum-annealed bars were heated to 1400 F for about 24 hours under a vacuum down to about 0.08 micron of mercury. This treatment lowered the hydrogen content to about 25 ppm, as shown by Analyses 7 to 16 in Table 17.

A new, larger Sieverts apparatus was built to make measured hydrogen additions to about 1.6 pounds of material at one time. This apparatus had a reaction tube large enough to hold four of the 3/4-inch-square bars or two of the 5/8- by 1-1/2-inch bars up to 5 inches long. The hydrogen-measuring volumes were accurate to ±3 per cent for the quantities added to the 1.6 pounds of material in each batch. Hydrogenation was done in two steps, to reduce the over-all time necessary to treat all of the material. The first step was the addition made in the Sieverts apparatus. This was done by putting the bars to be hydrogenated into the reaction tube. The tube was then evacuated to less than 10 microns and heated to 1400 F. A measured quantity of hydrogen was then allowed to enter the reaction tube. The end of the reaction was noted by the drop in pressure, indicating that the hydrogen had been absorbed by the metal charge. When the reaction was complete, the furnace was withdrawn from the reaction tube, and the tube was cooled rapidly with a water spray. The batch of bars was removed when the temperature was about 600 F, and the Sieverts apparatus was then ready for making a hydrogen addition to a new batch of bars.

The second step in hydrogenation was homogenization. This was necessary because the short time needed for completing the reaction leaves the hydrogen quite highly concentrated at the surface. Therefore, the bars that had hydrogen added were sealed under vacuum in separate Vycor capsules. These capsules were then heated for 24 hours at 1400 F to homogenize the hydrogen content. Good homogeneity was obtained by this treatment, as shown by Analyses 28 through 33, inclusive, Table 17, for the end, center, and end of two bars from a single batch. The variation within the two bars

TABLE 17. VACUUM-FUSION HYDROGEN ANALYSES

Analysis	Stage of Material Processing		Hydrogen Content, ppm by weight
1	3/4-inch-square forged bar		43
2	Duplicate		43
3	3/4-inch-square forged bar		48
4	Duplicate		52
5	5/8 x 1-1/2-Inch forged bar		77
6	Duplicate		79
	Vacuum-Annealed Level		
7	3/4-inch-square bar as vacuum annealed		23
8	3/4-inch-square bar as vacuum annealed		9.3
9	5/8 x1-1/2-inch bar as vacuum annealed		17
10	1/2-inch-diameter bar rolled at 1400 F - End		26
11	- Center		32
12	- End		34
13	Gage section of round Test Bar 250		30
14	Gage section of round Test Bar 350		28
15	Gage section of round Test Bar 351		19
16	Grip end of sheet Specimen 13		28
		Average	24.6
	90-PPM Level		
17	1/2-inch-diameter bar stock rolled at 1400 F from Batch 9182-95 - End		78
18	- Center		80
19	- End		80
20	1/2-inch-diameter bar stock rolled at 1400 F from Batch 9182-97 - End		110
21	- Center		100
22	- End		100
23	Gage section of round Test Bar 453 from Batch 9182-96		87
24	Gage section of round Test Bar 373 from Batch 9182-93		78
25	Gage section of round Test Bar 471 from Batch 9182-97		105
26	Gage section of round Test Bar 472 from Batch 9182-97		106
27	Grip end of sheet Specimen 53 from Batch 9642-1		106
		Average	93.6
	140-PPM Level		
28	1/2-inch-diameter bar rolled at 1400 F from Batch 9182-45 - End		123
29	- Center		122
30	- End		120
31	1/2-inch-diameter bar rolled at 1600 F from Batch 9182-45 - End		130
32	- Center		128
33	- End		128

TABLE 17. (Continued)

Analysis	Stage of Material Processing	Hydrogen Content, ppm by weight
	140-PPM Level (Continued)	
34	1/2-inch-diameter bar rolled at 1600 F from Batch 9182-61 - End	148
35	- Center	141
36	- End	153
37	Gage section of round Test Bar 10 from Batch 9182-55	158
38	Gage section of round Test Bar 94 from Batch 9182-63	154
39	Grip end of sheet Specimen 93 from Batch 9182-72	148
40	Grip end of sheet Specimen 113 from Batch 9182-73	168
	Average	140.0
	260-PPM Level	
41	1/2-inch-diameter bar stock rolled at 1400 F from Batch 9182-76 - End	260
42	- Center	260
43	- End	280
44	1/2-inch-diameter bar stock rolled at 1600 F from Batch 9182-81 - End	260
45	- Center	250
46	- End	250
47	Gage section of round Test Bar 121 from Batch 9182-67	241
48	Gage section of round Test Bar 232 from Batch 9182-80	289
49	Gage section of round Test Bar 225 from Batch 9182-82	259
50	Gage section of round Test Bar 204 from Batch 9182-81	260
51	Grip end of sheet Specimen 134 from Batch 9182-87	283
	Average	263

was only 3 ppm, whereas the average contents for the bars were 122 and 129 ppm of hydrogen. This difference of 7 ppm is probably not significant, as the error in analysis may be ±10 relative per cent. It may be further noted that heat treatment after fabrication did not change the hydrogen content. Analyses 20, 21, and 22, Table 17, had a range of 100 to 110 ppm of hydrogen for the rolled bar, and Analyses 25 and 26 had 105 and 106 ppm, respectively, for samples taken from the gage section of heat-treated tensile bars from the same batch of hydrogenated bars.

The 3/4-inch-square bars were rolled at 1400 or 1600 F to 1/2-inch-diameter bar stock, and the rectangular bars were rolled to 14-gage sheet at 1400 F in a direction 90 degrees to the long axis of the forged bar. Tensile blanks 3 inches long from the 1/2-inch-diameter bar stock were then solution treated at 1300 or 1600 F and quenched in cold water. Some of the bars were age hardened to two strength levels, and one set was retained in the as-quenched condition. All heat treatment of bar stock was done in air. The sheet material was cut to blanks about 1 inch wide by about 5 inches long, parallel to the rolling direction. These bars were racked to keep them from touching, so that a uniform quench could be obtained, and solution treated at 1300 F in a purified-argon atmosphere. They were rapidly withdrawn from the argon furnace and quenched in cold water prior to aging. The sheet specimens were tested in two conditions of aging, selected to provide a high strength and a low strength level.

The bar-stock specimens were machined to standard 1/4-inch-diameter test bars. The sheet material was machined to produce a gage section 1/2 inch wide by about 2 inches long in the test specimens. After machining, the sheet specimens that had been aged at 900 F or higher were treated for 40 minutes in a Virgo salt bath at about 880 F. The sheet material aged at 800 F was grit blasted to remove the adherent hot-rolling and heat-treating scale. After the Virgo or vapor-blasting treatment, the sheet specimens were pickled in a 70 per cent nitric acid-10 per cent hydrofluoric acid-20 per cent water pickling solution. A minimum of about 0.004 inch was removed from the thickness of the specimens by pickling.

Bar-Stock Tensile Testing and Evaluation

Bar-stock specimens representing each condition of hydrogen level and heat treatment were tensile tested at the following strain rates:

(1) 0.002 inch per inch per minute to fracture

(2) 0.005 inch per inch per minute to fracture

(3) 0.005 inch per inch per minute to the yield strength, the 0.1 inch per minute to fracture

(4) 0.1 inch per minute to fracture.

The two low strain rates were followed by using a strain pacer, and the high rate was determined by platen speed. In certain cases at the low speeds, where ductility was very low, it was not feasible to use the extensometer, so the strain pacer could not be used. For these specimens, a simulated strain rate was obtained by adjusting the hydraulic valve at increments of load to correspond to settings determined for specimens for which the strain pacer was used. Tests using this technique are noted in the tables. The tensile-test data are given in Tables 18, 19, and 20.

The first effect to note is that hydrogen does not seem to contribute appreciably to beta embrittlement. At least, the low-hydrogen-level material still shows embrittlement or loss of ductility when solution treated or rolled in the beta field. Specimens solution treated and aged to a strength of about 120,000 psi had about 30 per cent elongation when treated in the alpha-beta-phase region. However, the elongation decreased to about 16 per cent for specimens rolled in the beta-phase region and solution treated in the alpha-beta-phase region, with a further decrease in elongation to about 6 per cent for specimens solution treated in the beta-phase region.

The effects of hydrogen content on the tensile properties were similar for these three basic conditions of rolling and solution treating:

A - Rolled and solution treated in the alpha-beta-phase region

B - Rolled in the beta-phase region and solution treated in the alpha-beta-phase region

C - Rolled in the alpha-beta-phase region and solution treated in the beta-phase region.

The strength-ductility data for Condition A are more complete because for all conditions a little ductility was obtained, whereas many of the high-strength or high-hydrogen specimens in the beta-embrittled conditions, B and C, had zero ductility. Therefore, the data from specimens rolled and solution treated in the alpha-beta-phase region have been plotted in Figure 17. In this figure, the reduction of area was plotted as a function of the hydrogen content at constant strain rates. Each set of curves represents one of the following heat-treated conditions:

A - Quenched from 1300 F and aged 48 hours at 800 F. This treatment produced a high strength of about 180,000 psi.

B - As quenched from 1300 F. This treatment produced an intermediate-strength level of about 145,000 psi.

C - Quenched from 1300 F and aged 8 hours at 1100 F. This treatment produced a low strength level of about 120,000 psi.

TABLE 18. EFFECTS OF HYDROGEN CONTENT, HEAT TREATMENT, AND STRAIN RATE ON TENSILE PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY ROLLED IN THE ALPHA-BETA FIELD (1400 F) AND SOLUTION TREATED[(a)] IN THE ALPHA-BETA FIELD (1300 F)

Average Hydrogen Content, ppm by weight	Aging		Strain Rate[(b)]	Elongation, per cent in 1 inch[(c)]	Reduction of Area, per cent[(c)]	Yield Strength, 0.2 Per Cent Offset, psi[(c)]	Ultimate Tensile Strength, psi[(c)]
	Time, hours	Temperature, F					
30	--	--	A[(d)]	19.0	39.5	127,500	144,000
30	--	--	B	22.5	46.0	128,000	144,000
30	--	--	C	21.0	48.5	128,500	142,000
30	--	--	D	20.0	47.5	--	142,000
30	48	800	A[(d)]	9.0	14.0	142,500	174,000
30	48	800	B	11.0	20.0	142,500	175,500
30	48	800	C	10.5	22.5	143,500[(d)]	177,000
30	48	800	D	11.5	22.0	--	176,000
30	8	1100	A[(d)]	27.0	56.5	106,000	121,500
30	8	1100	B	31.0	58.0	107,000	119,500
30	8	1100	C	29.0	56.0	106,500	120,000
30	8	1100	D	29.0	47.5	--	121,500
90	--	--	A[(d)]	16.0	26.0	132,000	148,500
90	--	--	B	19.0	36.0	143,000[(d)]	150,500
90	--	--	C	17.0	31.5	138,000	148,000
90	--	--	D	17.5	43.5	--	146,000
90	48	800	A[(d)]	9.0	14.5	150,000	178,000
90	48	800	B	10.5	19.5	157,000	178,000
90	48	800	C	9.0	23.5	148,000	179,000
90	48	800	D	8.0[(d)]	14.5[(d)]	--	181,500[(d)]
90	8	1100	A[(d)]	30.0	51.0	106,000	120,000
90	8	1100	B	30.0	55.5	107,000	120,000
90	8	1100	C	28.5	56.0	106,000	120,000
90	8	1100	D	28.0	54.5	--	120,500
140	--	--	A[(d)]	13.0	26.0	142,500	158,500
140	--	--	B	12.5	30.5	142,500	156,500
140	--	--	C	13.0	29.5	142,500	157,000
140	--	--	D	12.0	32.0	--	157,000
140	48	800	A[(d)]	2.0	3.5	141,000	175,500
140	48	800	B	2.5	3.5	148,000	178,500
140	48	800	C	3.5	6.0	150,000	180,000
140	48	800	D	3.0	6.0	--	180,500

TABLE 18. (Continued)

Average Hydrogen Content, ppm by weight	Aging Time, hours	Aging Temperature, F	Strain Rate(b)	Elongation, per cent in 1 inch (c)	Reduction of Area, per cent(c)	Yield Strength, 0.2 Per Cent Offset, psi(c)	Ultimate Tensile Strength, psi(c)
140	8	1100	A(d)	25.0	40.0	104,500	119,500
140	8	1100	B	27.0	47.0	104,500	119,500
140	8	1100	C	27.5	50.5	102,500	120,500
140	8	1100	-(e)	25.5	48.0	--	121,000
260	--	--	A(d)	12.0	22.0	150,500	158,500
260	--	--	B	21.0(d)	39.5(d)	141,500(d)	153,500(d)
260	--	--	C	14.5	39.5	146,000	156,000
260	--	--	D	13.0	39.5	--	156,000
260	48	800	A(d)	0.5	1.0	148,500	172,500
260	48	800	B*	--	--	--	174,000(d)
260	48	800	C	--	--	--	--
260	48	800	D	2.0	2.5	--	179,000
260	8	1100	A(d)	15.0	22.5	110,000	121,000
260	8	1100	B	28.0	48.0	109,500	122,000
260	8	1100	C	27.5	49.0	108,500	121,500
260	8	1100	D	23.5	59.0	--	119,500

(a) Cold-water quenched after 1 hour at temperature.

(b) A - 0.002 inch per inch per minute to fracture

B - 0.005 inch per inch per minute to fracture

C - 0.005 inch per inch per minute to yield strength and then 0.1 inch per minute to fracture

D - 0.1 inch per minute to fracture

B* - Simulated 0.005 inch per inch per minute to fracture by adjusting the valve setting according to load.

(c) Average of two values, except where noted.

(d) Single values.

(e) Strain rate of 0.010 inch per inch per minute to fracture.

TABLE 19. EFFECTS OF HYDROGEN CONTENT, HEAT TREATMENT, AND STRAIN RATE ON TENSILE PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY ROLLED IN THE BETA FIELD (1600 F) AND SOLUTION TREATED[a] IN THE ALPHA-BETA FIELD (1300 F)

Average Hydrogen Content, ppm by weight	Aging		Strain Rate[b]	Elongation, per cent in 1 inch[c]	Reduction of Area, per cent[c]	Yield Strength, 0.2 Per Cent Offset, psi[c]	Ultimate Tensile Strength, psi[c]
	Time, hours	Temperature, F					
30	--	--	A	--	--	--	--
30	--	--	B	9.5	14.0	138,500	147,500
30	--	--	C	8.0	14.0	139,500	149,000
30	--	--	D	10.0	17.5	--	150,000
30	24	900	A[d]	5.0	10.0	129,500	143,000
30	24	900	B	7.0	12.5	122,000	141,000
30	24	900	C[d]	7.0	12.0	125,000	144,500
30	24	900	D	4.5	13.0	--	143,000
30	8	1100	A[d]	15.0	20.5	105,500	120,000
30	8	1100	B	17.0	20.0	108,000	121,500
30	8	1100	C	17.5	23.0	106,000	121,500
30	8	1100	D	16.5	20.0	--	122,000
90	--	--	A	--	--	--	--
90	--	--	B	6.0	10.5	144,000	150,000
90	--	--	C	7.0	13.0	138,500	150,000
90	--	--	D	7.5	16.5	--	151,500
90	24	900	A	--	--	--	--
90	24	900	B	5.0	10.0	125,000	146,000
90	24	900	C	8.0	12.5	121,500	144,000
90	24	900	D	14.5	16.0	--	144,500
90	8	1100	A	16.0	25.5	102,000	119,000
90	8	1100	B	18.5	24.0	101,500	120,000
90	8	1100	C	18.0	23.0	103,500	120,500
90	8	1100	D	16.5	27.5	--	119,500
140	--	--	A	--	--	--	--
140	--	--	B	7.5	16.0	140,500[d]	155,500
140	--	--	C	10.0	23.5	137,500	149,000
140	--	--	D	9.0	23.5	150,500[d]	155,000
140	24	900	A	--	--	--	--
140	24	900	B	4.0	6.5	127,000	148,000
140	24	900	C	4.0	8.0	124,500	146,000
140	24	900	D	4.5	8.0	--	147,500

TABLE 19. (Continued)

Average Hydrogen Content, ppm by weight	Aging Time, hours	Aging Temperature, F	Strain Rate(b)	Elongation, per cent in 1 inch(c)	Reduction of Area, per cent(c)	Yield Strength, 0.2 Per Cent Offset, psi(c)	Ultimate Tensile Strength, psi(c)
140	8	1100	A(d)	21.0	24.0	104,500	120,000
140	8	1100	B	21.0	25.0	104,500	119,000
140	8	1100	C	20.0	29.5	105,000	120,000
140	8	1100	D	19.0	31.0	114,000	120,500
260	--	--	A	--	--	--	--
260	--	--	B	5.5	11.5	152,000	158,500
260	--	--	C	8.5	18.0	150,000	160,500
260	--	--	D	6.0	12.5	--	165,500
260	24	900	A	--	--	--	--
260	24	900	B*	0	1.5	--	123,500
260	24	900	C	--	--	--	--
260	24	900	D	1.0	3.0	--	146,500
260	8	1100	A	12.0	19.5	105,500	118,000
260	8	1100	B	12.0	12.5	108,000	122,000
260	8	1100	C	14.0	20.5	108,000	122,500
260	8	1100	D	16.0	21.5	115,000(d)	124,500

(a) Cold-water quenched after 1 hour at temperature.

(b) A - 0.002 inch per inch per minute to fracture
B - 0.005 inch per inch per minute to fracture
C - 0.005 inch per inch per minute to yield strength and then 0.1 inch per minute to fracture
D - 0.1 inch per minute to fracture
B* - Simulated 0.005 inch per inch per minute to fracture by adjusting the valve setting according to the load.

(c) Average of two values, except where noted.

(d) Single values.

TABLE 20. EFFECTS OF HYDROGEN CONTENT, HEAT TREATMENT, AND STRAIN RATE ON TENSILE PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY ROLLED IN THE ALPHA-BETA FIELD (1400 F) AND SOLUTION TREATED[a] IN THE BETA FIELD (1600 F)

Average Hydrogen Content, ppm by weight	Aging Time, hours	Aging Temperature, F	Strain Rate[b]	Elongation, per cent in 1 inch[c]	Reduction of Area, per cent[c]	Yield Strength, 0.2 Per Cent Offset, psi[c]	Ultimate Tensile Strength, psi[c]
30	--	--	A	--	--	--	--
30	--	--	B*	0	--	--	145,000
30	--	--	C	--	--	--	--
30	--	--	D	0	--	--	149,500
30	24	900	A	--	--	--	--
30	24	900	B	3.0	6.0	158,000	165,500
30	24	900	C	2.5	6.0	162,000	172,000
30	24	900	D	2.5	5.0	--	170,000
30	8	1100	A	--	--	--	--
30	8	1100	B	5.5	10.5	106,500	120,000
30	8	1100	C	5.5	11.5	108,000	121,500
30	8	1100	D	7.0	13.0	--	122,500
90	--	--	A	--	--	--	--
90	--	--	B*	0	--	--	146,500
90	--	--	C	--	--	--	--
90	--	--	D	0	--	--	152,000
90	24	900	A	--	--	--	--
90	24	900	B	2.5	5.0	163,000	170,000
90	24	900	C	1.5	4.5	156,000	166,500
90	24	900	D	2.5	6.0	--	168,000
90	8	1100	A	--	--	--	--
90	8	1100	B	6.0	11.0	108,000	120,000
90	8	1100	C	6.5	11.5	106,500	122,500
90	8	1100	D	8.0	12.5	--	123,000
140	--	--	A	--	--	--	--
140	--	--	B*	0	0	--	133,000
140	--	--	C	--	--	--	--
140	--	--	D	--	--	--	142,500
140	24	900	A	--	--	--	--
140	24	900	B*	0.5	2.0	--	177,400
140	24	900	C	--	--	--	--
140	24	900	D	2.5	5.5	172,000[d]	180,500

TABLE 20. (Continued)

Average Hydrogen Content, ppm by weight	Aging Time, hours	Aging Temperature, F	Strain Rate(b)	Elongation, per cent in 1 inch(c)	Reduction of Area, per cent(c)	Yield Strength, 0.2 Per Cent Offset, psi(c)	Ultimate Tensile Strength, psi(c)
140	8	1100	A(d)	7.0	9.0	104,500	118,500
140	8	1100	B	7.0	10.5	105,000	120,000
140	8	1100	C	7.5	12.0	107,500	124,500
140	8	1100	D	6.5	10.5	--	122,500
260	--	--	A	--	--	--	--
260	--	--	B*	--	--	--	141,000
260	--	--	C	--	--	--	--
260	--	--	D	0(d)	--	--	158,500
260	24	900	A	--	--	--	--
260	24	900	B*	0	1.5	--	174,000
260	24	900	C	--	--	--	--
260	24	900	D	1.0	3.0	--	176,500
260	8	1100	A	--	--	--	--
260	8	1100	B	6.0	8.5	103,500	118,500
260	8	1100	C	6.5	9.5	106,500	125,000
260	8	1100	D	5.5	10.5	110,500	122,000

(a) Cold-water quenched after 1 hour at temperature.

(b) A - 0.002 inch per inch per minute to fracture
B - 0.005 inch per inch per minute to fracture
C - 0.005 inch per inch per minute to yield strength and then 0.1 inch per minute to fracture
D - 0.1 inch per minute to fracture
B* - Simulated 0.005 inch per inch per minute to fracture by adjusting the valve setting according to load.

(c) Average of two values, except where noted.

(d) Single values.

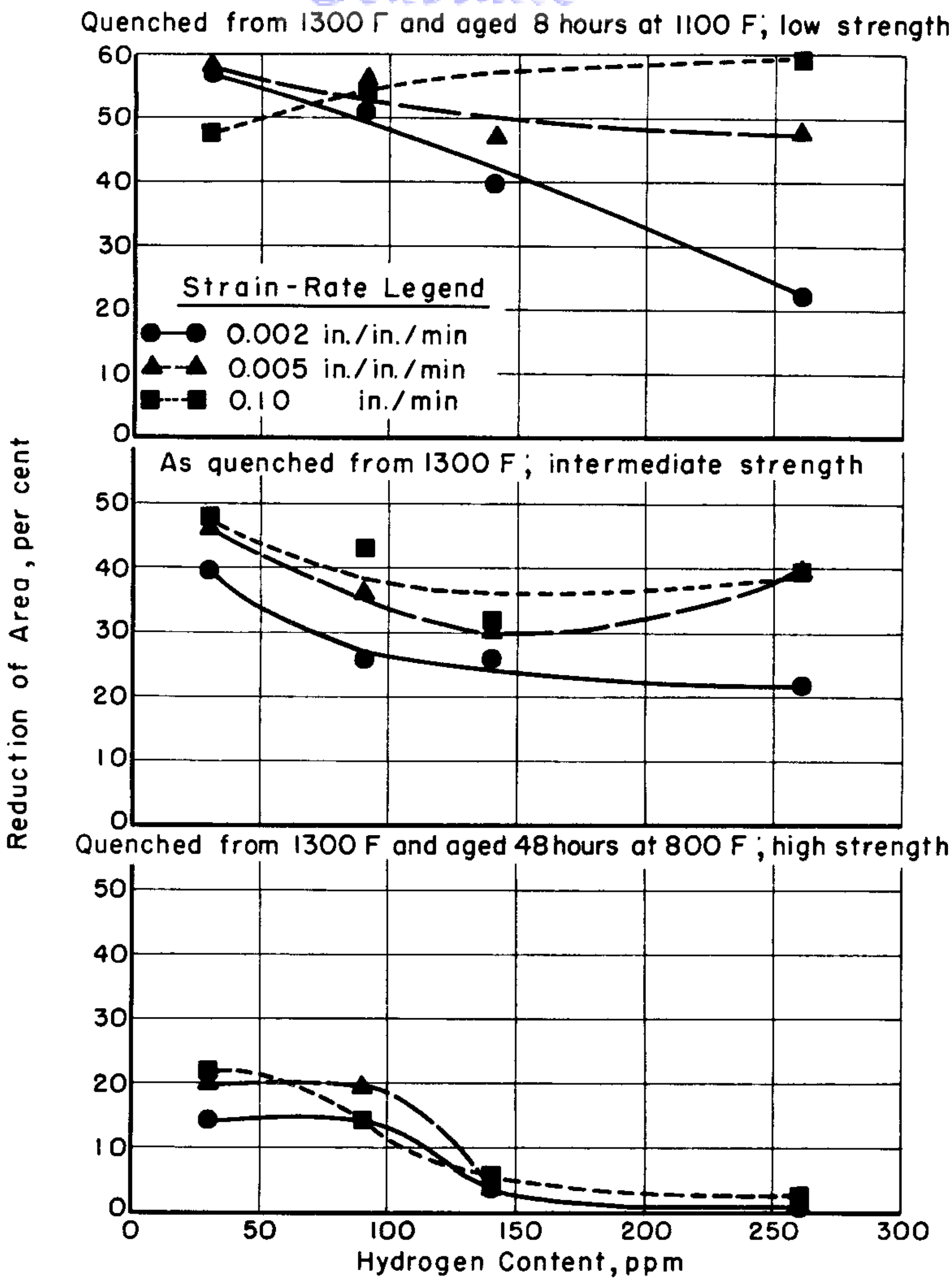


FIGURE 17. EFFECT OF HYDROGEN CONTENT IN THE Ti-3Mn-COMPLEX ALLOY ON TENSILE REDUCTION OF AREA FOR VARIOUS STRAIN RATES

All specimens rolled at 1400 F in the alpha-beta-phase region prior to heat treating.

A-13490

These curves show that the effect of hydrogen on the ductility of the Ti-3Mn-complex alloy varies with the hydrogen content, the strain rate, and the heat treatment. In the high-strength condition, strain rate, per se, had little effect on the ductility of specimens of a given hydrogen content. It is significant, however, that as little as 140 ppm of hydrogen drastically lowered the reduction-of-area values of the high-strength specimens.

In the intermediate and low-strength conditions, strain-rate effects were more pronounced, as may be seen by the spread between the curves in Figure 17. The general trend was for the ductility to increase somewhat as the strain rate increased from 0.002 inch per inch per minute to 0.1 inch per minute. At the 260-ppm hydrogen level, this increase in ductility with strain rate was rather pronounced. The specimens containing 260 ppm of hydrogen for the low and intermediate strengths and for the two higher strain rates exhibit ductilities equal to or greater than that obtained for the specimens containing 140 ppm of hydrogen. No reason for this behavior is apparent, but it has also been shown for certain alloys by Lenning, Craighead, and Jaffee*. In their work, a minimum in the curves of reduction of area versus hydrogen content was shown for 120 ppm of hydrogen for a testing speed of 0.005 inch per minute and a test temperature of 100 C. Minimums were also shown for a testing speed of 0.5 inch per minute at 120 and 170 ppm of hydrogen for testing temperatures of 100 and -40 C, respectively.

Effect of Hydrogen on the Room-Temperature Stress-Rupture Properties of the Ti-3Mn-Complex Alloy

Room-temperature notched-stress-rupture testing was used to evaluate further the embrittlement associated with higher levels of hydrogen in the Ti-3Mn-complex alloy. Notched bars usually impose a more severe condition of embrittlement and were used in this limited investigation. There was not enough time to make stress-rupture tests for all the conditions of rolling and heat treatment described in the preceding section. The specimens tested were rolled and solution treated in the alpha-beta-phase region. A 60 degree V notch with a 0.010-inch root radius was cut at the mid-point of a standard 0.250-inch-diameter test bar. The area under the notch was approximately one-half the area of the 0.250-inch-diameter cross section. Specimens were loaded to between 90 and 110 per cent of the unnotched ultimate tensile strength based on the area under the notch. The load and rupture times for the specimens tested are presented in Table 21. Some specimens were loaded at more than one stress level to speed up testing. It must be realized that the lower loads first applied may have had some effect on the resulting rupture times for higher loads. For this reason, the data may not be exactly quantitative, but the relative embrittlement of

* Summary Report to Watertown Arsenal, "The Effect of Hydrogen on the Mechanical Properties of Titanium and Titanium Alloys", July 31, 1954, Contract No. DA-33-019-ORD-938, p 21.

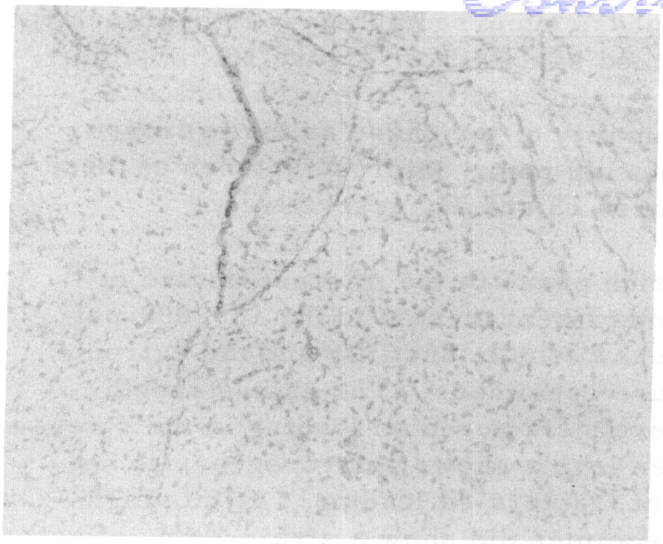
TABLE 21. STRESS-RUPTURE DATA FOR NOTCHED SPECIMENS OF HYDROGENATED Ti-3Mn-COMPLEX ALLOY AT ROOM TEMPERATURE (80 F)

		Original Stress		Stress Increased						
Average Hydrogen Content, ppm	Unnotched Ultimate Tensile Strength, psi	Per Cent of Ultimate Tensile Strength	Time at This Stress, hours	Per Cent of Ultimate Tensile Strength	Time at This Stress, hours	Per Cent of Ultimate Tensile Strength	Time at This Stress, hours	Per Cent of Ultimate Tensile Strength	Time at This Stress, hours	Time to Rupture at Maximum Stress, hours
Cold-Water Quenched After 1 Hour at 1300 F and Aged 48 Hours at 800 F										
260	175,000	95	--	--	--	--	--	--	--	On loading
140	178,500	95	--	--	--	--	--	--	--	On loading
90	179,000	95	6.1	--	--	--	--	--	--	6.1
30	175,500	95	240.7	--	--	--	--	--	--	Discontinued
Cold-Water Quenched After 1 Hour at 1300 F										
260	156,000	90	69.4	95	173	104	68.7	110	93.7	Discontinued
140	157,000	110	92.3	--	--	--	--	--	--	Discontinued
Cold-Water Quenched After 1 Hour at 1300 F and Aged 8 Hours at 1100 F										
260	121,000	95	167.7	110	6.3	--	--	--	--	6.3
140	120,000	110	92.3	--	--	--	--	--	--	Discontinued

the various conditions is well shown. Specimens at the 175,000 to 180,000-psi strength level showed severe embrittlement at all hydrogen levels above 30 ppm. Specimens containing 260 and 140 ppm of hydrogen fractured on loading to 95 per cent of the unnotched ultimate tensile strength, and the one containing 90 ppm ruptured in 6.1 hours at the same load level. Rupture did not occur in the specimen with 30 ppm of hydrogen in over 200 hours at 95 per cent of the unnotched ultimate tensile strength. Specimens in the solution-treated condition showed no embrittlement at 260 or 140 ppm of hydrogen. Neither specimen broke in 90 hours at 110 per cent of the unnotched ultimate tensile strength, which was about 155,000 psi. The two lower hydrogen levels were not tested in the solution-treated condition. The low-strength, 120,000-psi specimen containing 260 ppm of hydrogen had a rupture life of 6.3 hours under a load of 110 per cent of the unnotched ultimate tensile strength, whereas the bar with 140 ppm of hydrogen did not break at an equivalent load in over 90 hours. The hydrogen levels that showed embrittlement for the three heat-treated conditions may be summarized as follows:

	Hydrogen Content, ppm by weight	
Heat Treatment	Nonembrittled	Embrittled
1 hour at 1300 F, cold-water quenched, and aged 48 hours at 800 F	30	90, 140, 260
1 hour at 1300 F, cold-water quenched, and aged 8 hours at 1100 F	30, 90, 140	260
1 hour at 1300 F and cold-water quenched	30, 90, 140, 260	-

The degrees of susceptibility to hydrogen embrittlement shown by specimens given various heat treatments may be explained by considering the microstructures. At the high-strength level, a dark-etching phase was noted in specimens containing 260 or 140 ppm of hydrogen, as shown in Figures 18 and 21, respectively. Etching time may cause some variation in the appearance of specimens containing the dark-etching phase, as shown in Figures 19 and 20. It is demonstrated in these figures that the dark-etching phase is not alpha, but probably a hydrogen-rich phase. The structure of Figure 19 was obtained by light etching, which shows only a hydrogen-rich phase. In Figure 20, the specimen has been etched heavily, to show the alpha (faint white areas) to be much greater in area than the hydrogen-rich areas of the structure in Figure 19. The dark particles shown in Figure 19 seem to be masked by the over-all darkening of the matrix, as in Figure 20. The hydrogen-rich phase also seems to occur intergranularly in the deformed

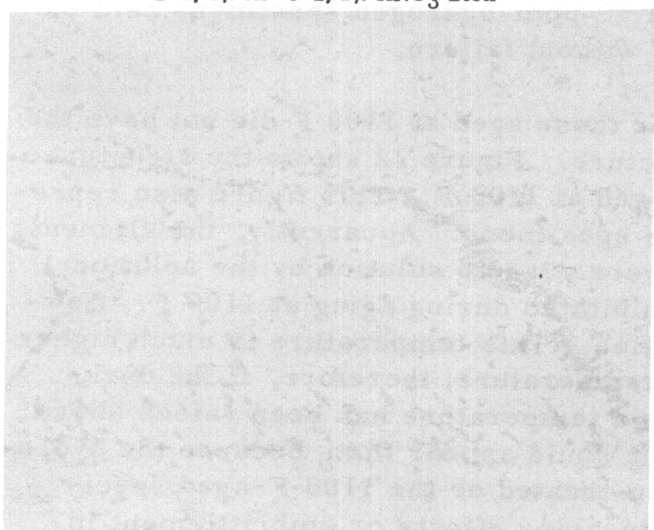


500X N18634

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 18. Ti-3Mn-COMPLEX ALLOY WITH 260 PPM OF HYDROGEN, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION AND AGED 48 HOURS AT 800 F; MEDIUM ETCH ON UNDEFORMED SPECIMEN

Structure: Dark, spheroidal, and intergranular hydrogen-rich phase. Lighter background is alpha in beta.

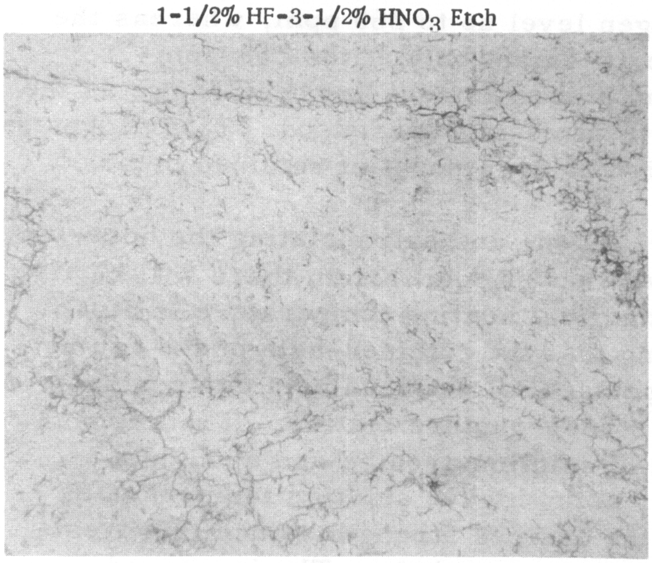


500X N18662

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 19. Ti-3Mn-COMPLEX ALLOY WITH 260 PPM OF HYDROGEN, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION AND AGED 48 HOURS AT 800 F; LIGHT ETCH ON COLD-DEFORMED GAGE SECTION OF SPECIMEN IN FIGURE 18

Structure: Dark, spheroidal, and thin intergranular hydrogen-rich phase. Light matrix same as in Figure 18.



500X N18664

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 20. Ti-3Mn-COMPLEX ALLOY WITH 260 PPM OF HYDROGEN, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION AND AGED 48 HOURS AT 800 F; HEAVY ETCH ON COLD-DEFORMED GAGE SECTION OF SPECIMEN IN FIGURE 18

Same Area as Figure 19, With a Heavier Etch.

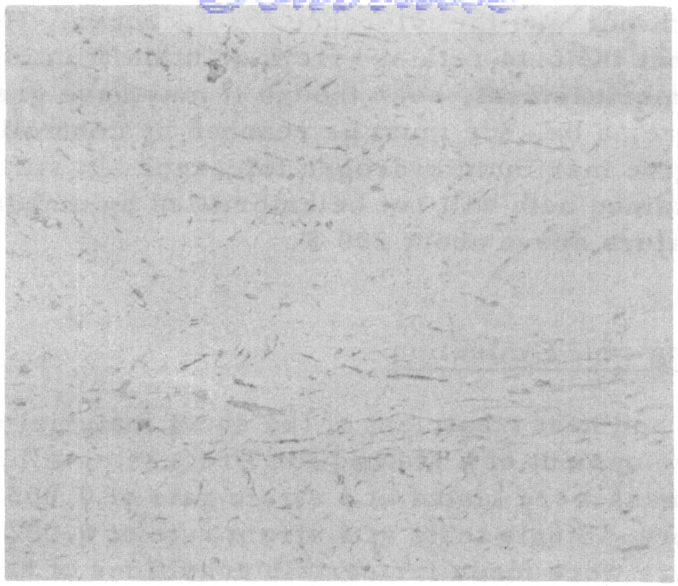
Structure: Dark, intergranular, hydrogen-rich phase. Indistinct light phase is alpha in a slightly darkened beta containing a fine, unresolved precipitate of alpha.

specimen of Figure 20, whereas this phase occurred as heavy intergranular or spheroidal particles in the undeformed specimen of Figure 18. Further evidence that the dark-etching phase is related to the hydrogen content is shown by the noticeable decrease in quantity of the phase as the hydrogen content was decreased from 260 ppm to 140 ppm. Microstructures of these two hydrogen levels are shown in Figures 18 and 21, respectively.

The occurrence of the hydrogen-rich phase in the microstructure of the 140- and 260-ppm-hydrogen specimens heat treated to the 180,000-psi strength level may be the cause of the low tensile ductility and the extreme embrittlement of notched stress-rupture specimens. The high-strength specimens at the two high-hydrogen levels fractured on loading in the stress-rupture testing. The two low-hydrogen levels shows no hydrogen-rich phase in the microstructure and did not fracture on loading. At this high strength level the 90-ppm-hydrogen specimen did show a strain-aging effect by its short rupture life of about 6 hours when loaded to 95 per cent of the ultimate tensile strength, whereas the 30-ppm-hydrogen specimens held the same relative load for over 200 hours without failure.

The solution-treated specimens or those aged at 1100 F did not have the hydrogen-rich phase in the microstructure. Figure 22 shows the structure for the 260-ppm-hydrogen specimen aged at 1100 F. This figure also represents the structure of solution-treated specimens. Apparently, the elements that make up the dark-etching phase were put into solution by the solution treatment at 1300 F and were not precipitated during aging at 1100 F. However, aging at 800 F caused precipitation. This temperature is much higher than the titanium-hydrogen-eutectoid temperature; therefore, if the dark-etching phase is a hydride, its eutectoid temperature has been raised above 800 F in the Ti-3Mn-complex alloy. It would appear that, because the hydrogen was not precipitated in the solution-treated or the 1100 F-aged specimens, they might exhibit greater strain-aging effects or embrittlement in the notched-stress-rupture tests. However, the solution-treated specimens showed no embrittlement at any hydrogen level up to 260 ppm, whereas the specimens aged at 1100 F showed embrittlement only at the 260-ppm-hydrogen level. Therefore, increasing embrittlement has been shown as the temperature of the final stage of heat treatment is decreased. This suggests that the embrittlement may be a function of the amount of retained beta.

Assuming no metallic-eutectoid reaction, and extrapolating the beta-transus line to 800 F, it is readily seen that at equilibrium there will be decreasing amounts of beta phase as the final heating temperature is decreased from 1300 to 800 F. Assuming that the retained-beta phase can act as a reservoir for the hydrogen, the per cent of retained beta can control the hydrogen embrittlement shown by stress-rupture testing. If this hypothesis is valid, hydrogen embrittlement of solution-treated and aged beta-stabilized alloys would be improved by using higher solution temperatures and higher aging temperatures to get the desired strength, since this treatment should result in greater amounts of retained beta. The increased

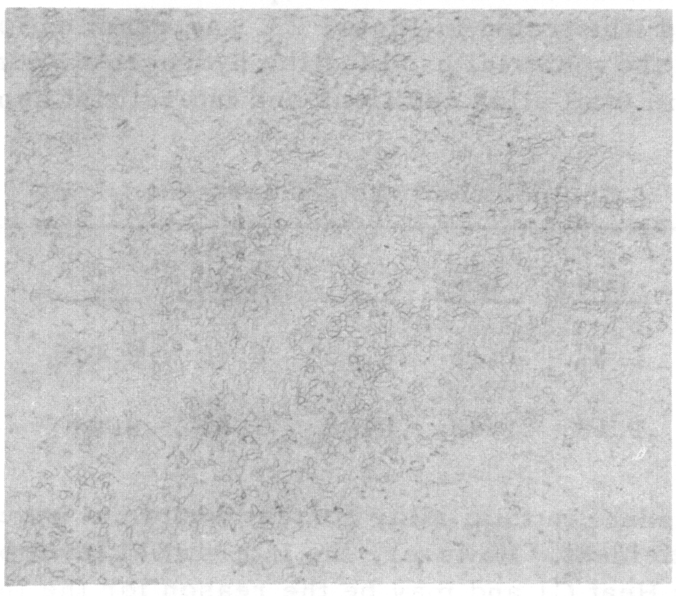


500X N18645

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 21. Ti-3Mn-COMPLEX ALLOY WITH 140 PPM OF HYDROGEN, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION AND AGED 48 HOURS AT 800 F; MEDIUM ETCH

Structure: Much less dark, spheroidal, and intergranular hydrogen-rich phase than in Figure 18. Lighter matrix is alpha in beta.



500X N18639

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 22. Ti-3Mn-COMPLEX ALLOY WITH 260 PPM OF HYDROGEN, ROLLED AND SOLUTION TREATED IN THE ALPHA-BETA-PHASE REGION AND AGED 8 HOURS AT 1100 F; HEAVY ETCH

Structure: Absence of any dark hydrogen-rich phase. Structure is primary alpha in a beta matrix containing unresolved precipitated alpha.

amount of beta would increase the tolerance for hydrogen. However, it must be cautioned that the beta retained from solution treating may be susceptible to omega embrittlement, even though it may have greater hydrogen tolerance. Therefore, a balance must be reached by controlling the amount of retained beta to give maximum hydrogen tolerance but still have sufficient stability that the retained beta will not be embrittled by omega formation on exposure to temperature above about 200 F.

Sheet Tensile Testing and Evaluation

The processing and heat treatment of the sheet material were discussed in the section, "Development of a Method for Processing Alloy Sheet". Heat-treated sheet specimens were tested at a strain rate of 0.005 inch per inch per minute to fracture. Single tests at a strain rate of 0.002 inch per inch per minute to fracture were made for certain conditions of heat treatment. The results of these tensile tests are in Table 22. The tensile properties show no definite change with increasing hydrogen content in either the high- or low-strength conditions after solution treating in the alpha-beta-phase region. Specimens solution treated at 1600 F and aged showed a decrease in ductility at the highest hydrogen level.

The reason for the apparent relative insensitivity of the tensile ductility of this sheet material to hydrogen contents up to 260 ppm is not known, since data that were obtained earlier from sheet specimens of Heat C1 did show an effect of hydrogen as illustrated in Figure 23. An examination of the analyses of Heat C1* and the material used for the hydrogen evaluation, Heat C32, showed differences in total alloy additions and interstitial contents as follows:

Composition, weight per cent

Heat	Mn	Fe	Cr	Mo	V	Total Alloy	C	O	N
C1	3.78	1.06	1.47	1.16	1.13	8.60	0.015	0.205	0.022
C32	3.36	0.91	1.18	1.08	0.98	7.51	0.02	0.09	0.008

The 1 per cent difference in total alloy content would not have much effect on hydrogen embrittlement. However, the interstitial level of Heat C32 is much lower than for Heat C1 and may be the reason for the insensitivity to hydrogen content of Heat C32. It would appear that, before a specification for hydrogen content can be established that would insure no embrittlement, its interaction effect with the other interstitials will have to be evaluated.

* Refer to section on "Development of a Method for Processing Alloy Sheet".

TABLE 22. EFFECT OF HYDROGEN CONTENT, HEAT TREATMENT, AND STRAIN RATE ON TENSILE PROPERTIES OF Ti-3Mn-COMPLEX ALLOY ROLLED IN THE ALPHA-BETA FIELD (1400 F) TO 14-GAGE SHEET

Average Hydrogen Content, ppm by weight	Solution Temperature, F[(a)]	Aging[(b)] Time, hours	Aging[(b)] Temperature, F	Strain Rate, in./in./min	Elongation, per cent in 2 inches[(c)]	Ultimate Tensile Strength, psi[(c)]
30	1300	48	800	0.005	9.5	167,000
30	1300	48	800	0.002	5.5[(d)]	164,500[(d)]
30	1300	8	1100	0.005	24.0	121,500
30	1300	8	1100	0.002	22.0[(d)]	111,000[(d)]
30	1600	24	900	0.005	1.5[(d)]	187,500
30	1600	8	1100	0.005	9.0	134,000
90	1300	48	800	0.005	9.5	162,000
90	1300	48	800	0.002	9.5[(d)]	162,000[(d)]
90	1300	8	1100	0.005	24.5	123,500
90	1600	24	900	0.005	0	171,000
90	1600	8	1100	0.005	7.5	124,000
140	1300	48	800	0.005	11.0	167,000
140	1300	48	800	0.002	3.5[(d)]	163,500[(d)]
140	1300	8	1100	0.005	23.5	121,500
140	1600	24	900	0.005	1.0[(e)]	174,000
140	1600	8	1100	0.005	8.0	125,000
260	1300	48	800	0.005	11.0	159,000
260	1300	48	800	0.002	8.5[(d)]	157,500[(d)]
260	1300	8	1100	0.005	23.5	116,500
260	1300	8	1100	0.002	24.0[(d)]	117,500[(d)]
260	1600	24	900	0.005	1.0[(d)]	162,000[(d)]
260	1600	8	1100	0.005	3.5	125,500

(a) Cold-water quenched after 1/2 hour at temperature in purified argon.
(b) Aged in air.
(c) Average of two, except where noted.
(d) Single values.
(e) Uniform-elongation specimens broke outside gage marks.

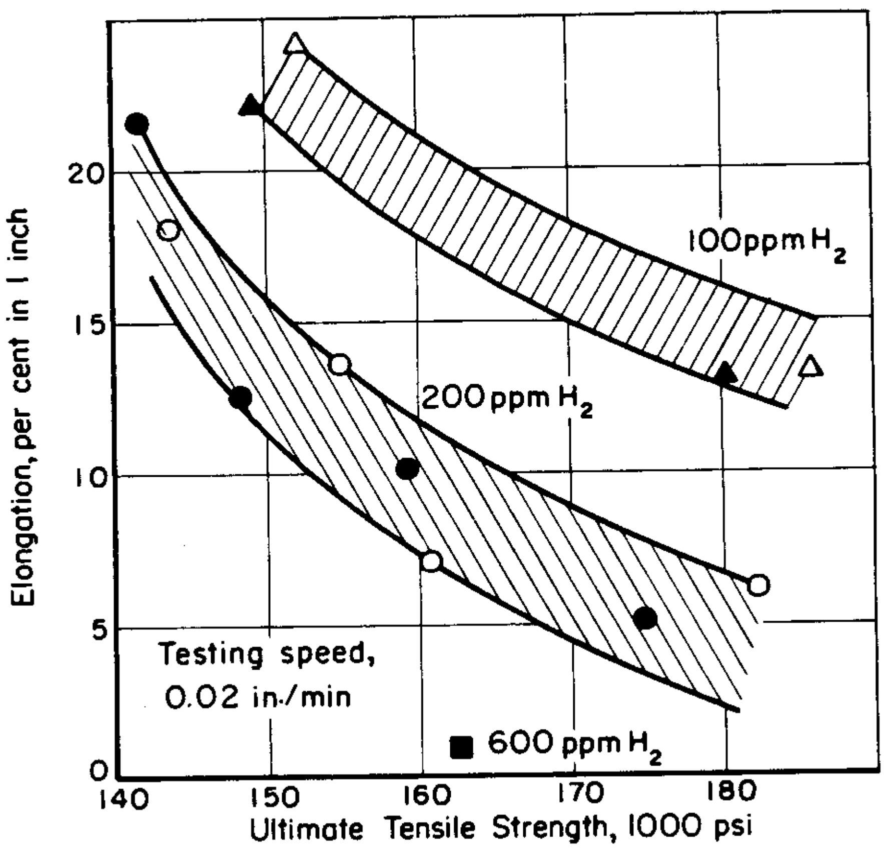


FIGURE 23. EFFECT OF HYDROGEN CONTENT ON THE DUCTILITY OF HEAT-TREATED Ti-3 Mn-COMPLEX-ALLOY SHEET, HEAT C1

N-18174

ELEVATED-TEMPERATURE PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY

The room-temperature, heat-treated tensile properties of the Ti-3Mn-complex alloys are somewhat better than those of the present commercial titanium alloys. This alloy was also relatively stable for 1000 hours at 800 F at a strength of about 170,000 psi (WADC Technical Report 54-205, pp 35-44. The data presented in this report were obtained to substantiate the usefulness of this beta-stabilized alloy for service at elevated temperatures.

The diamond-pyramid hardness, the elevated-temperature tensile properties, and the dynamic Young's modulus were determined from room temperature to 1000 F. In addition, the 100-hour stress-rupture strengths were determined at 650 and 800 F.

Effect of Temperature on Hardness

Hot hardness usually indicates the trend of elevated-temperature tensile strengths and is much less expensive to determine. For metals, a plot of the log of the hardness or tensile strength versus temperature results in straight, parallel lines up to the softening temperature. Hardness was obtained for several heat-treated conditions of the Ti-3Mn-complex alloy, and then two conditions of heat treatment were tensile tested to check the relationship of elevated-temperature hardness and tensile strength. The different strength levels for the hardness specimens were obtained by heat treating 1-1/2-inch lengths of 1/2-inch-diameter bar stock as follows:

Nominal Strength, psi	Solution Temperature, F	Aging	
		Time, hours	Temperature, F
190,000	1300	48	800
165,000	1300	24	900
150,000	1300	4	1000
135,000	1300	8	1100
180,000	1400	4	1000

Two 1/4-inch-thick sections were cut from the centers of the heat-treated bars (two were needed to provide enough area for all of the hardness impressions). The hardness values were obtained in a transverse section of the bars in a special apparatus. This apparatus consists of a vacuum chamber containing a movable table, six independent indenters, and a radiation heat source. The table is moved by controls from outside the chamber and will hold six specimens. Each specimen is placed under one of the six indenters, which are 136-degree square-pyramid sapphires mounted in a holder. Both specimens and indenters are heated slowly in vacuum by radiation. When the desired temperature is reached, three or more impressions are made on each specimen by allowing the six indenter assemblies to apply their full load for 10 seconds on each specimen. After cooling, the impressions are measured at room temperature, with no correction for thermal contraction, which is assumed to be negligible. The average diamond-pyramid-hardness value for each specimen and temperature is given in Table 23. It may be seen that the initial heat treatment has an effect on the elevated-temperature hardnesses. As-heat-treated, room-temperature hardnesses ranged from 332 to 417 DPH. At 1000 F, a range of 72.5 to 108 DPH was still evident. It will be shown in the next section that the initial heat treatment also affected the elevated-temperature tensile strength.

TABLE 23. EFFECT OF TEMPERATURE ON HARDNESS OF THE Ti-3Mn-COMPLEX ALLOY AFTER VARIOUS HEAT TREATMENTS

Heat Treatment			Diamond-Pyramid Hardness Number[b] for Test Temperature, F					
Solution Temperature[a], F	Aging Time, hours	Aging Temperature, F	Room Temp	300	500	800	900	1000
1300	48	800	417	331	294	213	182	94.8
1300	24	900	376	307	288	202	149	85.5
1300	4	1000	362	280	247	208	133	84.9
1300	8	1100	332	247	212	156	114	72.5
1400	4	1000	389	305	263	216	166	108

(a) Quenched into cold water after 1 hour at temperature.

(b) 730-gram load; average of 3 or more impressions.

Elevated-Temperature Tensile Strengths

Elevated-temperature tensile strengths were determined at 300, 500, 800, and 900 or 1000 F for two strength levels of the Ti-3Mn-complex alloy. A strength level of about 135,000 psi was obtained by solution treating 1 hour at 1300 F, cold-water quenching, and aging for 8 hours at 1100 F. The strength level of about 180,000 psi was obtained by solution treating 1 hour at 1400 F, cold-water quenching, and aging for 4 hours at 1000 F. The tensile tests were made in a standard tensile machine at a head speed of 0.01 inch per minute. A resistance-heating furnace was used and the specimens were held for 1/2 hour at temperature before testing. The furnace was a variable-tap type which can be adjusted to give a uniform temperature over the entire gage length of the specimen. Tensile properties for each specimen are given in Table 24. Included in this table are the hot hardnesses

TABLE 24. VARIATION OF TENSILE PROPERTIES AND HARDNESSES WITH TEMPERATURE FOR THE Ti-3Mn-COMPLEX ALLOY HEAT TREATED TO TWO STRENGTH LEVELS

Initial Heat Treatment	Test Temperature, F	Elongation, per cent, in 1 Inch	Elongation, per cent, in 2 Inches	Reduction of Area, per cent	Yield Strength[a], 0.2 Per Cent Offset, psi	Ultimate Tensile Strength, psi	DPH
Solution treated 1 hour at 1300 F, cold-water quenched, and aged 8 hours at 1100 F	Room temperature	36	23	58	135,000	135,500	332
	300	33	26	63	109,000	121,000	247
	500	28	20	60	87,500	104,000	212
	800	41	26	88	69,500	76,000	156
	1000	89	50	∽100	24,500	31,000	72.5
Solution treated 1 hour at 1400 F, cold-water quenched, and aged 4 hours at 1000 F	Room temperature	1.5[b]	2.0[b]	5[b]	164,500	166,500[b]	389
	300	24	16	60	132,500	150,000	305
	500	22	15	61	109,000	132,000	263
	800	33	19	90	85,000	109,000	216
	900	50	28	∽100	57,000	73,500	166

(a) Strain rate of 0.01 inch per minute.

(b) This specimen broke in a surface defect in the gage length. Thus, all the tensile properties except the yield strength are probably in error. Room-temperature ultimate tensile strength should be about 180,000 psi for the heat treatment given.

obtained for the same heat treatments. The log of the ultimate tensile strength and hardness versus temperature are plotted in Figure 24.

The hardness and tensile-strength curves are represented by straight lines up to about 800 F. Above this temperature, there is a sharp decrease in both values. The hardness and tensile strength for each strength level have been drawn parallel, and only a few points show a significant deviation. Included in this figure is a curve showing the variation of tensile strength with temperature of C130AM alloy*. The 180,000-psi strength level for the Ti-3Mn-complex alloy retains a higher strength than does the C130AM alloy in the commercial annealed condition up to about 830 F. At this temperature, the strength curves cross, and C130AM has higher strengths above this temperature.

Dynamic Young's Modulus

The dynamic Young's modulus of the Ti-3Mn-complex alloy was determined at temperatures up to 1000 F. The same heat-treated conditions as used for the elevated-temperature tensile tests were tested. Modulus was calculated from the resonant frequency of test specimens by means of the formula:

$$E = 9.18 \times 10^{-6} \times \frac{L^4}{d^4} \times \frac{w}{L} \times f^2,$$

where

E = Young's modulus
L = length of specimen, inches
d = diameter, inches
w = weight, grams
f = resonant frequency, cycles/second.

Resonant frequency was determined by a method similar to that used by C. W. Andrews**. The specimen (3/16-inch diameter by 5 inches long) is hung from two asbestos strings, one connected to the exciting system and the other to a receiver. The exciting string is connected to a speaker coil energized by a variable-frequency oscillator. As the natural frequency of the specimen is approached, its amplitude of vibration increases, reaching a maximum when the exciting and natural frequencies coincide. The amplitude of vibration is converted into electrical oscillations by means of a crystal pickup supporting the receiving string. The output from this crystal is measured with an oscilloscope. For elevated-temperature

* Metal Progr., 66 (1-A), 89 (July, 1954).

** Andrews, C. W., "Effect of Temperature on Modulus of Elasticity", Metal Progr., 58, 85 (July, 1950).

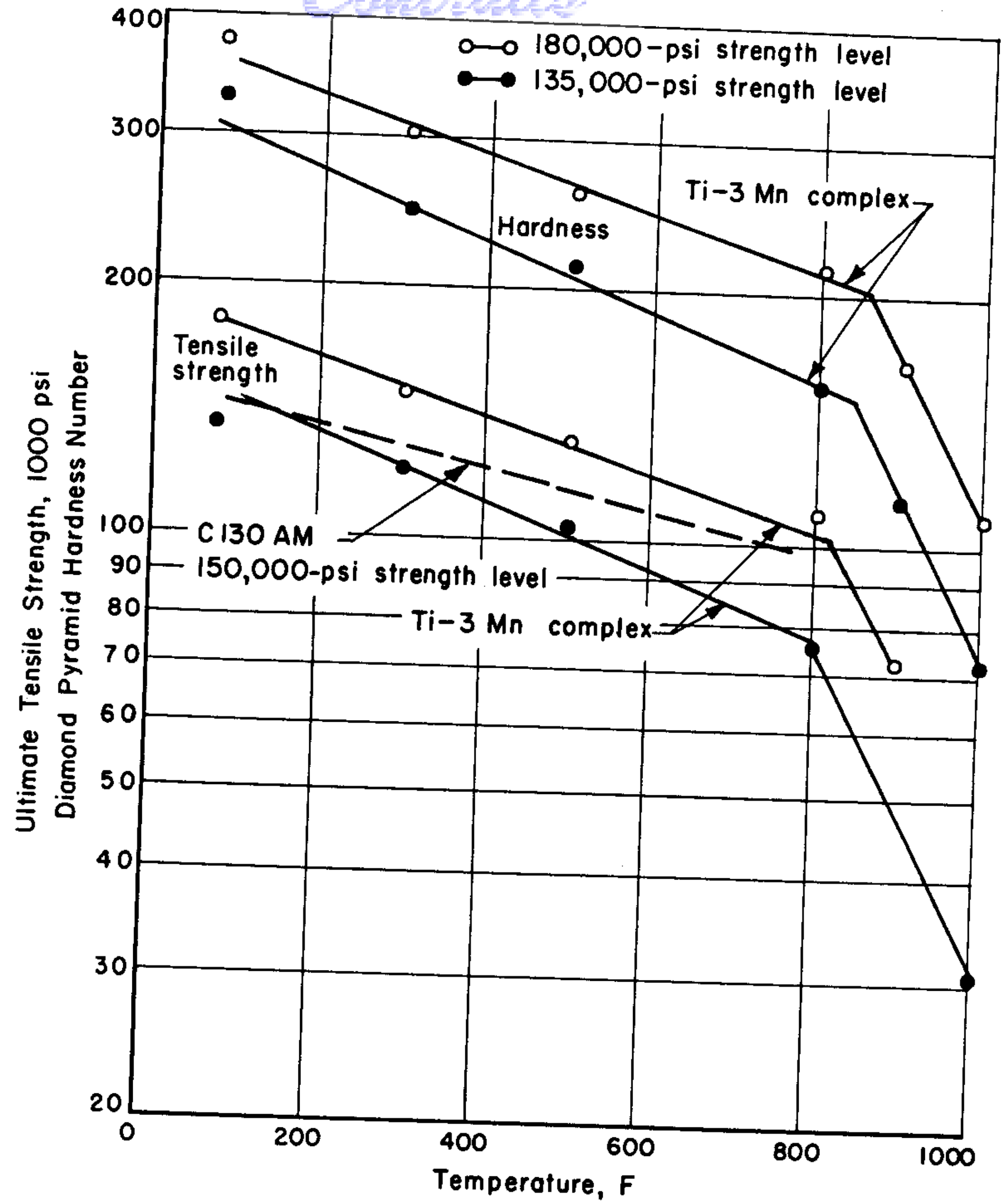


FIGURE 24. EFFECT OF TEMPERATURE ON HARDNESS AND TENSILE STRENGTH FOR THE Ti-3 Mn-COMPLEX ALLOY SOLUTION TREATED AND AGED TO TWO STRENGTH LEVELS

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determinations, the test specimen is supported in a Vycor tube equipped with side tubes through which the asbestos strings pass. The Vycor tube is heated by a resistance furnace. During testing, an atmosphere of purified-argon gas was maintained within the tube.

The dynamic Young's modulus values for the two heat-treated conditions are presented in Table 25. The values decreased uniformly with increasing temperature from about 15.5 million psi at room temperature to 11.5 million psi at 1000 F. The effect of the two different heat treatments on the dynamic Young's modulus was insignificant.

Stress-Rupture Testing

The elevated-temperature tensile properties of the Ti-3Mn-complex alloy indicate that it may have good stress-rupture characteristics. The 100-hour stress-rupture strengths were obtained at 650 and 800 F for the two heat-treated conditions used in the elevated-temperature tensile testing. The results of these tests are given in Table 26. The loads and rupture times are plotted on a log-log scale in Figure 25. On a log-log plot, the variation of rupture time with stress is usually a linear function. By extrapolating the data obtained, the following stress-rupture strengths were obtained:

Initial Room-Temperature Strength Level, psi	Yield Strength, 0.2 Per Cent Offset, psi		Ultimate Tensile Strength, psi		Stress-Rupture Strength, psi		
	650 F	800 F	650 F	800 F	100 Hours, 650 F	1000 Hours, 650 F	100 Hours, 800 F
135,000	78,500	69,500	88,000	76,000	96,000	94,000	38,000
180,000	96,000	85,000	116,000	109,000	116,000	94,000	46,500

The 100-hour stress-rupture strengths at 800 F were, as expected, considerably lower than the tensile yield strengths at this temperature. However, at 650 F, the 100-hour stress-rupture strengths were equal to or higher than the elevated-temperature tensile strengths for both heat-treated conditions. This behavior indicates a possible strain-aging reaction. The 1000-hour rupture strengths at 650 F for the two conditions of heat treatment were the same, but still about equal to or higher than the elevated-temperature yield strength.

TABLE 25. VARIATION OF DYNAMIC YOUNG'S MODULUS WITH TEMPERATURE FOR THE Ti-3Mn-COMPLEX ALLOY SOLUTION TREATED AND AGED TO TWO STRENGTH LEVELS

Test Temperature, F	135,000-Psi Level, Solution Treated 1 Hour at 1300 F, Cold-Water Quenched, and Aged 8 Hours at 1100 F	180,000-Psi Level, Solution Treated 1 Hour at 1400 F, Cold-Water Quenched, and Aged 4 Hours at 1000 F
	Dynamic Young's Modulus, 10^6 psi	
Room temperature	15.2	15.6
200	14.7	15.1
300	14.4	14.6
400	14.1	14.2
500	13.8	13.8
600	13.4	13.3
700	13.0	12.8
800	12.5	12.3
900	11.9	11.9
1000	11.6	11.4
	Resonant Frequency of Vibration, cps	
Room temperature	1680	1688
500	1596	1581
1000	1465	1436

TABLE 26. VARIATION OF STRESS-RUPTURE PROPERTIES WITH TEMPERATURE AND HEAT TREATMENT FOR THE Ti-3Mn-COMPLEX ALLOY

Initial Heat Treatment	Test Temperature, F	Stress, psi	Rupture Time, hours	Elongation, per cent	Reduction of Area, per cent	Minimum Creep Rate, per cent/hour
	135,000-Psi Ultimate-Tensile-Strength Level					
Solution treated 1 hour at 1300 F, cold-water quenched, and aged 8 hours at 1100 F	800	62,000	2.1	33.1	77.3	-
	800	40,000	74.1	52.9	84.1	0.22
	650	100,000	1.4	23.7	56.8	5.6
	650	98,000	7.4	21.2	59.3	2.98
	650	95,000	413.3	24.1	51.6	0.025
	180,000-Psi Ultimate-Tensile-Strength Level					
Solution treated 1 hour at 1400 F, cold-water quenched, and aged 4 hours at 1000 F	800	60,000	28.8	38.6	85.6	0.32
	800	45,000	116.1	56.3	89.4	0.086
	650	130,000	12.6	17.0	50.2	0.275
	650	110,000	256.6	21.6	57.9	0.022
	650	100,000	675.0	23.1	66.2	0.0092

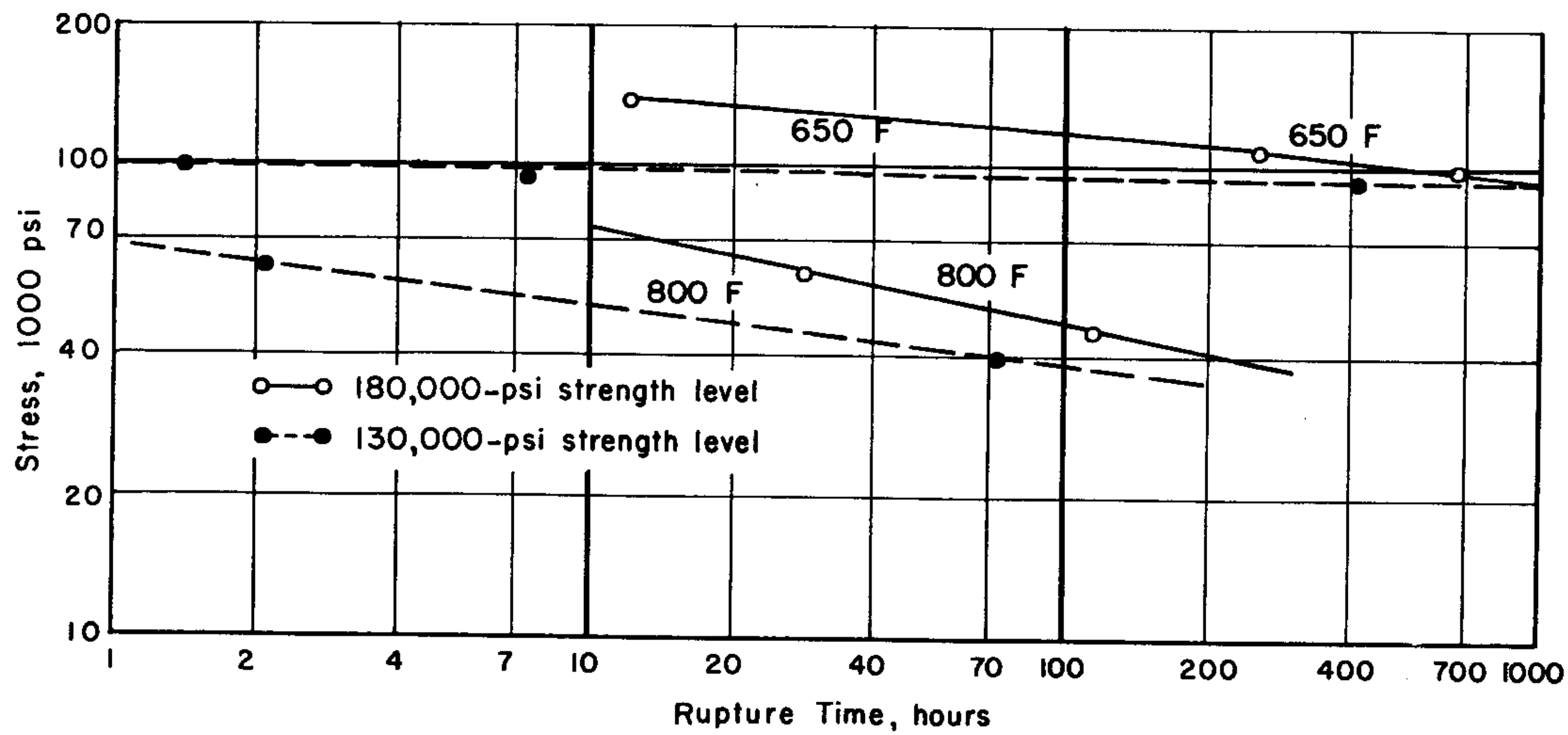

FIGURE 25. STRESS-RUPTURE PROPERTIES OF THE Ti-3 Mn-COMPLEX ALLOY HEAT TREATED TO TWO STRENGTH LEVELS

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INVESTIGATION OF HEAT TREATMENTS TO MINIMIZE BETA EMBRITTLEMENT

With the exception of certain vanadium binary alloys, all of the beta-stabilized alloys evaluated under this contract have shown decreased ductility after heating into the beta-phase region subsequent to fabrication. Attempts to determine the exact cause of this phenomenon have been unsuccessful thus far. However, one microstructural condition has been found to be common to all embrittled specimens, namely, a continuous or semi-continuous network of alpha at the beta-grain boundaries. In the previous summary report, the fracture of a beta-embrittled specimen was shown to occur between the grain-boundary alpha and the beta matrix (WADC Technical Report 54-205, p 28). This indicates that perhaps the alpha, per se, is not the embrittling agent, but that some impurity phase or concentration of impurities at the alpha-beta interface is responsible. Nevertheless, the presence of the alpha in a continuous network is undoubtedly a major factor contributing to the embrittlement. The possibility exists that the continuous nature of the grain-boundary alpha could be altered by suitable heat treatment. The investigation described in this section was undertaken with this objective in mind. In the course of the investigation, some data also were obtained on the effects of heat treatment and alloy composition on the beta- and alpha-grain sizes and the shape of the precipitated alpha phase.

The nominal compositions of the alloys selected for the heat-treatment study were as follows:

Ti-3Mn-1Cr-1Fe-1Mo-1V (3Mn complex)
Ti-3Mn-1Cr-1Fe-1Mo-0.5Al
Ti-5Mn-2.5Cr
Ti-5Mn-2.5Cr-0.5Al
Ti-5Mn-2.5Cr-1Th.

The aluminum- and thorium-containing alloys were included because previous work had indicated that these elements reduced the susceptibility of alloys to beta embrittlement. The heat treatments applied to each alloy were designed to provide varying conditions for the nucleation and growth of the alpha phase from an all-beta structure. They were as follows:

A - One hour at 1600 F, water quench
One hour at 1300 F, water quench

B - One hour at 1600 F, water quench
Age 24 hours at 800 F, air cool
One hour at 1300 F, water quench

C - One hour at 1600 F, water quench
Age 2 hours at 1100 F, air cool
One hour at 1300 F, water quench

D - One hour at 1600 F, quench into lead at 1300 F, hold 1 hour, water quench

E - One hour at 1600 F, quench into lead at 800 F, hold 24 hours, water quench, reheat at 1300 F 1 hour, water quench

F - Same as E, except reheat to 1300 F directly from 800 F lead bath, hold 1 hour, water quench

G - One hour at 1600 F, water quench, reheat to a temperature 100 F below the beta transus of the alloy, hold 1 hour, furnace cool 100 F, hold 1 hour, water quench

H - Same as G, except that alloy is furnace cooled from 1600 F to the temperature 100 F below the beta transus.

The general features of the microstructures produced by these heat treatments are summarized in Table 27.

None of the heat treatments had a beneficial effect on the grain-boundary alpha. It was present as a continuous network in all cases. However, the different heat treatments did affect appreciably the size and shape of the alpha occurring within the grains. The effect of a given heat treatment on the alpha varied somewhat with alloy composition but, generally speaking, the treatments that involved furnace cooling in the alpha-beta temperature range resulted in large, acicular alpha grains (Treatments G and H), as shown in Figure 28. The remaining treatments generally produced small alpha grains with a low length-to-width ratio, as illustrated in Figures 26 and 27. This tendency toward equiaxed alpha grains was strongest in the Ti-3Mn-complex alloy.

The most striking observation made in these tests was of the effect of alloy composition on beta-grain size. Generally, all heat treatments produced very large beta grains in the Ti-5Mn-2.5Cr alloy and somewhat smaller grains in the Ti-3Mn-complex alloy. The addition of 1 per cent thorium to the former alloy sharply reduced the resultant beta-grain size. Some idea of the magnitude of the effect may be obtained from Figures 26 and 27. It may be seen that the grain size of the Ti-5Mn-2.5Cr-1Th alloy was appreciably smaller than that of the Ti-3Mn-complex alloy, which, in turn, had a smaller grain size than did the Ti-5Mn-2.5Cr alloy, without thorium.

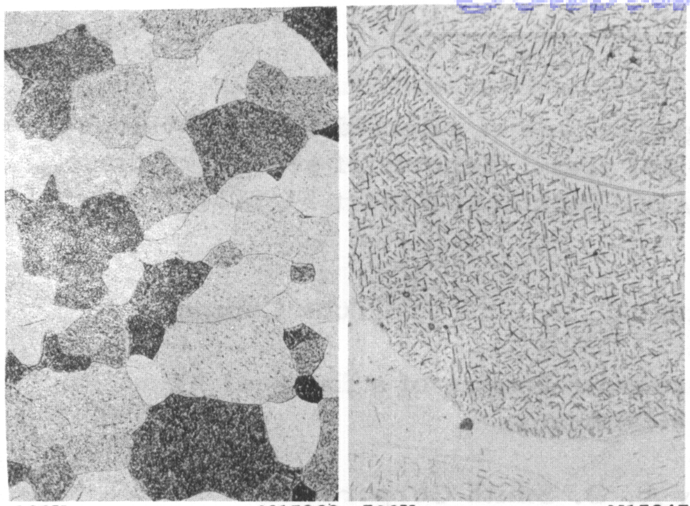
To our knowledge, this is the first evidence of a grain-refining action directly traceable to the presence of a relatively small amount of one element in a beta-stabilized titanium alloy. In this respect, thorium appears to be analogous to aluminum in steel, although its effect is not so pronounced. If other elements can be found that will intensify the effect of thorium, it is possible that the beta embrittlement of titanium alloys can be minimized, if not completely eliminated.

TABLE 27. EFFECTS OF SUBSEQUENT HEAT TREATMENTS ON THE GENERAL MICROSTRUCTURAL CHARACTERISTICS OF VARIOUS ALLOYS HEATED INTO THE BETA-PHASE REGION

Nominal Alloy Composition, per cent	Heat Treatments[a] That Produce the Following							
	Relative Beta-Grain Size			Relative Alpha-Grain Size			Alpha-Grain Shape	
	Large	Medium	Small	Large	Medium	Small	Nearly Equiaxed[b]	Acicular Widmanstätten
Ti-3Mn-1Cr-1Fe-1Mo-1V	-	All	-	H	D	A, B, C, E, F, G	A, B, C, E, F	D, G, H
Ti-3Mn-1Cr-1Fe-1Mo-0.5Al	-	All	-	D, G	H	A, B, C, E, F	A, B, C, F	D, E, G, H
Ti-5Mn-2.5Cr	All	-	-	D, H	-	A, B, C, E, F, G	A, B, C, E, F	D, G, H
Ti-5Mn-2.5Cr-0.5Al	All	-	-	H	D, G	A, B, C, E, F	A, B, C	D, E, F, G, H
Ti-5Mn-2.5Cr-1Th	-	-	All	-	G, H	A, B, C, D, E, F	B, E, F	A, C, D, G, H

(a) Heat treatments — see pages 90 and 91 for meaning of letters.

(b) Alpha was considered equiaxed if length-to-width ratio did not exceed 2:1.

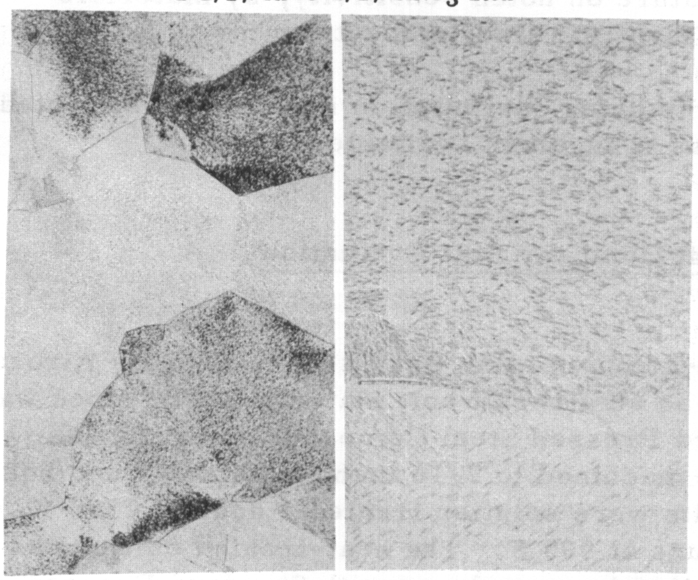


100X N15362 500X N15347

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 26. Ti-5Mn-2.5Cr-1Th ALLOY HEATED 1 HOUR AT 1600 F, COLD-WATER QUENCHED, AGED 2 HOURS AT 1100 F, AIR COOLED, REHEATED 1 HOUR AT 1300 F, COLD-WATER QUENCHED

Small beta-gram size
Small Widmanstätten-alpha-grain size.



100X N15360. 500X N15345

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 27. Ti-3Mn-COMPLEX ALLOY HEATED 1 HOUR AT 1600 F, COLD-WATER QUENCHED, AGED 2 HOURS AT 1100 F, AIR COOLED, REHEATED 1 HOUR AT 1300 F, COLD-WATER QUENCHED

Medium beta-grain size
Very small equiaxed alpha

100X N15355

1-1/2% HF-3-1/2% HNO_3 Etch

FIGURE 28. Ti-3Mn-COMPLEX ALLOY HEATED 1 HOUR AT 1600 F, FURNACE COOLED TO 1450 F, HELD FOR 1 HOUR AND FURNACE COOLED 100 F, HELD FOR 1 HOUR, COLD-WATER QUENCHED

Large beta-grain size
Large Widmanstätten-alpha-grain size

INDUSTRIAL EVALUATION

Evaluation by industrial organizations of selected alloys developed under this contract was started last year. Reports of several of these investigations are covered in WADC Technical Report 54-205. These included:

(1) Cold drawing of tubing, by Superior Tube Company

(2) Experimental closed-die forging of a flat gear blank by Wyman-Gordon Company

(3) Flash welding, by A. O. Smith Corporation

(4) Effect of temperature on notch sensitivity, by Materials Laboratory of Wright Air Development Center.

The industrial-evaluation work is being continued on materials distributed last year and on new material as it becomes available.

Douglas Aircraft Company Bolt Evaluation

Several feet of the Ti-3Mn-complex alloy were sent to Douglas Aircraft Company, Inc., for evaluation as an aircraft bolt material. The stock was fabricated into bolts by Standard Pressed Steel Company, using the following procedure. The bar stock was machined to 7/16 inch in diameter and hot headed. After heading, the bolts were solution treated 1 hour at 1300 F, water quenched, and aged 8 hours at 900 F. The heat-treated bolt blanks were thread rolled and fillet rolled in accordance with Standard Pressed Steel Company procedure for making MS20004 series steel bolts. No further treatment was given the bolts before tensile and fatigue testing.

Three bolts were machined to standard 1/4-inch-diameter tensile specimens and tested, with the following results:

Elongation, per cent in 1 inch	Reduction of Area, per cent	Yield Strength, psi	Ultimate Tensile Strength, psi
17	46	156,000	169,000
17	49	156,000	170,000
18	52	158,000	171,000

Two bolts were tested in tension with no reduced section. The breaking strengths, calculated on the minimum thread-root section, were 191,000 and 204,000 psi.

Three bolts were fatigue tested at 69,000-psi maximum stress (minimum stress 10 per cent of maximum, axially loaded). Two bolts failed in the threads at 14,400 cycles, and the third broke under the head at 34,200 cycles. For the fatigue stress tested, a minimum life of 80,000 cycles and a mean life of 150,000 cycles would be expected of the MIL-B-7838A steel bolts. There are several possible reasons for the low fatigue life of the titanium bolts, which indicates the need for further work.

The failure under the head was probably due to hot heading in the beta field. The microstructure of the heads was large grained with intergranular and Widmanstätten alpha, which usually results in low tensile ductility. The effect of this type of structure on fatigue properties is not known, but it is reasonable to assume, from its effect on tensile properties, that it would be detrimental. The thread failures, as indicated by Douglas, may have been due to a small thread-root radius of 0.004 inch. A radius of 0.006 inch is specified for steel bolts of MIL-B-7838A series. Douglas further reports that RC-130-B alloy given a similar heat treatment had low fatigue life with a 0.004-inch thread-root radius. Another lot of RC-130-B, however, with a larger radius of 0.005 inch, had fatigue life very close to that for steel.

Cold rolling of the heat-treated titanium may have produced unfavorable residual stresses. Therefore, a stress anneal may improve the fatigue life of the Ti-3Mn-complex-alloy bolts. These variables should be investigated.

Ti-3Mn-Complex Alloy Distributed for Industrial Evaluation

The large ingot of the Ti-3Mn-complex alloy melted by the United States Bureau of Mines was to be distributed as 7/8-inch-round stock. After most of this material had been distributed to various companies, it was found to have a high hydrogen content (200 ppm). Most of this high-hydrogen material has been returned on request. Low-hydrogen stock of this alloy, fabricated at Battelle, was sent to the following companies and personnel:

	Low-Hydrogen Stock Distributed
Consolidated-Vultee Aircraft Corporation, Mr. Frank Fink	12 ft, 1/2-inch round
Curtiss-Wright Corporation, Propeller Division, Mr. Wm. C. Schulte	12 ft, 1/2-inch round

	Low-Hydrogen Stock Distributed
North American Aviation, Inc., Los Angeles Division, Mr. C. V. Hansen	12 ft, 1/2-inch round
Lockheed Aircraft Corporation, Mr. Tiktinsky	12 ft, 1/2-inch round

Reports on this material have not yet been received.

Data on which this report is based are recorded in Battelle Laboratory Record Books No. 8135, pages 35 through 100, and No. 9609, pages 1 through 31.