WADC TECHNICAL REPORT 52-334

DEVELOPMENT OF TITANIUM-BASE ALLOYS

Battelle Memorial Institute
Columbus 1, Ohio

December 31, 1952

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RDO No. 615-11

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared at Battelle Memorial Institute under Wright-Patterson Air Force Base Contract No. AF 33(038)-3736. The project was administered by the Research Division of the Materials Laboratory, Air Development Center. Dr. H. K. Adenstedt and Lt. J. R. Pequignot of that office were the Project Engineers.

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

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Arc Melting of Alloys

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Titanium-Extrusion Studies

G. H. Schippereit, A. M. Sabroff, P. D. Frost, and J. H. Jackson.

Welding Studies

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ABSTRACT

Suitable heat treatments for the high strength alloys have been developed. By varying the heat treatment it has been possible to obtain tensile strengths of 150,000 psi with an elongation of 25% in one inch. Solution treating at higher temperatures generally increases the strength but with a corresponding loss in ductility. It now seems commercially probable to solution treat, machine, and subsequently age, thus producing alloys with a high strength level.

A new phase, called omega, has been discovered by X-Ray diffraction studies. This omega phase seems to be responsible for the loss in ductility, or embrittlement which accompanies an increase in tensile strength upon heat treatment. Results indicate that the omega phase vanishes after a certain time at elevated temperatures. Therefore, the omega phase may be a transition product from beta to alpha. The omega phase is pseudocubic in nature and localized increased concentrations of alloying elements indicate that the omega phase is lower in alloy content than the original alpha. The omega phase definitely appears to be connected with the high hardness characteristics of beta stabilized alloys.
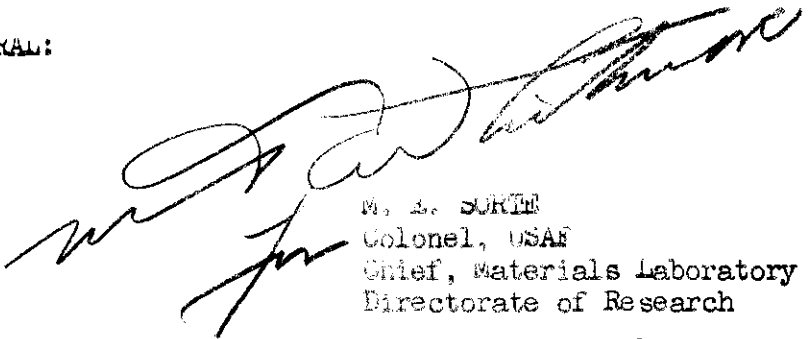
A significant development has been the production of ductile arc welds in alpha-beta alloys; varying degrees of ductility have been obtained in alpha-beta alloys by annealing or tempering after welding.

A program has been initiated whereby industrial concerns will evaluate the newly developed alloys of titanium for large scale usage.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDING GENERAL:

M. E. SORTE
Colonel, USAF
Chief, Materials Laboratory
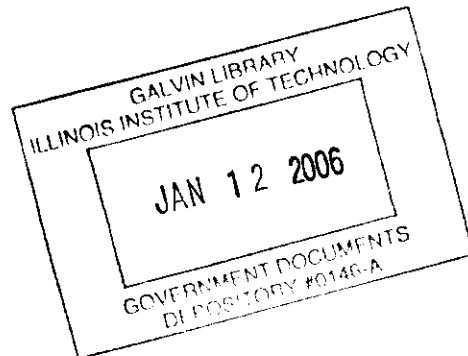Directorate of Research

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SUMMARY REPORT

Covering the Period May 19, 1952, to December 7, 1952

on

DEVELOPMENT OF TITANIUM-BASE ALLOYS

Contract No. AF 33(038)-3736

to

WRIGHT-PATTERSON AIR FORCE BASE
OHIO

from

BATTELLE MEMORIAL INSTITUTE

December 31, 1952

INTRODUCTION

This is the Final Report on Contract No. AF 33(038)-3736 covering the work done during the period from May 19, 1952, to December 7, 1952. The termination date of this contract was December 31, 1952.

This alloy-development program was started under Contract No. W 33-038 ac-21229, May 18, 1948. The present contract became effective May 18, 1949. In addition to this report, four previous summary reports on alloy development have been issued under these contracts:

Summary Report-Part III, July 30, 1949
AF Technical Report No. 6218-Part II, June 30, 1950
AF Technical Report No. 6623, June 18, 1951
WADC Technical Report No. 52-249, June 18, 1952

This work is being continued under Contract No. AF 33(616)-384 which started December 5, 1952.

SUMMARY

Evaluation of Selected Alloys

During the past six months, the major effort on this project was directed toward the development of heat treatments for promising high-strength alloys. The nominal compositions of the eight alloys finally selected for this investigation are as follows:

> 3.5Cr-3.5V
> 5Mn-2.5Cr
> 1Cr-1Fe-3Mn-1Mo-1V
> 1Cr-4Fe-1Mn-1Mo-1V
> 3.5Cr-3.5Mn
> 4.5Mo-3.5Fe
> 5Mn-2Mo
> 15Cr

A number of exploratory heat treatments have been applied to both 14-gage sheet and 1/2-inch-round bar stock of the first four compositions. These compositions were melted as five-, ten-, or twenty-pound ingots and fabricated to the desired shape by forging and rolling. Two different rolling temperatures, 1450 and 1600 F, were used for the 1/2-inch-round bar stock.

The first four alloys selected had good intermediate strength properties when heated at 1300 F and quenched or air cooled. In this condition, strengths of the order of 150,000 psi with elongations up to 25 per cent in one inch were obtained. Sheet specimens so treated all had a minimum bend radius of 1 T or less. Solutions treating at higher temperatures increased the strength of the alloys but, in general, lowered the ductility disproportionately.

Long-time aging treatments applied to solution-treated bar stock of three of the alloys produced mechanical properties much superior to any obtained previously in all of the alloys investigated under this contract. Excellent high strength properties were produced in the Ti-3.5Cr-3.5V, Ti-5Mn-2.5Cr, and Ti-3Mn complex alloys by various combinations of solution treatment at 1300 or 1400 F and aging for 24 and 52 hours at 800 F or 8 and 24 hours at 900 F. The latter alloy had particularly good properties, ranging from tensile strengths of 162,000 psi with 21 per cent elongation to 225,000 psi with 6 per cent elongation. The properties obtained on the three alloys are presented in bar-chart form in Figures I, II, and III.

In the past, when solution-treated alloys were aged at temperatures up to 700 F and for short times at 800 and 900 F, they usually had high strength but very low ductility. As a result of careful X-ray diffraction studies, a new phase, tentatively called omega phase, was discovered which is believed to be partly or wholly responsible for this embrittlement. The omega phase disappears after prolonged aging and the ductility of the alloys increases.
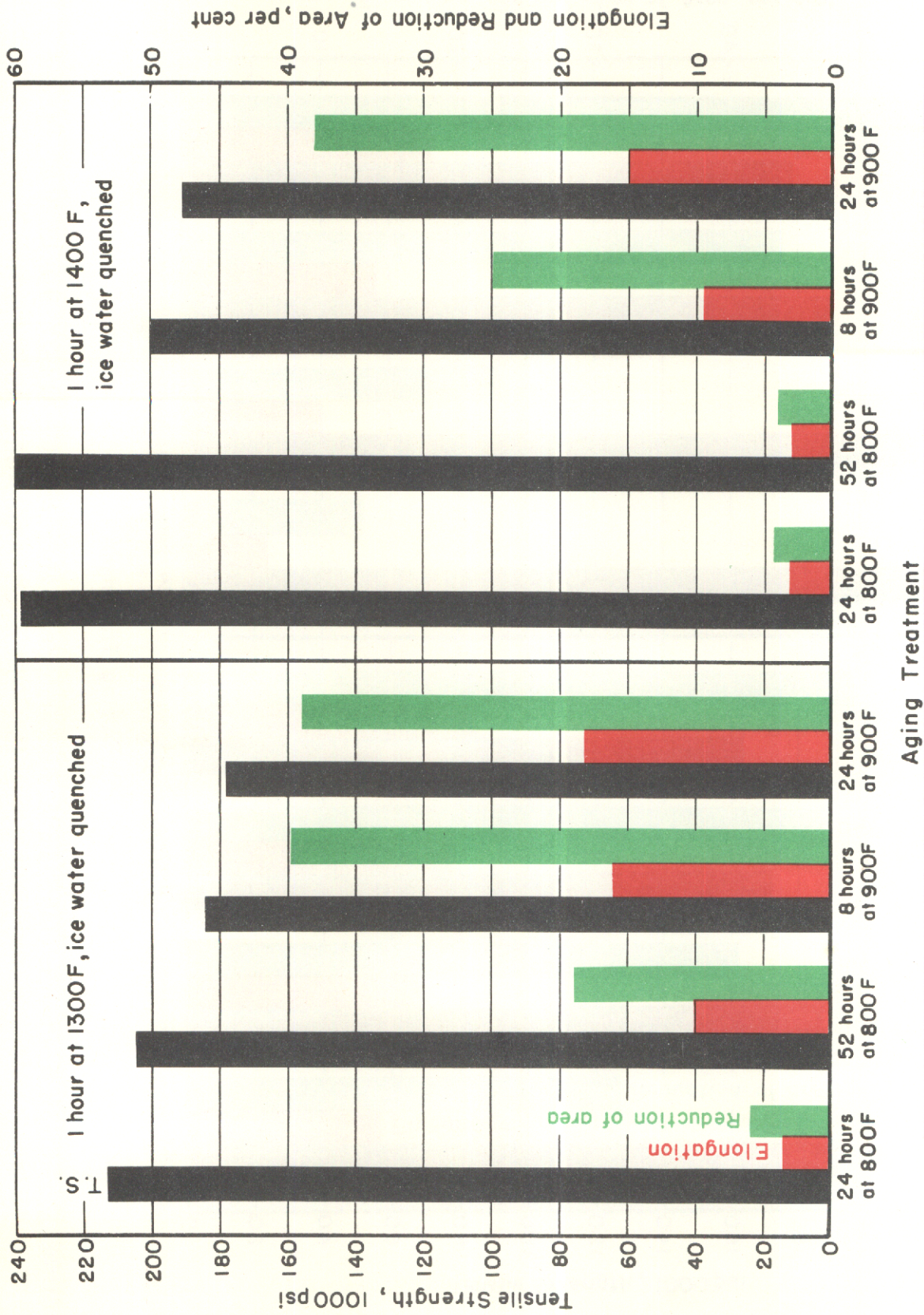
A-4053

FIGURE I. TENSILE PROPERTIES OF A Ti–5 Mn–2.5 Cr ALLOY ROLLED AT 1450F TO $\frac{1}{2}$–INCH ROUND AND SOLUTION TREATED AND AGED AS SHOWN
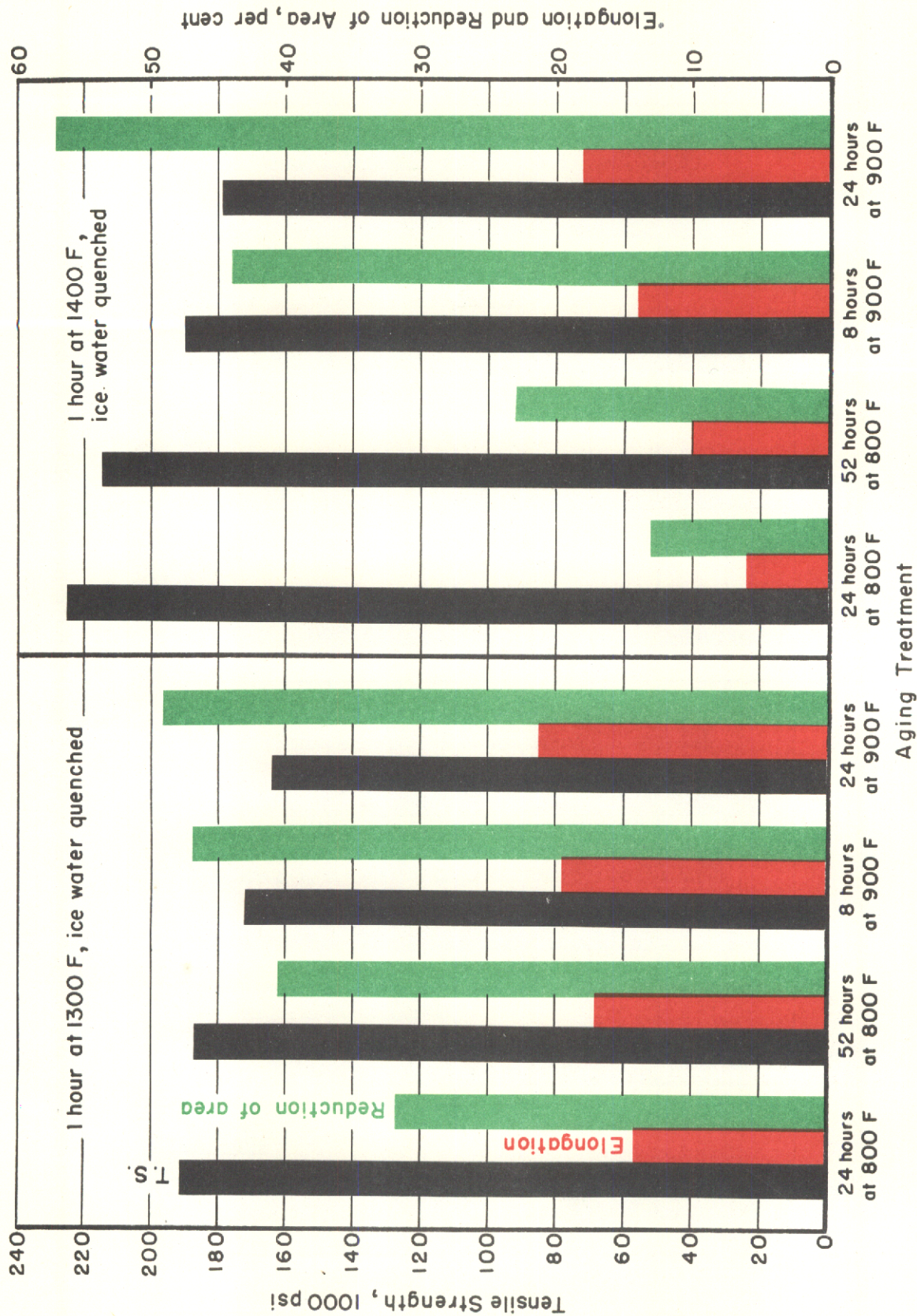
FIGURE II. TENSILE PROPERTIES OF A Ti-1Cr-1Fe-3Mn-1Mo-1V ALLOY ROLLED AT 1450 F TO ½-INCH ROUND AND SOLUTION TREATED AND AGED AS SHOWN

A-4054
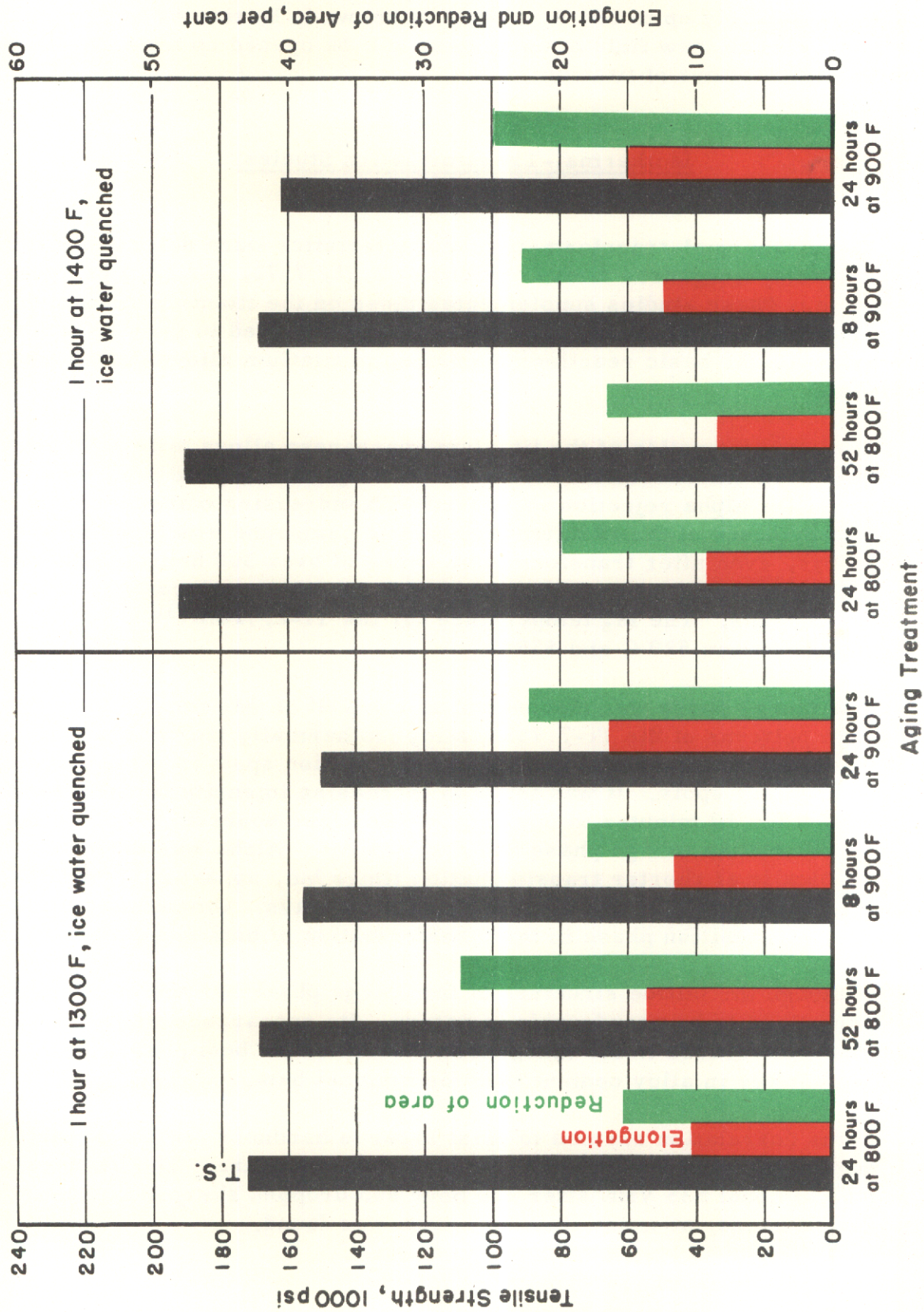
FIGURE III. TENSILE PROPERTIES OF A Ti-3.5Cr-3.5V ALLOY ROLLED AT 1450 F TO $\frac{1}{2}$-INCH ROUND AND SOLUTION TREATED AND AGED AS SHOWN

A-4055

The long-time aging treatments have excellent potentialities as commercial heat treatments. Thus, it may now be possible to solution treat an alloy at, say, 1300 F, machine or form a part, and age harden the part to a high strength level. It is also very possible that alloys given this type of heat treatment will be stable for extended periods of time at elevated temperatures, possibly up to 600 F. If this is shown to be true by work now in progress, a whole new field of usefulness will be opened up for high-strength, beta-stabilized alloys of titanium.

Isothermal-Transformation Studies

The isothermal-transformation characteristics were determined for three titanium-manganese alloys containing 2.91, 7.72, and 12.3 per cent manganese. These studies supplemented those on the titanium-chromium alloys described in WADC-TR-52-249 and were designed to provide additional information on the basic reactions occurring in titatium alloys during heat treatment.

The transformation of the titanium-manganese alloys differed from that of the titanium-chromium alloys in two respects: (1) the time for initiation of the alpha rejection increased with increasing manganese content, and (2) no evidence of the titanium-manganese compound was found in any of the alloys, even after transformation times of over 300 hours. Otherwise, the two alloy systems behaved similarly with respect to microstructures and hardness changes. The M_S temperature for the Ti-2.91Mn alloy was established between 520 C and 535 C.

The omega phase was discovered as a result of this work. X-ray diffraction patterns of the Ti-7.74Mn alloy isothermally transformed at 450 C revealed the new phase as discrete diffraction spots in close proximity to the beta-phase spots. It was found in increasing intensity in specimens transformed for 10 minutes to one hour. After four hours at 450 C, alpha was present and the omega phase had disappeared. Alpha was detected microscopically at shorter transformation times but, apparently, was not present in sufficient quantity to be detected by X-rays. Omega may be, therefore, a transition phase in the transformation of beta to alpha.

Although the lattice structure of the omega phase has not been determined yet, it appeared to be pseudocubic in nature. Its appearance was accompanied by localized increases in the manganese content of the beta phase, indicating that it was lower in alloy content than the original beta.

Some correlation seemed to exist between hardness and the presence of the omega phase at the 450 C transformation temperature. The hardness increased to about 535 VHN after one hour and dropped sharply to about 475 VHN after 4 hours, at which time no omega was found.

Hardening Mechanism in Beta-Stabilized Titanium Alloys

In order to understand more fully the reactions occurring in beta-stabilized alloys during heat treatment, a limited investigation of the hardening mechanism was initiated. Since it is believed generally that a coherency or age-hardening reaction is involved, initial work was done on age-hardened titanium-chromium, titanium-molybdenum, and titanium-iron alloys. The progress of the hardening was followed by means of electron microscopy and X-ray diffraction.

A very fine precipitate was found in quenched and fully age-hardened titanium-chromium and titanium-molybdenum alloys. In the unaged condition, only traces of the precipitate appeared. Fully hardened titanium-iron alloys did not show a precipitate, but this may be due to the etching techniques used.

The new omega phase was found by X-ray diffraction in the fully hardened specimens of alloys of all three systems. It was also found in a Ti-4Fe alloy, as quenched in iced brine, and in a Ti-8Cr alloy as quenched in liquid nitrogen. Both of these alloys had high as-quenched hardnesses. The omega phase found in these age-hardened alloys appeared to be isomorphous with the phase found in the isothermally transformed Ti-8Mn alloy. The structure and composition of this phase are as yet unknown. However, omega appears definitely to be connected with the high hardnesses attainable in beta-stabilized alloys.

The X-ray results suggest that the precipitate found in electron micrographs of age-hardened titanium-chromium and titanium-molybdenum alloys is the omega phase, but this is still uncertain.

The apparent relationship between the omega phase and high hardness does not rule out other hardening mechanisms, such as alpha nuclei coherent with the beta matrix or with the omega phase. These possibilities are being investigated under the new contract.

Weldability Studies

One of the significant developments during the past six months was the production of ductile arc welds in alpha-beta alloys. Varying degrees of ductility were attained in alpha-beta alloys having intermediate and high strengths in the as-hot-rolled condition by annealing after welding. A metastable beta Ti-6Fe-8Mo alloy also had some ductility as welded. Microscopic examination of the welds showed some correlation between weld ductility and freezing segregation in the alloys. This investigation was very limited in scope but will be expanded during the coming year.

Extrusion of Titanium

During the past six months, approximately 820 pounds of titanium, comprising 40 billets of about 15 pounds each, were melted, fabricated, and shipped to Metal Trims, Incorporated, of Youngstown, Ohio, for extrusion. It is expected that the first experimental extrusions will be made during January, 1953.

Evaluation of Selected Alloys by Outside Companies

Experimental tube-drawing tests are being conducted at Superior Tube Company, Norristown, Pennsylvania, on 1/2-inch-round bar stock of four selected alloys. The material is being cold drawn in the annealed condition, with intermediate anneals between each drawing operation. Total reductions in area up to 35 per cent have been achieved to date. However, some cracking on the internal surfaces has been noted. Tests are being continued.

Two 20-pound ingots of the Ti-1Cr-1Fe-3Mn-1Mo-1V alloy have been sent to the Wyman-Gordon Company, Worcester, Massachussetts, for closed-die forging tests. A small aircraft part will be made from these ingots in order to evaluate the forgeability of the alloy.

Five 20-pound ingots of the same alloy are being melted and will be forged at Battelle into rolling slabs. These will be sent to the Republic Steel Corporation at Massillon, Ohio, for rolling into sheet for evaluation by aircraft companies.

During the next year, ingots of the selected alloys, weighing up to 100 to 300 pounds, will be prepared for evaluation by industrial organizations.

EVALUATION OF EXPLORATORY TITANIUM-IRON ALLOYS

Data obtained on a number of complex titanium-base alloys have indicated that iron is a very potent strengthener of titanium. It appears to be most effective when added in amounts up to 4 per cent. Since previous work done on binary titanium-iron alloys did not cover this range of composition adequately, a new series of alloys containing from 2 to 6 per cent iron were melted as 1-pound ingots and fabricated to 14-gage sheet for testing. Additional heats containing 7.5, 10, and 15 per cent iron were included in the series to check the results of earlier work. Sheet specimens of each alloy were tested in the as-hot-rolled condition and after various process-annealing treatments.

In the as-hot-rolled condition, the strength of these alloys increased with increasing iron content to a maximum of about 206,000 psi at 6 per cent iron, as shown in Table 1. The ductility decreased with increasing alloy content to a value of about 2 per cent in 1 inch at the 206,000-psi strength level. Alloys containing 10 and 15 per cent iron were extremely brittle and could not be tested. The results of the tests on the binary titanium-iron alloys are presented graphically in Figure 1.

Tensile blanks cut from the as-hot-rolled sheet were subjected to heat treatments which consisted of heating for 1 hour in an argon atmosphere at temperatures of 1150, 1250, 1350, and 1450 F, followed by cooling in still air. Complete tensile and hardness data for the heat-treated specimens are presented in Table 1. Again, the 10 and 15 per cent iron alloys were too brittle to test after all heat treatments. The hardness data for the Ti-10Fe alloy are included in the table to show the relatively low hardness level at which this alloy exhibited low ductility.

A minimum in strength was attained in all except the Ti-6Fe alloy by annealing at 1150 or 1250 F. The ductility of all except the 6 per cent iron alloy reached a maximum after the 1250 F anneal. The Ti-6Fe alloy attained its minimum strength after the 1450 F anneal and its maximum ductility after the 1150 F anneal. In all cases, the strength of the alloys after annealing was considerably lower than that obtained in the as-hot-rolled condition. Contrary to expectations, the strength and hardnesses of the alloys containing from 2 to 6 per cent iron showed no large increases at annealing temperatures above 1250 F. However, the ductility dropped off very sharply at the higher annealing temperatures. The majority of the alloys had very low ductilities at relatively low hardness levels after the 1450 F annealing treatment. No correlation between microstructure and mechanical properties could be found in these specimens.

The extreme brittleness of the 10 and 15 per cent iron alloys in both the as-hot-rolled and annealed conditions was very puzzling. As mentioned previously, the hardnesses of the 10 per cent iron alloy after the annealing treatments were relatively low. The microstructure of this alloy was essentially all beta as hot rolled and after annealing at temperatures of 1250 F and higher. At 1150 F, some alpha was present. The 15 per cent iron alloy was essentially 100 per cent beta in all conditions, and had hardnesses above 400 VHN. In contrast to the highly ductile, Ti-15Cr alloy, however, the corresponding iron composition was very brittle in all conditions.

The properties of the titanium-iron alloys were not particularly good in any of the conditions described. No further work is contemplated on these alloys at the present time.

TABLE 1. PROPERTIES OF BINARY Ti-Fe ALLOYS

Heat No.	Intended Composition, %	As Hot Rolled at 1450 F			Heated One Hour at 1150 F		
		Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	VHN	Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	VHN
WT98A	2Fe	123,400	17.0	306	105,300	5.0	241
WT100A	3Fe	143,000	12.5	325	119,700	13.0	292
WT99A	4Fe	157,700	13.0	351	126,900	11.0	261
WT101A	5Fe	180,500	4.5	403	138,500	3.5	333
WT97A	6Fe[3]	206,300	2.0	430	163,800	14.0	384
WT103A	7.5Fe	196,300	1.0	453	156,600	2.0	303
WT96A	10.0Fe	(4)	--	--	(4)	--	351

(1) All heat treatments were carried out in a dried-argon atmosphere.

(2) Substandard specimen, 0.375 x 0.064 x 5 inches, with a reduced section 0.250 x 0.064 x 1-1/4-inches.

(3) Actual composition.

(4) Specimens too brittle to test.

IN HOT-ROLLED AND HEAT-TREATED CONDITIONS

| Temperature, Air Cooled[1] | | | | | | | | |
| 1250 F | | | 1350 F | | | 1450 F | | |
Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	VHN	Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	VHN	Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	VHN
102,600	19.0	244	110,500	14.0	234	112,000	1.0*	248
119,000	20.0	282	128,300	14.0	317	131,000	2.0	308
126,300	19.0	275	142,700	8.0	287	143,900	4.0*	297
141,800	14.0	342	150,800	3.0	401	153,100	1.0	497
168,300	4.5	369	168,700	2.5	366	151,200	2.0	358
153,100	4.0	312	187,500	1.5	336	(4)	--	357
(4)	--	363	(4)	--	366	(4)	--	374
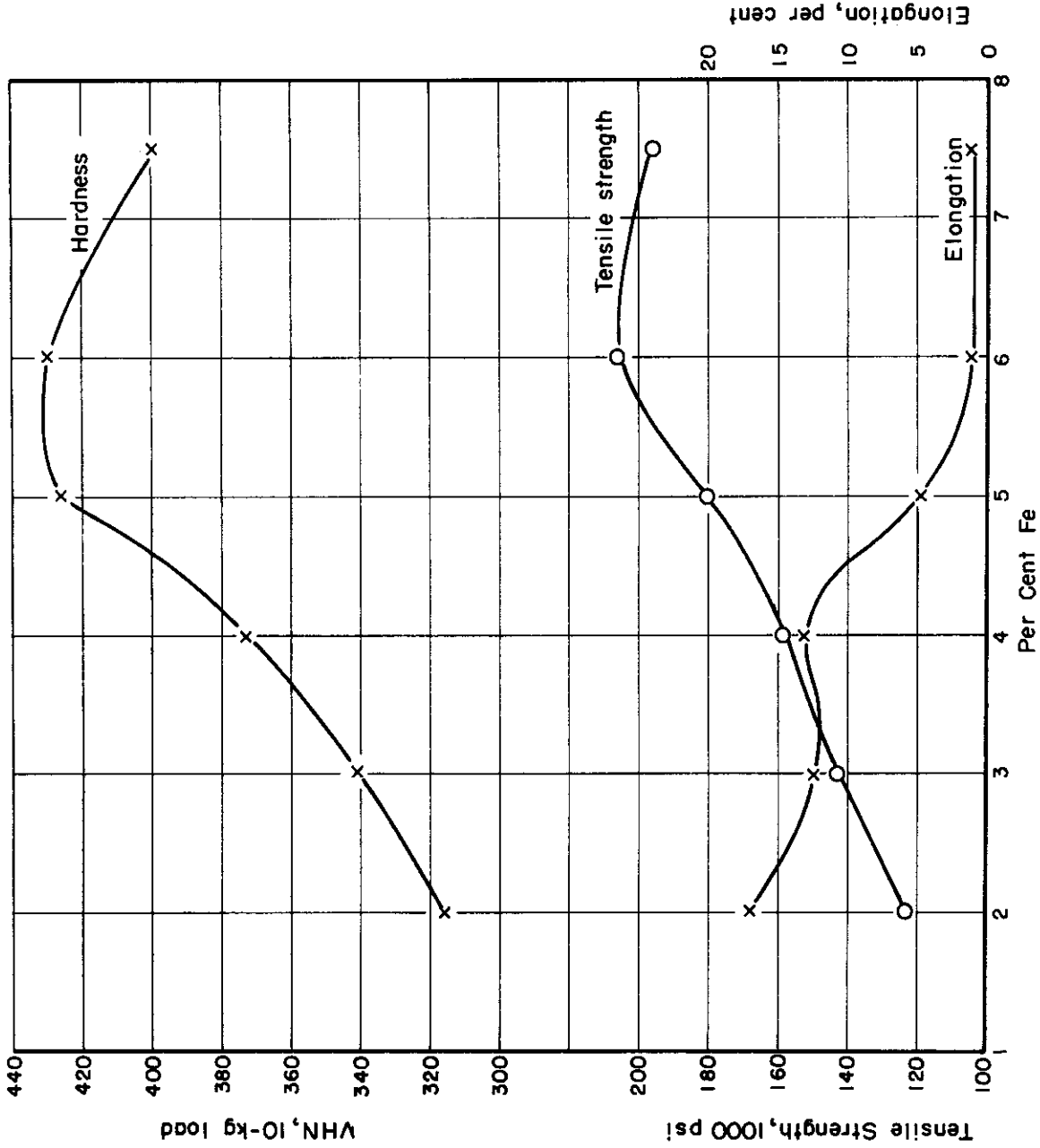
FIGURE 1. MECHANICAL PROPERTIES OF Ti-Fe ALLOYS AS HOT ROLLED AT 1450 F TO 0.064-INCH SHEET

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REPRODUCIBILITY OF PROPERTIES IN SELECTED ALLOYS

In the interim between the end of last year's exploratory alloy program and the investigation of larger ingots of the selected alloys, a limited investigation of the reproducibility of properties from heat to heat in various selected alloys was carried out. The following compositions were chosen for study:

> 3.5Cr-3.5V
> 2.5Cr-5Mn
> 3.5Cr-3Mn
> 5Cr-1.5Fe
> 5Mn-2Fe
> 5Mn-2Mo
> 5Mo-4Fe

Two one-pound ingots of each of these compositions were double melted and fabricated to 14-gage sheet, using the standard upset-reduction forging technique and the rolling process described in Appendix II of WADC-TR-52-249.

The as-hot-rolled properties of both heats and the properties after various annealing treatments for one heat of each alloy were presented in the aforementioned report. During the period covered by the present report, tests were completed on the second heat of each composition. The heat treatments applied to the second heats consisted of heating for one-half hour at 1400 or 1500 F, followed by air cooling. The complete properties of all the heats used in this investigation are given in Table 2.

As mentioned in the earlier work, the properties, as hot rolled, of heats of the same nominal compositions varied considerably. Chemical analyses also showed appreciable variations in the two heats in some cases. However, even where chemical analyses were essentially the same, the properties as hot rolled differed widely. For example, in the nominal Ti-5Mn-2Fe alloy, the chemical compositions of the two heats were very similar. However, Heat WS281A was extremely brittle in the as-rolled condition, as shown by the low tensile strength and zero ductility, while Heat WS294A had good ductility and high strength. Therefore, the variations observed in the duplicate heat, as hot rolled, were probably due, in large part, to variations in fabrication procedures.

It may be seen from Table 2 that the heat treatments at 1400 F produced much more consistent properties among heats of the same nominal compositions. Two compositions, Ti-2.5Cr-5Mn and Ti-5Mo-4Fe, had very good high-strength properties after this heat treatment. The 1500 F treatment resulted in a considerably wider spread in properties between duplicate heats, in some cases. In general, the properties were also somewhat poorer than those developed at 1400 F.

TABLE 2. COMPARISON OF PROPERTIES OF DUPLICATE HEATS OF SELECTED ALLOYS IN HOT-ROLLED[4] AND HEAT-TREATED CONDITIONS

Heat No.	Intended Composition, %	Actual Composition, %	As Hot Rolled at 1450 F			Heated 1/2 Hour at 1400 F, Air Cooled [2]			Heated 1/2 Hour at 1500, Air Cooled [2]		
			Ultimate Tensile [1] Strength, psi	Elong, % in 1 inch	VHN	Ultimate Tensile [1] Strength, psi	Elong, % in 1 inch	VHN	Ultimate Tensile [1] Strength, psi	Elong, % in 1 inch	VHN
WS285A	3.5Cr, 3.5V	3.37Cr, 3.00V	176,500[3]	10.0	373	158,900	15.5	367	153,400	11.0	355
WS304A	3.5Cr, 3.5V	4.15Cr, 3.01V	186,100	4.5	373	165,600	11.0	360	144,300	11.5	342
WS282A	2.5Cr, 5Mn	2.13Cr, 5.02Mn	211,400[3]	3.0	421	184,400	6.0	422	188,900	0.0	401
WS291A	2.5Cr, 5Mn	2.35Cr, 4.28Mn	186,000	8.0	390	184,800	8.5	381	175,200	6.0	396
WS289A	3.5Cr, 3Mn	3.09Cr, 2.74Mn	197,700[3]	5.0	418	168,700	13.5	376	163,200	8.5	366
WS305A	3.5Cr, 3Mn	2.93Cr, 2.52Mn	185,000	4.0	387	160,600	11.5	346	142,100	12.5	323
WS296A	5Cr, 1.5Fe	4.30Cr, 1.43Fe	187,100[3]	6.5	421	165,900	12.0	370	156,800	11.0	357
WS301A	5Cr, 1.5Fe	4.87Cr, 1.52Fe	211,600	1.5	397	179,600	9.5	390	175,000	7.0	382
WS281A	5Mn, 2Fe	4.46Mn, 1.87Fe	68,700[3]	0.0	459	183,800	2.5	391	182,700	8.0	369
WS294A	5Mn, 2Fe	4.64Mn, 1.88Fe	188,000	6.0	421	184,000	4.0	399	164,100	2.0	430
WS286A	5Mn, 2Mo	4.78Mn, 1.87Mo	192,600[3]	4.0	373	172,200	9.0	362	170,200	5.0	417
WS299A	5Mn, 2Mo	4.38Mn, 2.04Mo	195,000	4.0	366	162,200	11.5	335	158,400	9.0	342
WS287A	5Mo, 4Fe	4.79Mo, 3.43Fe	203,200[3]	6.0	413	181,000	11.0	376	168,800	1.0	445
WS290A	5Mo, 4Fe	4.67Mo, 3.55Fe	201,400	4.0	387	189,300	8.0	384	143,100	1.0	489

(1) Substandard specimen, 0.375 x 0.064 x 5 inches with a reduced section 0.250 x 0.064 x 1-1/4 inches.
(2) Heating was carried out in a dried-argon atmosphere.
(3) Only one specimen tested. All other data are the averages of two specimens.
(4) Hot rolled at 1450 F to 14-gage (0.064-inch) sheet.

EVALUATION OF SELECTED ALLOYS IN LARGER INGOT SIZES

To date, this evaluation has consisted of determining the response to heat treatment of four high-strength alloys melted as 5-, 10-, and 20-pound ingots and fabricated to 14-gage sheet or 1/2-inch-round bar stock. The four alloys investigated thus far had the following nominal compositions:

3.5Cr-3.5V
5Mn-2.5Cr
1Cr-1Fe-3Mn-1Mo-1V
1Cr-4Fe-1Mn-1Mo-1V

In addition to this heat-treatment program, an investigation of the cold-drawing properties of these alloys at the Superior Tube Company, Norristown, Pennsylvania, has been started.

The evaluation of the response of the alloys to heat treatments thus far has been confined to tensile and hardness tests after various heat treatments, including solution-treating and aging experiments. Excellent high-strength properties were attained in bar stock of three of the alloys by the latter type of treatment. It is highly possible that a good degree of stability at elevated temperatures may be achieved in these alloys by the overaging treatments employed. Thermal-stability tests are in progress.

Fabrication

The 5-pound ingots described herein were fabricated to 14-gage sheet. The ingots, about 2-1/2 inches in diameter by 5 inches long, were cut into approximately equal sections. Each section was then fabricated according to the upset-forging technique described in Appendix II of WADC-TR-52-249. Forging was done at 1750 to 1800 F, and rolling was carried out at 1450 F.

The 10-pound ingots were fabricated into 1/2-inch-round bar stock. Forging consisted of upsetting slightly to prevent end cracking and drawing out into a 1-1/8-inch-square bar at 1750 to 1850 F. This bar was cut into sections and given six passes at 1600 F in a set of rolls designed for mild steel. These rolls had round, square, and elliptical grooves. Thus, each pass involved a reduction and a change of shape; the bars were reheated between each pass.

The 20-pound ingots were also fabricated to 1/2-inch-round bar stock. These ingots were forged to 3/4-inch-square bars, which were then rolled to 9/16-inch squares in a set of rolls providing continuous reduction in square form. The 9/16-inch-square bars were then put through the last three passes in the rolls used for the 10-pound ingots. The rolling temperature was 1450 F.

Properties As-Hot-Rolled

Sheet Specimens

The properties as-hot-rolled of the 14-gage sheet fabricated from the 5-pound ingots were presented in Table 30 of WADC-TR-52-249. For convenience, they will be repeated in certain tables in this section.

One-Half-Inch-Round Bar Stock

The tensile properties and hardnesses of the bar stock rolled from both the 10- and 20-pound ingots are presented in Table 3. Actual chemical analyses taken from particular tensile specimens of each heat are presented also. The two rolling temperatures, 1600 and 1450 F, were used to obtain different grain sizes in each alloy. At 1600 F, all of these alloys were in the beta-phase region, and the as-rolled structure had a relatively large grain size. At 1450 F, on the other hand, the alloys were in the alpha-beta-phase field and a considerably finer grain structure was produced. The different rolling temperatures also resulted in different configurations of the alpha phase after subsequent heat treatment in the alpha-beta-phase field, as will be shown later in this section.

The chemical analyses shown in Table 3 were quite consistent, both at different locations within a single heat and among heats of the same nominal compositions. The tungsten analyses shown for Heat WT125A are not to be considered typical for all heats in this table. This particular heat had a very high electrode loss during melting, and was expected to have a rather high tungsten content. The remaining heats had normal electrode losses, and were estimated to have tungsten contents of the order of 0.2 per cent or less.

The Ti-5Mn-2.5Cr alloy and the Ti-4Fe complex alloy consistently developed high strength (greater than 180,000 psi) in the as-hot-rolled condition. The Ti-3.5Cr-3.5V alloy and the Ti-3Mn complex alloy had intermediate strengths. In general, the tensile properties developed in a single heat were reasonably consistent. A major exception to this was the Ti-4Fe complex alloy rolled at 1600 F (Heat WT86A). This alloy had reasonably consistent tensile strength, but the elongation varied from 1 to 11 per cent in different parts of the ingot. Comparison of the properties obtained at the two rolling temperatures indicated, in general, that the heats rolled at 1450 F developed equal or higher tensile strength and considerably better ductility than the same compositions rolled at 1600 F.

TABLE 3. PROPERTIES, AS HOT ROLLED, OF SELECTED
HIGH-STRENGTH TITANIUM ALLOYS

Specimens Machined from 1/2-Inch-Diameter Bar Stock

Heat No.	Intended Composition, %	Actual Composition, %	Rolling Temp, F	Ultimate Tensile Strength, psi(3)	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT42A(1)	3.5Cr, 3.5V	4.71Cr, 3.46V	1600	158,900	11.0	18.0	351
			1600	158,400	11.0	21.0	341
		4.52Cr, 3.23V	1600	155,300	10.5	13.0	336
WT125A(2)	3.5Cr, 3.5V	4.52Cr, 3.22V, 0.78W	1450	167,600	11.5	16.0	342
			1450	163,700	17.0	39.0	351
			1450	167,000	17.0	46.0	351
			1450	165,600	17.0	34.6	345
		4.49Cr, 3.21V, 0.81W	1450	167,000	14.0	23.0	351
			1450	164,800	18.0	40.7	345
WT51A(1)	5.0Mn, 2.5Cr	5.18Mn, 2.64Cr	1600	207,700	1.0	2.8	421
			1600	200,600	2.0	3.0	418
			1600	213,300	1.5	5.5	428
WT132A(2)	5.0Mn, 2.5Cr	5.23Mn, 2.62Cr	1450	207,500	11.0	16.2	421
		5.60Mn, 2.8Cr	1450	202,000	7.0	12.4	413
			1450	185,100	13.0	27.4	383
			1450	171,900	14.0	34.8	383
			1450	199,200	8.0	26.8	409
			1450	183,300	12.5	28.9	383
			1450	182,700	12.0	37.4	397

TABLE 3. (Continued)

Heat No.	Intended Composition, %	Actual Composition, %	Rolling Temp, F	Ultimate Tensile Strength, psi(3)	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT107A(1)	3.0Mn, 1.0Fe 1.0Cr, 1.0Mo, 1.0V	1.36Cr, 0.99Fe, 2.60Mn, 0.88 Mo, 0.98V	1600	166,500	13.5	--	363
			1600	166,500	15.0	--	363
			1600	166,000	14.5	--	363
			1600	163,400	16.0	--	363
		1.41Cr, 0.99Fe, 3.30Mn, 0.95Mo, 0.98V	1600	167,000	15.5	--	366
			1600	164,500	15.0	--	363
			1600	162,400	14.5	--	363
WT136A(2)	1Cr, 1Fe, 3Mn, 1Mo, 1V	1.04Cr, 0.99Fe, 3.0Mn, 0.90Mo, 0.94V	1450	188,400	18.0	33.6	375
			1450	196,500	10.0	38.6	--
			1450	189,900	9.0	22.6	383
WT86A(1)	1Cr, 4Fe, 1Mn, 1Mo, 1V	1.05Cr, 1.01Fe, 3.2Mn, 0.89 Mo, 0.91V	1450	169,000	20.0	49.4	330
			1450	177,200	17.0	47.6	383
			1450	174,600	13.0	24.0	387
		1.22Cr, 3.15Fe, 0.99Mn, 0.92Mo, 0.92V	1600	196,500	1.0(4)	1.6	413
			1600	194,000	3.0	5.5	381
		1.21Cr, 3.15Fe, 1.00Mn, 0.97Mo, 1.00V	1600	187,900	5.0	6.3	397
			1600	187,900	11.0	21.0	384

(1) Ten-pound, 4-inch-diameter ingot.
(2) Twenty-pound, 4-inch-diameter ingot.
(3) Standard 0.250-inch-diameter round specimen.
(4) Specimen broke outside gage marks – uniform elongation recorded.

Heat Treatments

Definition of Terms

The terms used to describe various heat treatments for other metals do not describe accurately the treatments used for titanium alloys. However, for the purposes of this report, the following terms will be used.

Solution Treating. This term signifies a heat treatment consisting of heating for any length of time in the alpha-beta or beta-phase field, followed by a reasonably fast cool, such as is provided by air, ice water, boiling water, brine, or liquid nitrogen.

Age Hardening. This type of heat treatment consists of reheating solution-treated specimens in the temperature range from 200 to 1000 F for a suitable length of time. It may be followed by either air cooling or water quenching.

Solution Treatments

14-Gage Sheet. The results of solution treatments at temperatures in the range from 1300 to 1500 F carried out on sheet specimens of the four selected alloys were given in Table 31 of WADC-TR-52-249. In general, heating at 1300 F, followed by air cooling, resulted in strengths of the order of 135,000 to 150,000 psi, with elongations ranging from about 12 to 21 per cent in 1 inch. The minimum bend radius after this treatment was 1T* or less in alloys. Solution treating at 1400 F resulted in somewhat higher strength with correspondingly lower ductility. Strengths as high as 190,000 psi in the Ti-4Fe complex alloy were attained by the 1400 F treatment. Solution treating in the temperature range from 1300 to 1400 F also resulted in reasonably consistent properties in a single alloy.

1/2-Inch-Round Bar Stock. The 1/2-inch-round bar stock of the four selected alloys was given solution treatments consisting of heating in the temperature range from 1300 to 1500 F, and quenching in either ice water or boiling water or air cooling. The quenching treatments were used to obtain cooling rates approaching those obtained in the air cooling of the sheet stock. Duplicate specimens cut from different bar sections of the same ingot were used for each treatment. The data obtained for each specimen are included in the table to give some indication of the consistency of the properties obtained by a given heat treatment. Complete results of tensile and hardness tests made on solution-treated specimens of the four selected alloys are given in Tables 4 through 10, inclusive, and the results are plotted graphically as

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*Where T is the thickness of the sheet.

TABLE 4. EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-3.5Cr-3.5V ALLOY ROLLED AT 1600 F TO 1/2-INCH-ROUND BAR STOCK

Heat WT-42A

Solution[1] Temp, F	Cooling Medium	Ultimate Tensile[2] Strength, psi	Elong, % in 1 Inch	Reduction of Area, %	VHN
1300	Ice water	161,400	9.0	--	388
"	Ditto	154,500	12.0	--	362
"	Boiling water	147,500	16.5	--	353
"	Ditto	152,000	15.0	--	348
"	Still air	164,000	10.0	--	388
"	Ditto	158,900	15.0	--	374
1350	Ice water	188,400	3.0	5.7	468
"	Ditto	188,400	4.0	10.1	464
"	Boiling water	185,800	2.0	1.6	390
"	Ditto	177,200	5.5	11.8	425
"	Still air	169,500	10.0	18.3	366
1400	Ice water	159,900	1.0[3]	--	480
"	Ditto	185,300	0.0[3]	--	461
"	Boiling water	181,300	1.0[3]	--	426
"	Ditto	187,900	2.0	--	421
"	Still air	164,300	11.0	--	358
"	Ditto	159,900	11.5	--	365
1450	Still air	168,400	10.0	16.9	354
"	Ditto	161,700	13.0	21.9	356

TABLE 4. (Continued)

Solution(1) Temp, F	Cooling Medium	Ultimate Tensile(2) Strength, psi	Elong, % in 1 Inch	Reduction of Area, %	VHN
1500	Ice water	122,200	0.0(3)	--	523
"	Ditto	124,700	0.0(3)	--	512
"	Boiling water	117,100	0.0(3)	--	527
"	Ditto	72,800(4)	0.0(4)	--	527
"	Still air	171,600	8.0	--	372
"	Ditto	164,300	8.0	--	356

(1) Heating was done in air; the time at temperature was 1 hour.
(2) Standard 0.250-inch-diameter round specimen.
(3) Specimen broke outside gage marks - uniform elongation recorded.
(4) Broke in threads.

TABLE 5.   EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-3.5Cr-3.5V ALLOY ROLLED AT 1450 F TO 1/2 -INCH-ROUND BAR STOCK

Heat WT-125A

Solution[1] Temp, F	Cooling Medium	Ultimate Tensile[2] Strength, psi	Elong, % in 1 inch	Reduction of Area, %	VHN
1300	Ice water	154,900	13.0	50.4	351
"	Ditto	162,300	10.0	27.6	373
"	Boiling water	161,500	9.0	22.7	363
"	Ditto	159,300	13.0	37.3	357
"	Still air	173,000	8.0	11.3	390
"	Ditto	167,600	9.0	12.1	387
1350	Ice water	200,400	1.0	1.0	409
"	Ditto	202,400	1.0	1.0	405
"	Boiling water	194,700	1.0	1.6	391
"	Ditto	186,600	2.0	3.2	394
"	Still air	183,300	10.0	19.1	380
"	Ditto	178,200	5.0	7.9	390
1400	Ice water	173,700	(3)	(3)	464
"	Ditto	178,600	0.5	0.0	433
"	Boiling water	165,300	(3)	(3)	478
"	Ditto	196,000	1.0	0.0	421
"	Still air	170,100	6.0	8.2	370
"	Ditto	170,600	9.5	11.3	363

TABLE 5. (Continued)

Solution(1) Temp, F	Cooling Medium	Ultimate Tensile(2) Strength, psi	Elong, % in 1 Inch	Reduction of Area, %	VHN
1450 F	Ice water	(4)	(4)	(4)	503
	Ditto	182,500	0.0	0.0	493
	Boiling water	166,200	0.0	0.0	468
	Ditto	(4)	(4)	(4)	519
	Still air	162,000	6.0	9.4	351
	Ditto	157,600	8.0	12.4	345
1500	Ice water	(4)	(4)	(4)	503
"	Ditto	182,600	1.0	0.8	503
"	Boiling water	147,800	1.0	0.8	514
"	Ditto	167,700	(3)	(3)	508
"	Still air	161,600	16.0	41.9	345
"	Ditto	156,600	9.0	14.3	351

(1) Heating was done in air; the time at temperature was 1 hour.
(2) Standard 0.250-inch-diameter round specimen.
(3) Broke in shoulder radius.
(4) Broke in threads.

TABLE 6. EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-5.0Mn-2.5Cr ALLOY ROLLED AT 1600 F TO 1/2-INCH-ROUND BAR STOCK

Heat Wt51A

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of Area, %	VHN
1300	Ice water	151,200	19.0	--	354
"	Ditto	150,200	20.5	--	353
"	Boiling water	147,700	20.5	--	359
"	Ditto	149,400	20.0	--	350
"	Still air	156,300	21.5	--	375
"	Ditto	153,500	20.0	--	376
1350	Ice water	170,600	9.5	25.4	383
"	Ditto	169,000	9.5	26.0	387
"	Boiling water	161,400	14.5	26.0	373
"	Ditto	159,400	16.0	33.6	351
"	Still air	193,500	3.0	3.2	363
"	Ditto	187,400	3.0	4.8	390
1400	Ice water	168,000	12.0	--	390
"	Ditto	166,000	14.5	--	376
"	Boiling water	170,400	1.5	--	405
"	Ditto	166,500	10.0	--	374
"	Still air	115,100(4)	0.0(3)	--	492
"	Ditto	145,100	0.5(3)	--	529
1450	Ice water	176,200	2.0	2.4	373
"	Ditto	159,500	1.0	4.8	373
"	Boiling water	179,600	3.0	--	401
"	Ditto	154,800	--	--	405

TABLE 6. (Continued)

Solution Temp, F[1]	Cooling Medium	Ultimate Tensile Strength, psi[2]	Elong, % in 1 inch	Reduction of Area, %	VHN
1500	Ice water	175,700	2.0	--	402
"	Ditto	174,600	3.5	--	371
"	Boiling water	153,000	0.0(3)	--	402
"	Ditto	169,600	7.0	--	388
"	Still air	(5)	(5)	--	532
"	Ditto	56,500	0.0	--	527

(1) Heating was done in air; the time at temperature was one hour.

(2) Standard 0.250-inch-diameter round specimen.

(3) Specimen broke outside gage marks - uniform elongation recorded.

(4) Broke in shoulder.

(5) Brittle specimen - broke during machining.

TABLE 7. EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-5.0Mn-2.5Cr ALLOY ROLLED AT 1450 F TO 1/2-INCH-ROUND BAR STOCK

Heat Wt 132A

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of Area, %	VHN
1300	Ice water	156,000	23.0	44.1	345
"	Ditto	154,200	24.0	42.3	363
"	Boiling water	152,700	20.5	44.8	339
"	Ditto	154,000	21.0	36.0	351
"	Still air	150,100	23.0	42.3	357
"	Ditto	155,300	23.0	44.1	357
1350	Ice water	173,500	11.0	34.2	367
"	Ditto	181,200	11.0	38.3	370
"	Boiling water	189,600	5.0	24.2	--
"	Ditto	168,200	17.0	37.4	345
"	Still air	187,400	8.0	19.7	405
"	Ditto	187,900	10.0	22.6	394
1400	Ice water	176,200	19.0	32.0	387
"	Ditto	174,100	15.0	26.8	397
"	Boiling water	177,200	10.5	21.9	380
"	Ditto	176,200	11.0	28.9	376
"	Still air	209,800	2.0	2.4	429
1450	Ice water	173,900	(3)	(3)	413
"	Ditto	166,700	(3)	(3)	455
"	Boiling water	164,300	(3)	(3)	429
"	Ditto	174,500	(3)	(3)	450
"	Still air	(4)	(4)	(4)	536
"	Ditto	(4)	(4)	(4)	525

TABLE 7. (Continued)

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of Area, %	VHN
1500	Ice water	185,700	3.5	3.0	437
"	Ditto	187,400	5.0	9.5	417
"	Boiling water	180,800	2.0	1.6	409
"	Ditto	159,200	(3)	(3)	409
"	Still air	(4)	(4)	(4)	503
"	Ditto	(4)	(4)	(4)	498

(1) Heating was done in air; the time at temperature was one hour.
(2) Standard 0.250-inch-diameter round specimen.
(3) Broke in shoulder radius.
(4) Broke in threads.

TABLE 8.   EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-1Cr-1Fe-3Mn-1Mo-1V ALLOY ROLLED AT 1600 F TO 1/2-INCH-ROUND BAR STOCK

Heat Wt107A

Solution Temp, F[1]	Cooling Medium	Ultimate Tensile Strength, psi[2]	Elong, % in 1 inch	Reduction of Area, %	VHN
1300	Ice water	149,400	19.0	--	348
"	Ditto	148,200	17.0	--	347
"	Boiling water	143,100	19.0	--	345
"	Ditto	143,100	21.5	--	346
"	Still air	139,000	21.0	--	343
"	Ditto	140,300	23.0	--	344
1350	Ice water	164,600	11.5	21.9	383
"	Ditto	169,000	12.0	23.4	390
"	Boiling water	155,200	13.0	21.1	360
"	Ditto	155,200	15.5	30.1	354
"	Still air	159,700	12.0	27.3	354
"	Ditto	159,700	13.0	23.4	363
1400	Ice water	184,300	6.5	--	417
"	Ditto	184,800	6.0	--	421
"	Boiling water	171,500	7.0	--	386
"	Ditto	173,000	6.5	--	397
"	Still air	169,400	7.0	--	386
"	Ditto	171,100	9.0	--	389
1450	Boiling water	205,500	1.0	4.6	409
"	Ditto	202,400	2.0	4.8	405
"	Still air	179,200	7.0	16.2	376
"	Ditto	177,200	8.0	17.8	383

TABLE 8. (Continued)

Solution Temp, F[1]	Cooling Medium	Ultimate Tensile Strength, psi[2]	Elong, % in 1 inch	Reduction of Area, %	VHN
1500	Ice water	142,700	0.0[3]	--	464
"	Ditto	124,700	0.0[3]	--	473
"	Boiling water	122,700	0.0	--	413
"	Ditto	126,300	0.0[3]	--	442
"	Still air	170,400	6.5	--	373
"	Ditto	165,500	10.0	--	363

(1) Heating was done in air; the time at temperature was one hour.
(2) Standard 0.250-inch-diameter round specimen.
(3) Specimen broke in shoulders.

TABLE 9.   EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-1Cr-1Fe-3Mn-1Mo-1V ALLOY ROLLED AT 1450 F TO 1/2-INCH-ROUND BAR STOCK

Heat Wt136A

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of Area, %	VHN
1300	Ice water	150,200	25.0	47.6	370
"	Ditto	147,700	25.0	55.3	336
"	Boiling water	149,200	26.5	48.2	354
"	Ditto	147,700	24.0	52.1	342
"	Still air	150,200	26.5	52.7	354
"	Ditto	146,600	24.0	49.8	351
1350	Ice water	185,200	9.0	36.9	401
"	Ditto	171,300	11.0	33.6	375
"	Boiling water	163,300	18.0	46.4	349
"	Ditto	162,600	22.5	41.8	359
"	Still air	180,900	12.0	28.9	388
"	Ditto	177,400	14.0	29.5	372
1400	Ice water	189,400	13.0	29.5	401
"	Ditto	192,000	8.5	21.9	413
"	Boiling water	188,400	7.5	15.4	421
"	Ditto	188,400	10.5	21.1	413
"	Still air	197,000	10.5	19.1	413
"	Ditto	196,500	12.0	21.1	421
1450	Ice water	197,100	6.0	17.7	421
"	Ditto	195,500	4.0	11.7	407
"	Boiling water	193,500	1.0	1.6	427
"	Ditto	196,900	2.0	2.4	414

TABLE 9. (Continued)

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of Area, %	VHN
1450	Still air	203,700	4.0	11.8	418
"	Ditto	185,300	13.0	28.9	376
1500	Ice water	(3)	(3)	(3)	448
"	Ditto	(3)	(3)	(3)	483
"	Boiling water	(3)	(3)	(3)	464
"	Ditto	(3)	(3)	(3)	493
"	Still air	196,000	7.0	4.8	413
"	Ditto	192,500	2.0	4.8	417

(1) Heating was done in air; the time at temperature was one hour.
(2) Standard 0.250-inch-diameter round specimens.
(3) Broke in threads.

TABLE 10. EFFECTS OF VARIOUS HEAT TREATMENTS ON PROPERTIES OF Ti-4Fe-1Cr-1Mn-1Mo-1V ALLOY ROLLED AT 1600 F TO 1/2-INCH-ROUND BAR STOCK

Heat Wt86A

Solution Temp, F(1)	Cooling Medium	Ultimate Tensile Strength, psi(2)	Elong, % in 1 inch	Reduction of area, %	VHN
1300	Ice water	158,900	19.0	--	367
"	Ditto	160,900	16.5	--	366
"	Boiling water	156,100	20.0	--	363
"	Ditto	153,800	19.0	--	354
"	Still air	155,800	22.0	--	370
"	Ditto	152,200	23.5	--	363
1350	Still air	174,700	10.0	15.4	397
"	Ditto	170,300	10.0	16.2	397
1400	Ice water	176,700	9.5	--	401
"	Ditto	177,700	11.0	--	405
"	Boiling water	170,600	11.0	--	397
"	Ditto	171,600	12.0	--	390
"	Still air	202,600	3.0	--	421
"	Ditto	204,200	3.0	--	428
1450	Ice water	191,900	1.0	1.0	417
"	Ditto	195,500	7.0	14.0	421
"	Boiling water	202,400	1.0	1.0	405
"	Ditto	213,800	1.0	1.0	421
1500	Ice water	188,900	1.0	--	425
"	Ditto	106,800	0.0(3)	--	418

TABLE 10. (Continued)

Solution Temp, F [1]	Cooling Medium	Ultimate Tensile Strength, psi [2]	Elong, % in 1 inch	Reduction of Area, %	VHN
1500	Boiling water	168,400	0.0	--	424
"	Ditto	138,500	0.0	--	420
"	S till air	127,800	0.0	--	532
"	Ditto	121,700	0.0	--	526

(1) Heating was done in air; the time at temperature was one hour.

(2) Standard 0.250-inch-diameter round specimen.

(3) Specimen broke outside gage marks - uniform elongation recorded.

a function of solution temperature in Figures 2 through 8, inclusive. The data points in the figures represent the average of results obtained on the two specimens given each heat treatment.

With the exception of the 1300 F treatment, the properties developed in all four alloys appeared to be a function of the alloy content, solution temperature, and cooling rate from the solution temperature. At 1300 F, the cooling rate from the solution temperature had very little effect on the resultant properties.

The Ti-3.5Cr-3.5V alloy (Tables 4 and 5 and Figures 2 and 3) showed the least promise as a heat-treatable, high-strength alloy. Although it developed reasonably good ductility at a strength level of 150,000 to 160,000 psi after the 1300 F treatment, its ductility at higher strength levels was relatively low. The two rolling temperatures used on the different heats of this alloy showed no consistent effects on the properties as-heat-treated. An unusual feature of this alloy was that in air-cooled specimens the tensile strength, hardness, and ductility varied only over a small range after all heat treatment from 1300 to 1500 F. The water-quenched specimens were excessively brittle after heat treatments at 1450 F and higher.

The Ti-5Mn-2.5Cr alloy (Tables 6 and 7 and Figures 4 and 5) had excellent ductility at a strength level of 150,000 to 160,000 psi after the 1300 F treatment. The tensile strengths of quenched specimens solution treated at 1350 to 1500 F fell within a relatively narrow range for both heats of this alloy. However, at temperatures higher than 1400 F, the ductilities dropped off very sharply. The air-cooled specimens attained maximum tensile strengths at 1350 and 1400 F, respectively, for the specimens rolled at 1600 and 1450 F. At higher solution temperatures, they were excessively brittle. Specimens of the heat rolled at 1450 F developed excellent tensile properties after heating at 1400 F and quenching in ice water. A tensile strength of 175,000 psi, with elongation of 17 per cent in 1 inch, was produced by this treatment. The alloy appeared to be very sensitive to cooling rates after heating at 1400 F, however, since specimens quenched in boiling water from this temperature had considerably lower ductility at about the same strength level.

Of all the alloys tested, the Ti-3Mn complex alloy (Tables 8 and 9 and Figures 6 and 7) attained the best high-strength properties after solution treating. Both heats of this alloy had excellent ductility at a strength level of 140,000 to 160,000 psi after the 1300 F treatments. After solution treating at 1400 F, the material rolled at 1600 F developed a strength of 170,000 to 185,000 psi, with ductilities of 6 to 8 per cent. The material rolled at 1450 F had strengths of 188,000 to 197,000 psi and elongations of 9 to 11-1/2 per cent after the 1450 F treatment. Cooling rates from 1450 F affected the results very little. At 1500 F, the strength of the material rolled at 1450 F increased slightly, but the ductility decreased quite sharply.
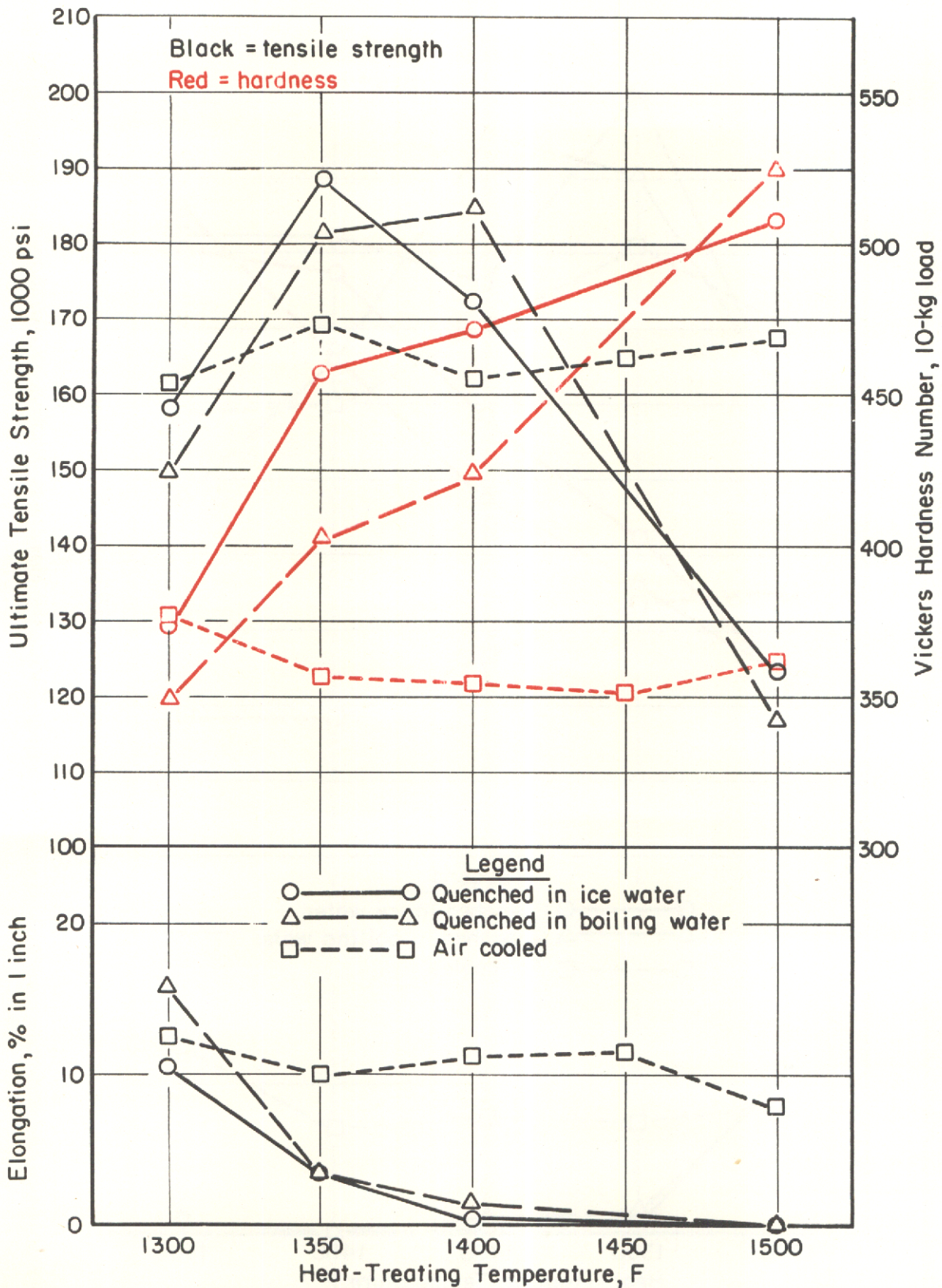
FIGURE 2. TENSILE PROPERTIES AND HARDNESSES OF $\frac{1}{2}$-INCH-ROUND BAR STOCK OF A Ti-3.5Cr-3.5V ALLOY HEAT TREATED AS SHOWN Heat WT42A; rolling temperature-1600 F

A-4057
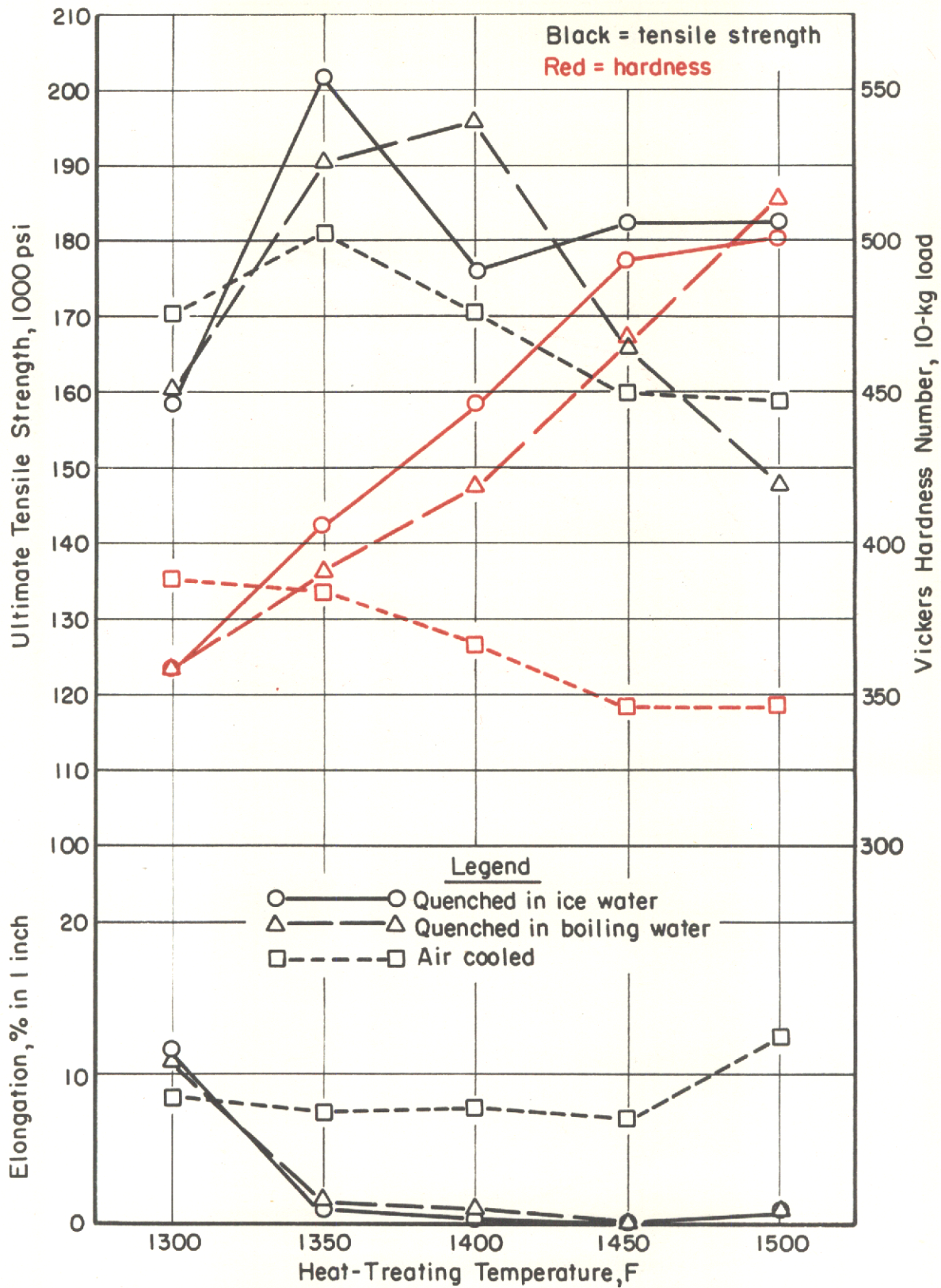
FIGURE 3. TENSILE PROPERTIES AND HARDNESS OF ½-INCH-ROUND BAR
STOCK OF A Ti-3.5Cr-3.5V ALLOY HEAT TREATED AS SHOWN
Heat WT125A ; rolling temperature -1450 F

A-4058
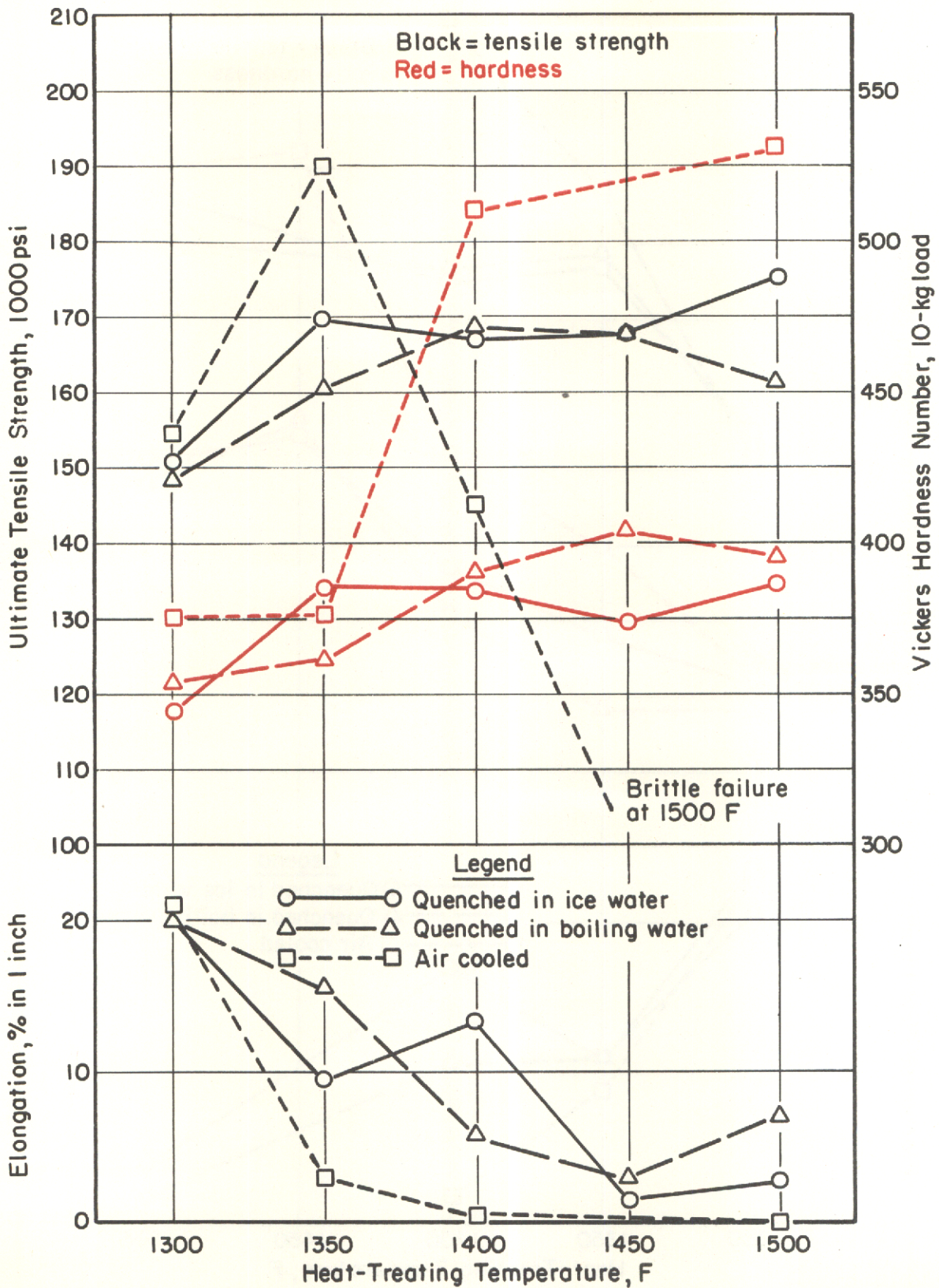
FIGURE 4. TENSILE PROPERTIES AND HARDNESSES OF $\frac{1}{2}$-INCH-ROUND BAR STOCK OF A Ti-5Mn-2.5Cr ALLOY HEAT TREATED AS SHOWN Heat WT51A ; rolling temperature -1600 F

A-4059
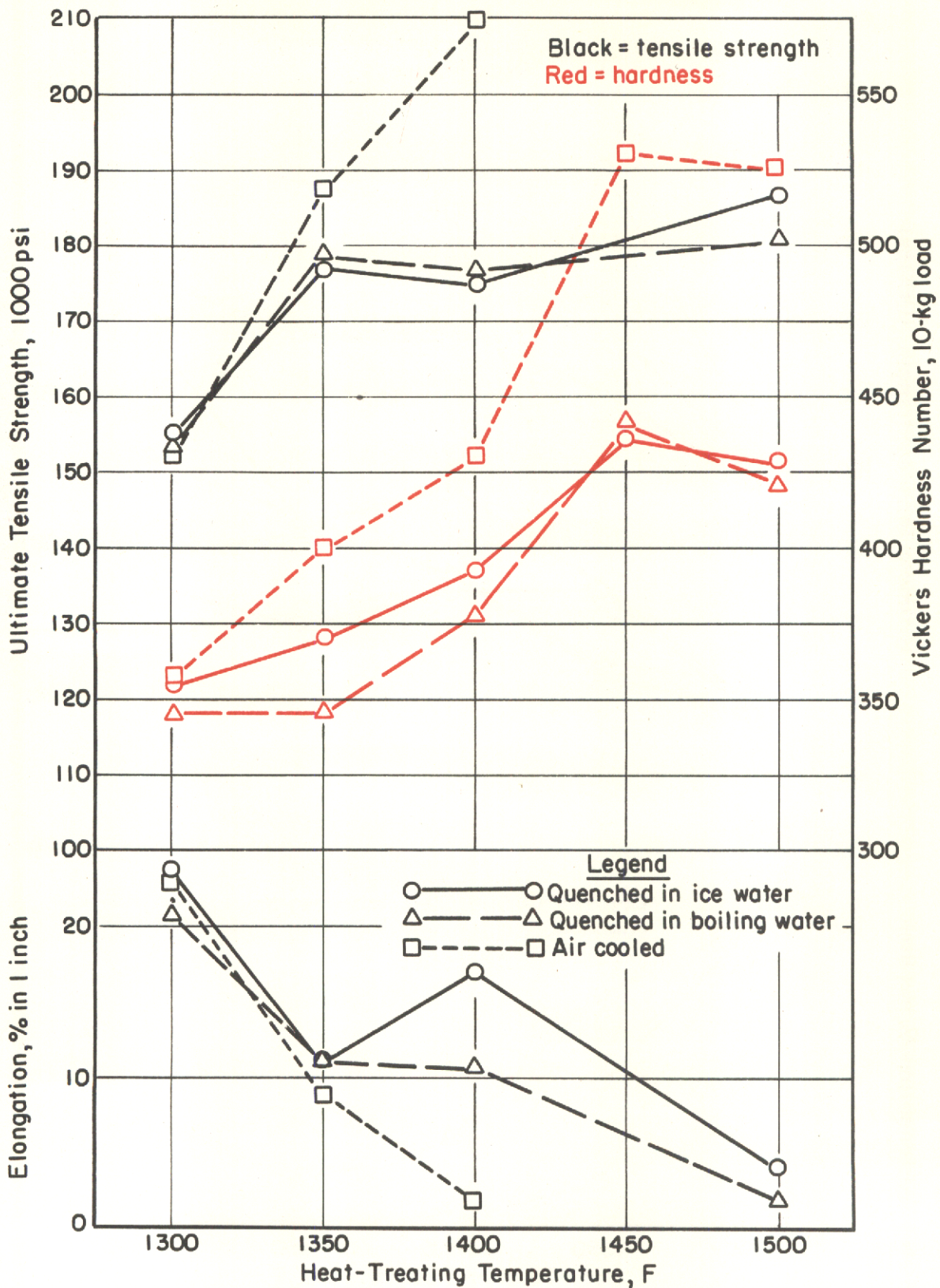
FIGURE 5. TENSILE PROPERTIES AND HARDNESSES OF $\frac{1}{2}$-INCH-ROUND BAR STOCK OF A Ti-5Mn-2.5Cr ALLOY HEAT TREATED AS SHOWN Heat WT132A; rolling temperature-1450 F

A-4060
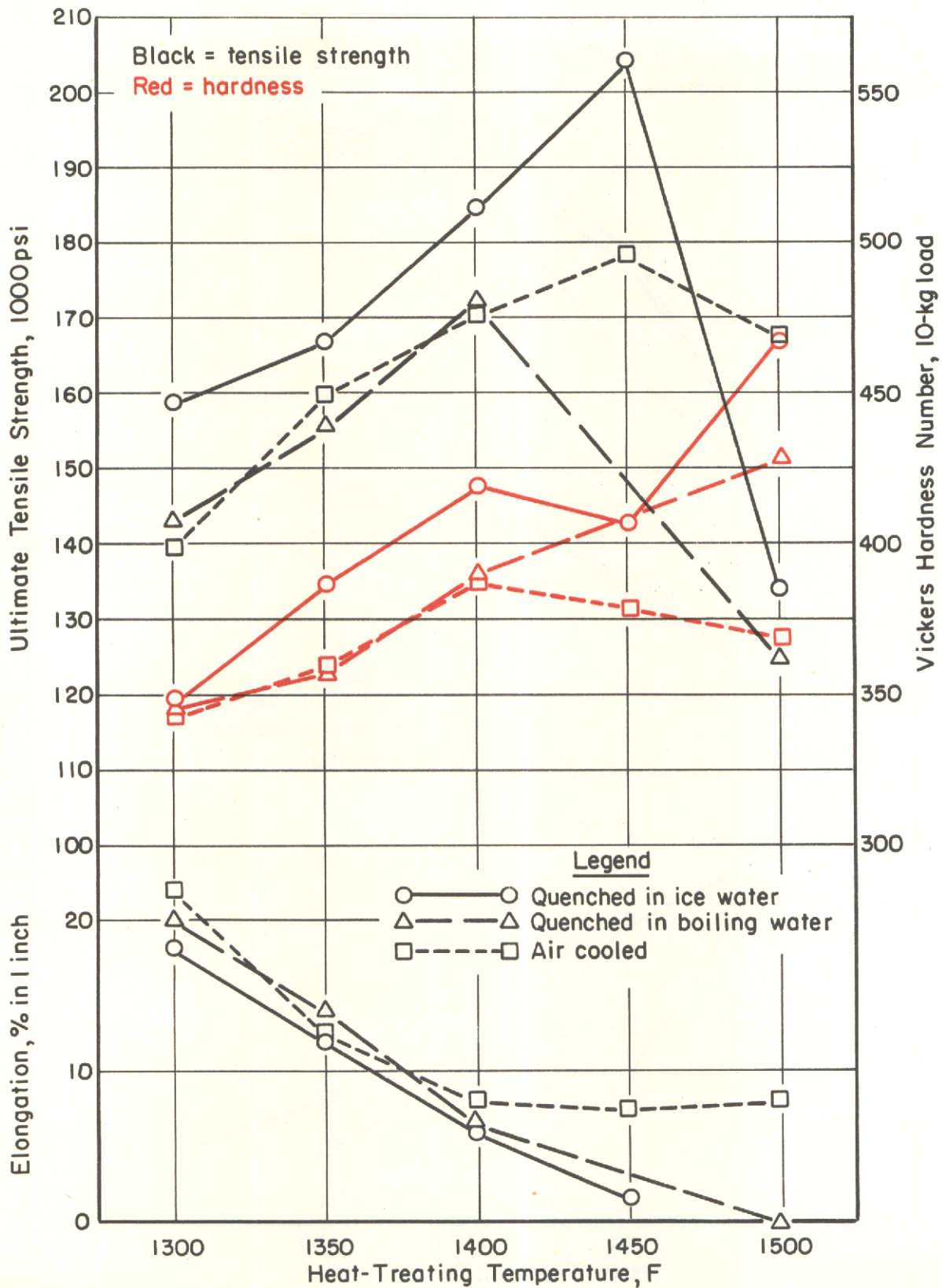
FIGURE 6. TENSILE PROPERTIES AND HARDNESSES OF $\frac{1}{2}$-INCH-ROUND BAR STOCK OF A Ti-1Cr-1Fe-3Mn-1Mo-1V ALLOY HEAT TREATED AS SHOWN

Heat WT107A; rolling temperature 1600 F

A-4061
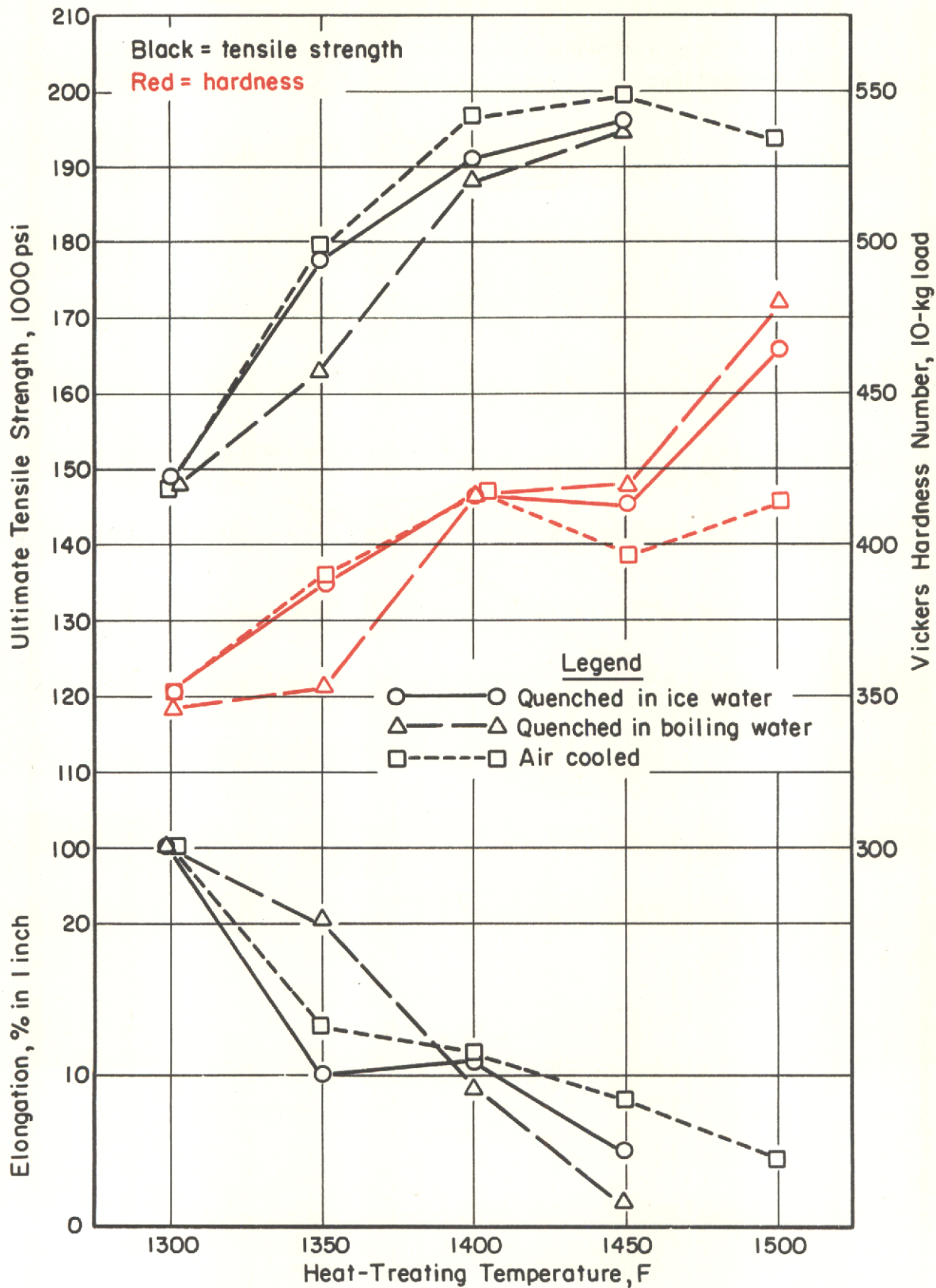
FIGURE 7. TENSILE PROPERTIES AND HARDNESSES OF $\frac{1}{2}$-INCH-ROUND BAR STOCK OF A Ti-ICr-IFe-3Mn-IMo-IV ALLOY HEAT TREATED AS SHOWN

Heat WT136A ; rolling temperature-1450 F

A-4062
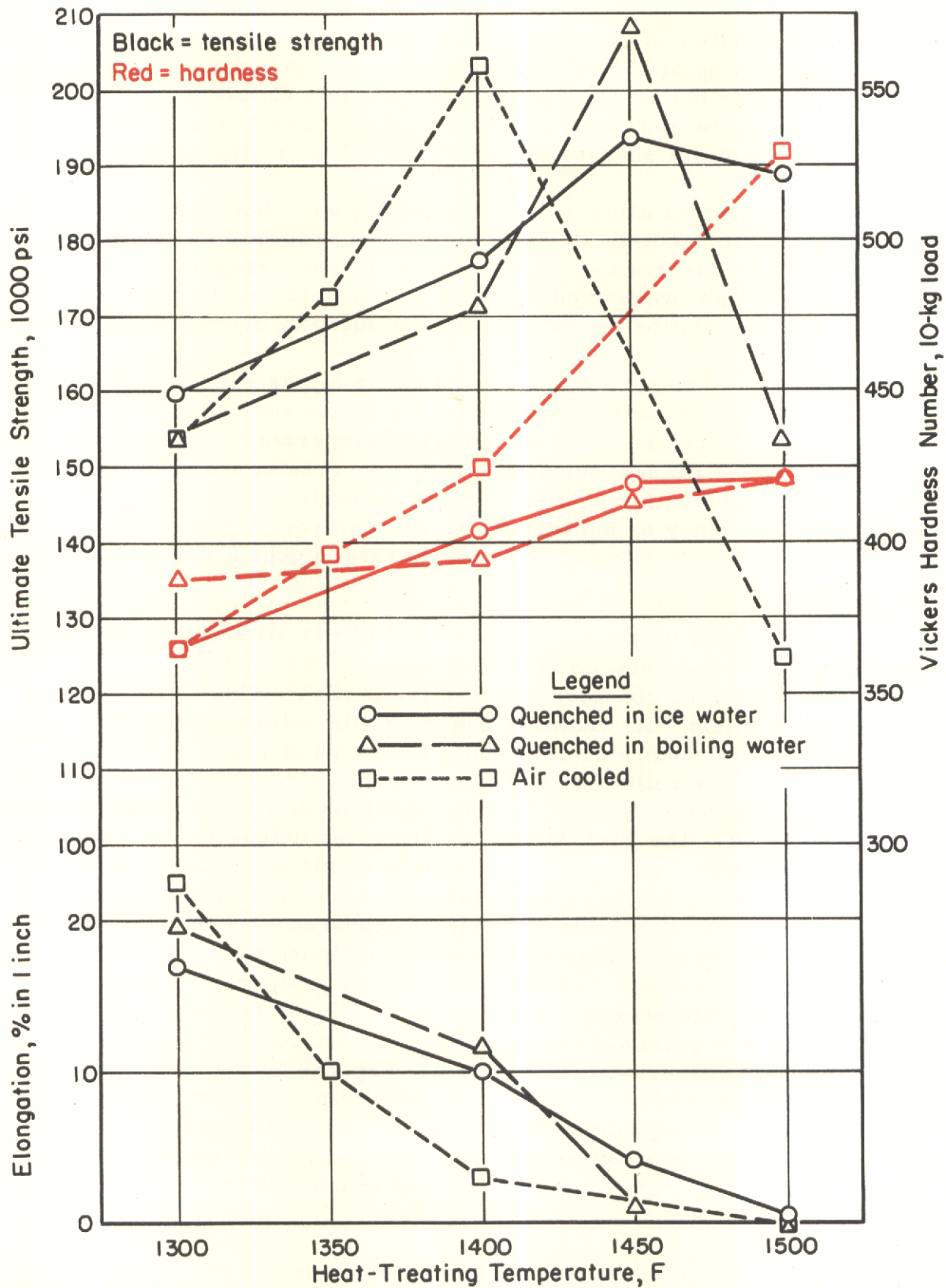
FIGURE 8. TENSILE PROPERTIES AND HARDNESSES OF ½-INCH-ROUND BAR
STOCK OF A Ti-4Fe-1Cr-1Mn-1Mo-1V ALLOY HEAT TREATED
AS SHOWN
Heat WT86A

A-4063

The reproducibility of properties in the Ti-3Mn complex alloy after solution treatments up to 1450 F was very good in both heats. This alloy is the only one thus far tested which indicated a definite difference in the effects of rolling temperature on heat-treated properties. The heat rolled at 1450 F had properties definitely superior to those of the heat rolled at 1600 F.

The Ti-4Fe complex alloy was available only as rolled at 1600 F. Again, good ductility at intermediate strength levels was produced by the 1300 F solution treatment. Reasonably good properties at a strength level of 170,000 to 180,000 psi were produced in this alloy also by quenching from 1400 F. At higher solution temperatures, the ductility decreased sharply.

The increase in strength and hardness of the alloys with increasing solution temperature evidently is associated with the amount of beta phase present and its alloy content. As the solution temperature is increased, the amount of beta phase present increases and, conversely, its alloy content decreases. The lower alloy beta is more susceptible to hardening during cooling by the coherency or age-hardening mechanism. The degree to which the beta phase of a given alloy content was hardened depends upon the cooling rate.

The microstructures of the heat-treated alloys all had the same basic constituents, alpha and beta, in varied amounts. The mode of occurrence of the alpha phase differed considerably in a given alloy rolled at 1450 or 1600 F after heat treatment. As shown in Figure 9, the alloys rolled at 1450 F and heat treated had a more or less spheriodal type of residual alpha. Heat treatment of the heats rolled at 1600 F resulted in a residual precipitate along the grain boundaries and Widmanstätten-type alpha within the grains as shown in Figure 10. The Ti-3.5Cr-3.5V alloy was unique in that, at solution temperatures of 1400 F or higher, a visible precipitate appeared in the retained-beta phase in the air-cooled specimens, as shown in Figure 11. After the 1500 F treatment, this precipitate appeared as a Widmanstätten alpha structure in the beta matrix, as shown in Figure 12. In the remaining three alloys, the beta phase was retained after all heat treatments. An unusual structure was noted in the Ti-3.5Cr-3.5V and Ti-5Mn-2.5Cr alloys rolled at 1450 F and quenched from 1500 F, and air cooled from 1400 F, respectively. This structure, illustrated in Figures 13 and 14, appeared to be a subboundary structure in the beta phase within individual grains. This structure is shown at 2000 diameters' magnification in Figures 15 and 16. It was not found in these particular alloys under any other conditions of heat treatment. It may be significant that the specimens exhibiting the structure were extremely brittle.

In general, the solution treatments as described in this section seemed to have some promise as possible commercial heat treatments. Relatively soft, highly ductile structures can be produced by solution treating at 1300 F. After fabricating or machining, the strength and hardness of the material may be raised by solution treating at an appropriately higher temperature. This type of treatment would be suitable only for materials to be used at about
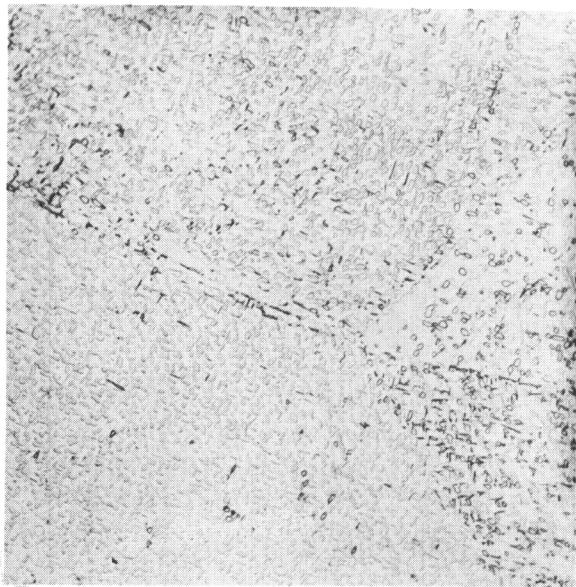
500X   96942

FIGURE 9. HEAT WT125A, A Ti-3.5Cr-3.5V ALLOY
ROLLED AT 1450 F, HEAT TREATED AT 1300 F, AND
WATER QUENCHED

Structure: Primary spheroidal-type alpha in beta matrix

500X   95562

FIGURE 10. HEAT WT42A, A Ti-3.5Cr-3.5V ALLOY
ROLLED AT 1600 F, HEAT TREATED AT 1300 F,
AND WATER QUENCHED

Structure: Primary Widmanstätten alpha in beta matrix
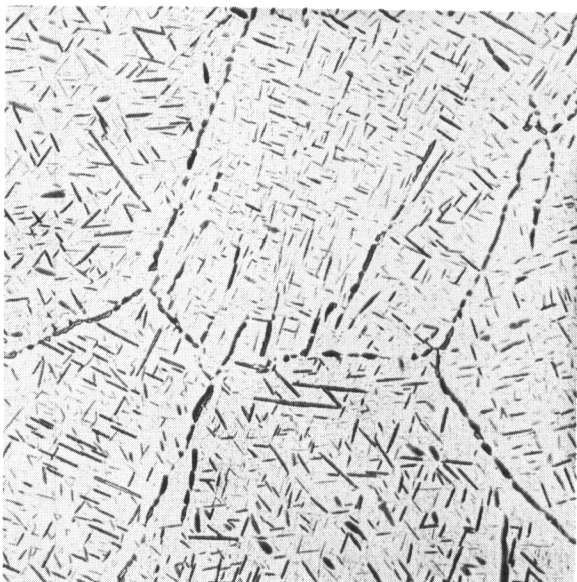
500X   95556

FIGURE 11. HEAT WT42A, A Ti-3.5Cr-3.5V ALLOY
ROLLED AT 1600 F, HEAT TREATED AT 1400 F, AND
AIR COOLED

Structure: Primary alpha plus alpha precipitate
in beta matrix
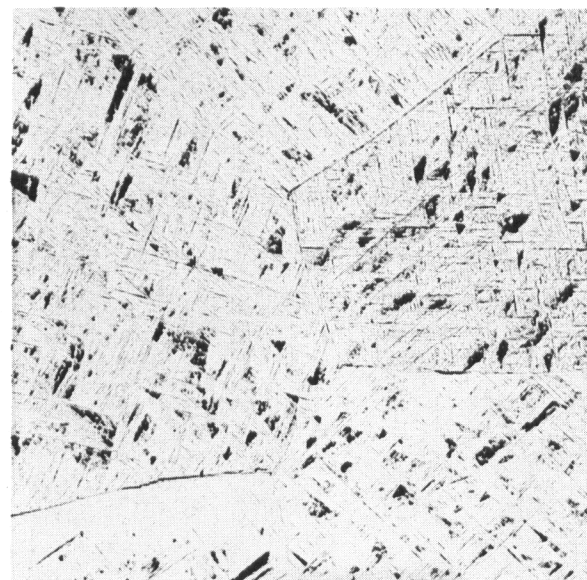
500X   95553

FIGURE 12. HEAT WT42A, A Ti-3.5 Cr-3.5V ALLOY
ROLLED AT 1600 F, HEAT TREATED AT 1500 F, AND
AIR COOLED

Structure: Widmanstätten alpha precipitate in beta matrix
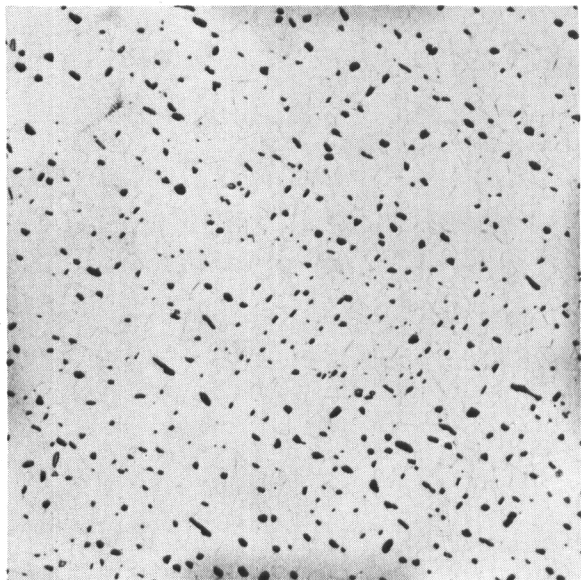
500X   96946

FIGURE 13. HEAT WT125A, A Ti-3.5Cr-3 5V
ALLOY ROLLED AT 1450 F, HEAT TREATED AT
1500 F, AND WATER QUENCHED

Structure: Primary alpha (black) in beta matrix.
Note subboundary structure; alpha was overetched
to reveal this structure.
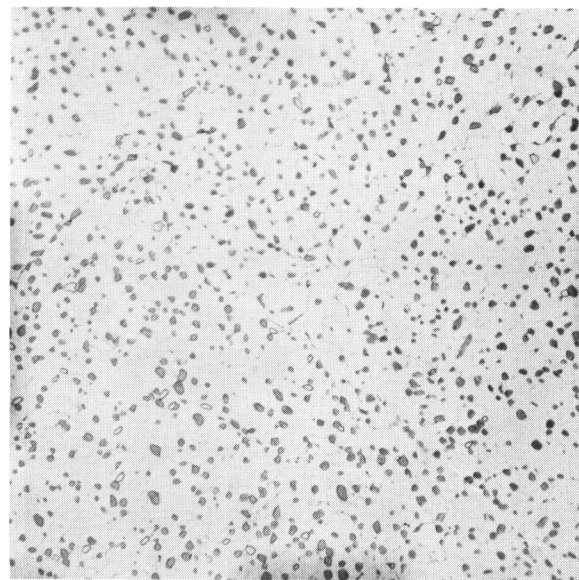
500X   96939

FIGURE 14. HEAT WT132A, A Ti-5Mn-2.5Cr ALLOY
ROLLED AT 1450 F, HEAT TREATED AT 1400 F,
AND AIR COOLED

Structure: Primary alpha in beta matrix. Note sub-
boundary structure in beta.
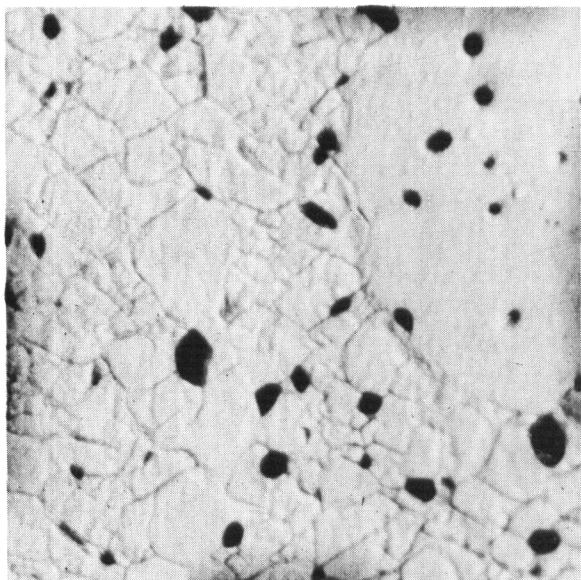
2000X   96934

FIGURE 15. HEAT WT125A, A Ti-3.5Cr-3.5V
ALLOY ROLLED AT 1450 F, HEAT TREATED AT
1500 F, AND WATER QUENCHED, SHOWING SUB-
BOUNDARY STRUCTURE AT HIGHER MAGNIFI-
CATION
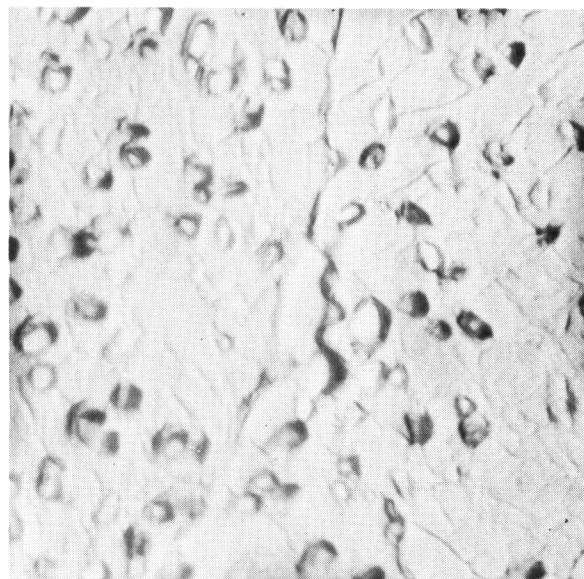
2000X   96924

FIGURE 16. HEAT WT132A, A Ti-5Mn-2.5Cr ALLOY
ROLLED AT 1450 F, HEAT TREATED AT 1400 F, AND
AIR COOLED, SHOWING SUBBOUNDARY STRUCTURE
AT HIGHER MAGNIFICATION

room temperature, however, since alloys so treated probably would age harden at temperatures of 300 F or higher.

Age Hardening

Sheet. Initial work on the age hardening of selected alloys consisted of establishing curves of hardness versus time for specimens solution treated at 1300, 1400, and 1600 F and aged at temperatures of 200 to 900 F. Complete curves for the alloys described in this section were given in WADC-TR-52-249, Figures 28 to 43, inclusive. Since only a limited amount of stock was available, the investigation of the mechanical properties of aged specimens had to be limited to relatively few conditions. Therefore, the solution and aging treatments to be applied to tensile specimens were selected on the basis of hardness. The hardness level chosen was 400 to 425 VHN, since the best high-strength properties in the as-hot-rolled condition occurred in this range. Tensile blanks cut from sheet sections representing both the top and bottom halves of each ingot were given the various solution and aging treatments necessary to produce the desired hardness. All heat treatments at temperatures above 500 F were carried out in an argon atmosphere to prevent any possibility of contamination. Solution treatments were followed by quenching in ice water; after aging, the specimens were air cooled. Complete results of tensile and hardness tests made on the solution-treated and aged specimens are given in Tables 11 through 14, inclusive. For comparison, the properties as-hot-rolled of each heat are included.

In general, the properties developed by the solution-treating and aging treatments were very poor. In some cases, strengths superior to those developed in the as-hot-rolled condition were produced but ductility was very low. An exception to the general rule were the specimens taken from the bottom half of the Ti-3.5Cr-3.5V ingot, solution treated at 1300 F, and aged for one-half hour at 700 F. Strengths of the order of 200,000 psi with 3 to 5 per cent elongation in 1 inch resulted from this treatment.

Specimens of the Ti-3.5Cr-3.5V alloy overaged at 800 or 900 had some ductility, but their ductilities were still low relative to their tensile strengths. The remaining alloys overaged at these temperatures were extremely brittle.

The properties developed by aging, although poor in all cases, seemed to be somewhat better after solution treatment at 1300 F than after the higher temperature solution treatments. Thus, it was indicated that even lower solution temperatures might result in better aged properties. Experiments along this line were conducted on bar stock and the results will be described in the next section. With the exception of the Ti-3.5Cr-3.5V alloy, all of the unaged alloys developed excellent intermediate-strength properties after the 1300 F solution treatment.

TABLE 11. PROPERTIES OF 0.064-INCH SHEET OF A Ti-3.5Cr-3.5V[1] ALLOY IN THE HOT-ROLLED AND SOLUTION-TREATED AND AGED CONDITIONS

As Hot Rolled, 1450 F

Heat No.	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
WT4A	T4	174,700	10.5	394
	T4	174,700	8.0	-
	B2	179,700	9.0	376
	B2	182,300	9.0	-

Solution Treated and Aged[4]

Solution Temp, F	Aging Temp, F	Aging Time, hr	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
1300	-	0	T2	152,900	9.0	333
	-	0	B1	147,400	12.5	330
	600	4	T2	174,700	1.0	397
	"	"	B1	153,500	1.0	401
	700	0.5	T2	176,000	2.0	421
	"	"	T5	173,400	1.0	436
	"	"	T5	202,500	0.0*	445
	"	"	B1	197,800	5.0*	429
	"	"	B1	200,600	3.0	390
	"	"	B1	200,900	3.0	417
1400	-	0	T2	144,200	6.0	374
	-	0	B1	144,900	3.5	429
	300	0.5	T2	186,300	4.0	417
	"	"	B1	179,200	4.5	413
	400	0.5	T2	181,500	2.0	442
	"	"	B1	185,900	2.0	417
	900	5	T2	153,200	8.0	342
	"	"	B1	153,500	4.0	346
1600	-	0	T2	103,500	1.5	357
	-	0	B1	129,700	2.0	327

(1) Actual analysis: Top of ingot - 3.65% Cr, 5.06% V; bottom of ingot - 3.65% Cr, 3.19% V.
(2) T = top half of ingot, B = bottom half of ingot. Number indicates location of 5-inch section of sheet relative to top of ingot section.
(3) Substandard specimen 0.375 x 0.064 x 5 inches with a reduced section 0.250 x 0.064 x 1-1/4 inches.
(4) All solution treating and aging at temperatures above 500 F was done in a dried-argon atmosphere.
* Specimen broke outside gage marks. Uniform elongation reported.

TABLE 12. PROPERTIES OF 0.064-INCH SHEET OF A Ti-5Mn-2.5Cr[1] ALLOY IN THE AS-HOT-ROLLED AND SOLUTION-TREATED AND AGED CONDITIONS

As Hot Rolled, 1450 F

Heat No.	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
WT15A	T4	191,100	5.0	429
	T4	197,500	6.0	-
	B4	178,600	8.0	405
	B4	167,800	12.0	-

Solution Treated and Aged[4]

Solution Temp, F	Aging Temp, F	Aging Time, hr	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
1300	-	0	T2	142,000	15.0	314
	-	0	B3	135,000	19.0	319
	700	0.5	T2	187,500	0.5*	436
	"	"	T3	182,400	0.5	433
	"	"	B2	188,700	3.0	410
	"	"	B2	186,200	3.0	413
	"	0.75	T2	193,400	1.0	416
	"	"	B3	151,000	0.5	397
1400	-	0	T2	155,100	8.5	371
	-	0	B3	147,700	11.0	356
	500	2	T2	175,200	0.0*	397
	"	"	B3	178,600	2.0	387
	800	16	T2	102,600	0.0	405
	"	"	B3	194,800	2.0	405
1600	-	0	T2	132,900	6.5	399
	-	0	B3	(5)	(5)	383
	300	0.5	T2	116,100	0.0*	413
	"	"	B3	111,300	0.0	431
	900	16	T2	(6)	(6)	417
	"	"	B3	(6)	(6)	417

(1) Actual analysis: Top of ingot - 4.48% Mn, 2.00% Cr; bottom of ingot - 3.12% Mn, 1.37% Cr.
(2) T = top half of ingot, B = bottom half of ingot. Number indicates location of 5-inch section of sheet relative to top of ingot section.
(3) Substandard specimen 0.375 x 0.064 x 5 inches with a reduced section 0.250 x 0.064 x 1-1/4 inches.
(4) All solution treating and aging at temperatures above 500 F was done in a dried-argon atmosphere.
(5) Defective specimen.
(6) Specimen too brittle to test.
* Specimen broke outside gage marks. Uniform elongation recorded.

TABLE 13. PROPERTIES OF 0.064-INCH SHEET OF A Ti-1Cr-1Fe-3Mn-1Mo-1V[1] ALLOY IN THE AS-HOT-ROLLED AND SOLUTION-TREATED AND AGED CONDITIONS

Heat No.	As Hot Rolled, 1450 F				Solution Treated and Aged[4]						
	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN	Solution Temp, F	Aging Temp, F	Aging Time, hr	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
WT25A	T3	192,500	5.0	417	1300	-	-	T3	138,400	17.0	317
	T3	193,400	6.0	-		-	-	B3	136,100	20.0	314
	B4	191,600	6.0	405		700	0.5	T5	189,900	1.5	429
	B4	186,300	7.5	-		"	"	T5	193,300	1.0	425
						"	"	B5	185,100	3.0	417
						"	"	B5	177,600	3.5	405
						"	1.0	T3	185,800	1.0	401
						"	1.0	B3	177,200	1.5	388
					1400	-	-	T3	161,900	4.5	363
						-	-	B3	161,900	7.0	373
						500	16	T3	193,800	1.0	413
						"	"	B3	189,900	0.0	413
						600	0.5	T3	177,400	0.0*	433
						"	"	B3	184,400	0.0*	417
						800	16	T3	181,300	1.0	416
						"	"	B3	176,300	0.0*	429
					1600	-	-	T3	98,700	0.0	423
						-	-	B3	106,900	0.0*	421
						200	8	T3	128,900	1.0	413
						"	"	B3	90,800	0.0*	454
						300	0.5	T3	120,300	1.0	437
						"	"	B3	116,800	0.0	437
						900	2	T3	104,400	0.5	459
						"	"	B3	187,500	0.0*	425

(1) Actual analysis: Top of ingot - 0.90% Cr, 1.00% Fe, 3.74% Mn, 1.05% Mo, 1.10% V; bottom of ingot - 1.20% Cr, 0.92% Fe, 3.48% Mn, 1.05% Mo, 0.98% V.

(2) T = top half of ingot; B = bottom half of ingot. Number indicates location of 5-inch section of sheet relative to top-of-ingot section.

(3) Substandard specimen 0.375 x 0.064 x 5 inches with a reduced section 0.250 x 0.064 x 1-1/4 inches.

(4) All solution treating and aging at temperatures above 500 F was done in a dried-argon atmosphere.

* Specimen broke outside gage marks. Uniform elongation recorded.

TABLE 14. PROPERTIES OF 0.064-INCH SHEET OF A Ti-1Cr-4Fe-1Mn-1Mo-1V[1] ALLOY
IN THE AS-HOT-ROLLED AND SOLUTION-TREATED AND AGED CONDITIONS

Heat No.	As Hot Rolled, 1450 F				Solution Treated and Aged[4]						
	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN	Solution Temp, F	Aging Temp, F	Aging Time, hr	Specimen Location[2]	Ultimate Tensile[3] Strength, psi	Elongation, % in 1 inch	VHN
WT32A	T4	223,800	2.0	442	1300	-	0	T5	152,900	5.0	339
	T4	220,900	2.5	-		-	0	B2	146,800	15.0	327
	B5	182,100	5.0	366		600	8	T5	159,000	1.0	409
	B5	176,000	8.0	-		"	"	B2	174,000	0.0*	394
						700	0.5	T3	131,600	0.0	449
						"	"	B1	95,000	0.0*	421
						"	"	B1	160,000	1.5	425
						800	4	T5	172,500	0.0*	405
						"	"	B2	165,200	1.0	446
					1400	-	0	T5	174,000	1.0	360
						-	0	B2	174,400	2.0	376
						500	4	T5	159,000	1.0	425
						"	"	B2	156,800	1.0	409
						600	0.5	T5	122,800	0.0*	455
						"	"	B2	141,200	1.5	429
						900	1	T5	104,000	0.0*	421
						"	"	B2	(5)	(5)	425
					1600	-	0	T5	178,200	2.5	390
						-	0	B2	122,800	1.0	429
						200	24	T5	173,500	0.0*	421
						"	"	B2	152,700	0.0	437
						400	1	T5	92,500	0.0*	417
						"	"	B2	62,500	0.5	450
						900	24	T5	(6)	(6)	437
						"	"	B2	(6)	(6)	437

(1) Actual analysis: Top of ingot - 0.76%Cr, 3.24%Fe, 1.65%Mn, 1.05%Mo, 0.98%V; bottom of ingot - 0.90%Cr, 3.15%Fe, 1.69%Mn, 1.12%Mo, 0.93%V.
(2) T = top half of ingot; B = bottom half of ingot. Number indicates location of 5-inch section of sheet relative to top of ingot section.
(3) Substandard specimen 0.375 x 0.064 x 5 inches with a reduced section 0.250 x 0.064 x 1-1/4 inches.
(4) All solution treating and aging at temperatures above 500 F was done in a dried-argon atmosphere.
(5) Specimen broke in grips.
(6) Specimens too brittle to test.

1/2-Inch-Round Bar Stock. Initially, the solution and aging treatments used on bar stock were the same as those used for the sheet specimens previously described. In addition, solution treatments at 1200 F followed by aging at 700 F were attempted. Primarily to promote thermal stability at elevated temperatures, a third series of specimens from the alloys rolled at 1450 F were subjected to a long-time overaging treatment at 800 to 900 F after solution treating at 1300 or 1400 F. The specific treatments used and the complete results of tensile and hardness tests made on the heat-treated specimens are given in Tables 15 through 17.

It may be seen that treatments consisting of solution treating at 1300 or 1400 F followed by relatively short-time aging treatments resulted in very poor properties, with one exception. The Ti-3Mn complex alloy solution treated at 1300 F and aged for 1 hour at 700 F (Table 17) had reasonably good ductility at a strength level of about 182,000 psi. In general, however, the ductilities of the specimens so treated were very low.

The specimens of the three alloys solution treated at 1200 F and aged at 700 F developed very good properties in many cases. The good results obtained by these treatments, however, were overshadowed by the excellent properties developed by the long-time overaging treatments. Combinations of properties much superior to those obtained under any previous condition of heat treatment or fabrication were obtained in all of the alloys by heat treatments consisting of solution treating at 1300 or 1400 F, and overaging at 800 F or 900 F for times ranging from 8 to 52 hours. Times of 24 and 52 hours were used at 800 F, and 8 and 24 hours at 900 F. The properties obtained by these treatments are shown in bar-chart form in Figures I, II, and III in the Summary to this report. Tensile strengths in the range of 160,000 to 240,000 psi with adequate ductility at all strength levels were produced by these treatments. The Ti-3Mn complex alloy developed partic- ularly good properties. For example, after solution treating at 1400 F and aging for 24 hours at 800 F, this alloy developed a tensile strength of about 225,000 psi with an average elongation of 6 per cent in 1 inch. Aging for 52 hours at 800 F lowered the tensile strength to about 215,000 psi, but in- creased the elongation to about 10 per cent in 1 inch. The ductility, as measured by reduction of area, was also very good at all strength levels in this alloy. It is highly possible that, in this particular condition of heat treatment, these alloys would be stable for extended periods of time at temperatures up to 600 F.

A good correlation existed between tensile strength and hardness in all three alloys after the long-time overaging treatments. This is shown graphically in Figure 17. To our knowledge, this is the first time such a correlation has been shown. A reasonably good correlation between ductility, expressed as tensile elongation, and hardness also existed for the Ti-5Mn- 2.5Cr and the Ti-3Mn complex alloys; this is shown in Figure 17 also. The ductilities for the Ti-3.5 Cr-3.5V alloy generally fell below the band created by the other two alloys. The low ductilities of this particular heat may be due in part to its very high tungsten content (0.8 per cent tungsten).

TABLE 15. PROPERTIES OF THE Ti-3.5Cr-3.5V ALLOY SOLUTION TREATED AND AGED AS 1/2-INCH-ROUND BAR STOCK

Heat No.	Actual(3) Composition, %	Solution(4) Temp, F	Aging Treatment	Ultimate Tensile(6) Strength, psi	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT42A(1)	4.62Cr, 3.34V	1300	1/2 hour at 700 F	188,800	0.5	0.0	438
		"	Ditto	(7)	(7)	(7)	478
		1400	1/2 hour at 300 F	145,600	0.5	0.0	488
		"	Ditto	(7)	(7)	(7)	478
		"	3 hours at 900 F	184,375	2.0	1.5	397
		"	Ditto	159,300	0.5	0.0	394
WT125A(2)	4.50Cr, 3.22V	1200	1 hour at 700 F	191,400	2.0	2.4	-
		"	Ditto	179,200	8.0	15.4	390
		"	4 hours at 700 F	185,300	4.0	8.7	390
		"	Ditto	192,300	6.0	7.9	401
		1200(5)	1 hour at 700 F	190,400	2.0	1.6	417
		" (5)	Ditto	182,300	6.0	9.5	394
		" (5)	4 hours at 700 F	176,000	1.0	1.6	401
		" (5)	Ditto	188,600	1.5	1.6	405
		1300	24 hours at 800 F	171,500	10.0	19.1	389
		"	Ditto	173,900	10.0	11.8	385
		"	52 hours at 800 F	168,600	10.0	21.1	366
		"	Ditto	168,600	17.0	33.6	365
		"	8 hours at 900 F	155,200	10.0	14.0	372
		"	Ditto	155,200	13.0	21.9	386
		"	24 hours at 900 F	149,900	15.5	21.9	329
		"	Ditto	150,300	16.5	23.4	340
		1400	24 hours at 800 F	193,100	7.0	10.9	401
		"	Ditto	190,600	11.5	30.1	382
		"	52 hours at 800 F	191,400	7.0	6.5	385
		"	Ditto	189,000	10.0	26.8	395
		"	8 hours at 900 F	167,000	11.0	14.6	386

TABLE 15. (Continued)

Heat No.	Actual(3) Composition, %	Solution(4) Temp, F	Aging Treatment	Ultimate Tensile(6) Strength, psi	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT125A(2)	4.50Cr, 3.22V	1400	8 hours at 900 F	168,200	14.0	30.1	360
		"	24 hours at 900 F	160,100	13.0	13.2	358
		"	Ditto	162,100	17.0	39.3	355

(1) Rolled at 1600 F.

(2) Rolled at 1450 F.

(3) Average composition.

(4) Time at temperature was 1 hour unless otherwise specified. Specimens were quenched in ice water.

(5) Time at temperature was 4 hours.

(6) Standard 0.250-inch-round tensile specimens.

(7) Specimen broke in shoulder radius.

TABLE 16. PROPERTIES OF THE Ti-5Mn-2.5Cr ALLOY SOLUTION TREATED AND AGED AS 1/2-INCH-ROUND BAR STOCK

Heat No.	Actual(3) Composition, %	Solution(4) Temp, F	Aging Treatment	Ultimate Tensile(6) Strength, psi	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT51A(1)	5.20Mn, 2.63Cr	1300	1/2 hour at 700 F	193,700	1.0	0.0	413
		"	Ditto	188,392	1.5	0.8	417
		1400	16 hours at 800 F	224,000	(7)	(7)	464
WT132A(2)	5.60Mn, 2.8Cr	1200	1 hour at 700 F	178,000	12.0	35.4	387
		"	Ditto	167,000	17.0	52.7	373
		"	4 hours at 700 F	198,600	10.5	21.9	429
		"	Ditto	176,200	17.0	41.1	380
		1200(5)	1 hour at 700 F	177,800	15.5	33.0	383
		"(5)	Ditto	196,300	4.0	4.0	413
		"(5)	4 hours at 700 F	198,200	10.0	18.4	421
		"(5)	Ditto	202,000	2.0	3.2	421
		1300	24 hours at 800 F	213,800	2.0	3.2	442
		"	Ditto	212,600	5.0	8.7	430
		"	52 hours at 800 F	206,100	9.5	16.9	413
		"	Ditto	202,900	10.0	19.7	425
		"	8 hours at 900 F	183,300	14.5	37.4	389
		"	Ditto	184,900	17.0	42.3	380
		"	24 hours at 900 F	177,600	17.0	39.9	381
		"	Ditto	177,600	19.0	38.6	377
		1400	24 hours at 800 F	239,900	4.0	5.7	456
		"	Ditto	238,700	1.5	3.2	461
		"	52 hours at 800 F	242,000	3.0	4.0	453
		"	Ditto	239,100	2.5	4.0	450
		"	8 hours at 900 F	201,200	9.0	24.2	416
		"	Ditto	199,200	10.0	26.0	402
		"	24 hours at 900 F	189,400	16.0	28.9	389
		"	Ditto	187,000	14.0	41.7	379

Footnotes appear on the following page.

Footnotes for Table 16:

(1) Rolled at 1600 F.

(2) Rolled at 1450 F.

(3) Average composition.

(4) Time at temperature was 1 hour unless otherwise specified.   Specimens were quenched in ice water.

(5) Time at temperature was 4 hours.

(6) Standard 0.250-inch-diameter round tensile specimens.

(7) Specimen broke in shoulder radius.

TABLE 17. PROPERTIES OF THE Ti-1Cr-1Fe-3Mn-1Mo-1V ALLOY SOLUTION TREATED AND AGED AS 1/2-INCH-ROUND BAR STOCK

Heat No.	Actual(3) Composition, %	Solution(4) Temp, F	Aging Treatment	Ultimate Tensile(6) Strength, psi	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT107A(1)	1.39Cr, 2.95Mn, 0.99Fe, 0.92Mo, 0.98V	1300	1 hour at 700 F	180,900	6.5	11.3	417
		"	Ditto	184,600	4.0	1.5	417
		1400	16 hours at 500 F	191,100	(7)	(7)	450
		"	Ditto	191,900	(7)	(7)	442
		"	16 hours at 800 F	201,327	(7)	(7)	450
		"	Ditto	217,100	2.5	2.4	468
WT136A(2)	1.04Cr, 1.00Fe, 3.1Mn, 0.90Mo, 0.93V	1200	1 hour at 700 F	183,300	14.0	32.1	383
		"	Ditto	181,700	11.0	32.1	390
		"	4 hours at 700 F	183,300	10.0	28.3	-
		"	Ditto	180,900	10.0	23.4	-
		1200(5)	1 hour at 700 F	173,100	14.0	41.7	-
		" (5)	Ditto	172,700	14.0	36.0	-
		" (5)	4 hours at 700 F	183,700	8.0	10.1	-
		" (5)	Ditto	184,800	11.0	18.4	-
		1300	24 hours at 800 F	193,400	12.0	28.9	394
		"	Ditto	189,000	16.0	35.4	394
		"	52 hours at 800 F	186,600	17.0	42.3	395
		"	Ditto	187,400	17.0	39.3	382
		"	8 hours at 900 F	171,100	19.0	44.8	375
		"	Ditto	172,900	20.0	49.8	363
		"	24 hours at 900 F	163,700	20.0	53.3	356
		"	Ditto	163,300	22.5	46.0	352
		1400	24 hours at 800 F	225,300	5.5	9.5	429
		"	Ditto	224,600	7.0	17.7	419
		"	52 hours at 800 F	214,700	12.0	25.4	418

TABLE 17. (Continued)

Heat No.	Actual(3) Composition, %	Solution(4) Temp, F	Aging Treatment	Ultimate Tensile(6) Strength, psi	Elongation, % in 1 inch	Reduction of Area, %	VHN
WT136A(2)	1.04Cr, 1.00Fe, 3.1Mn, 0.90Mo, 0.93V	1400	52 hours at 800 F	214,900	8.0	22.6	421
		"	8 hours at 900 F	191,400	13.0	42.3	390
		"	Ditto	189,300	15.5	45.5	389
		"	24 hours at 900 F	181,100	17.0	55.6	369
		"	Ditto	178,600	19.0	58.7	372

(1) Rolled at 1600 F.

(2) Rolled at 1450 F.

(3) Average composition.

(4) Time at temperature was 1 hour unless specified otherwise. Specimens were quenched in ice water.

(5) Time at temperature was 4 hours.

(6) Standard 0.250-inch-diameter round tensile specimens.

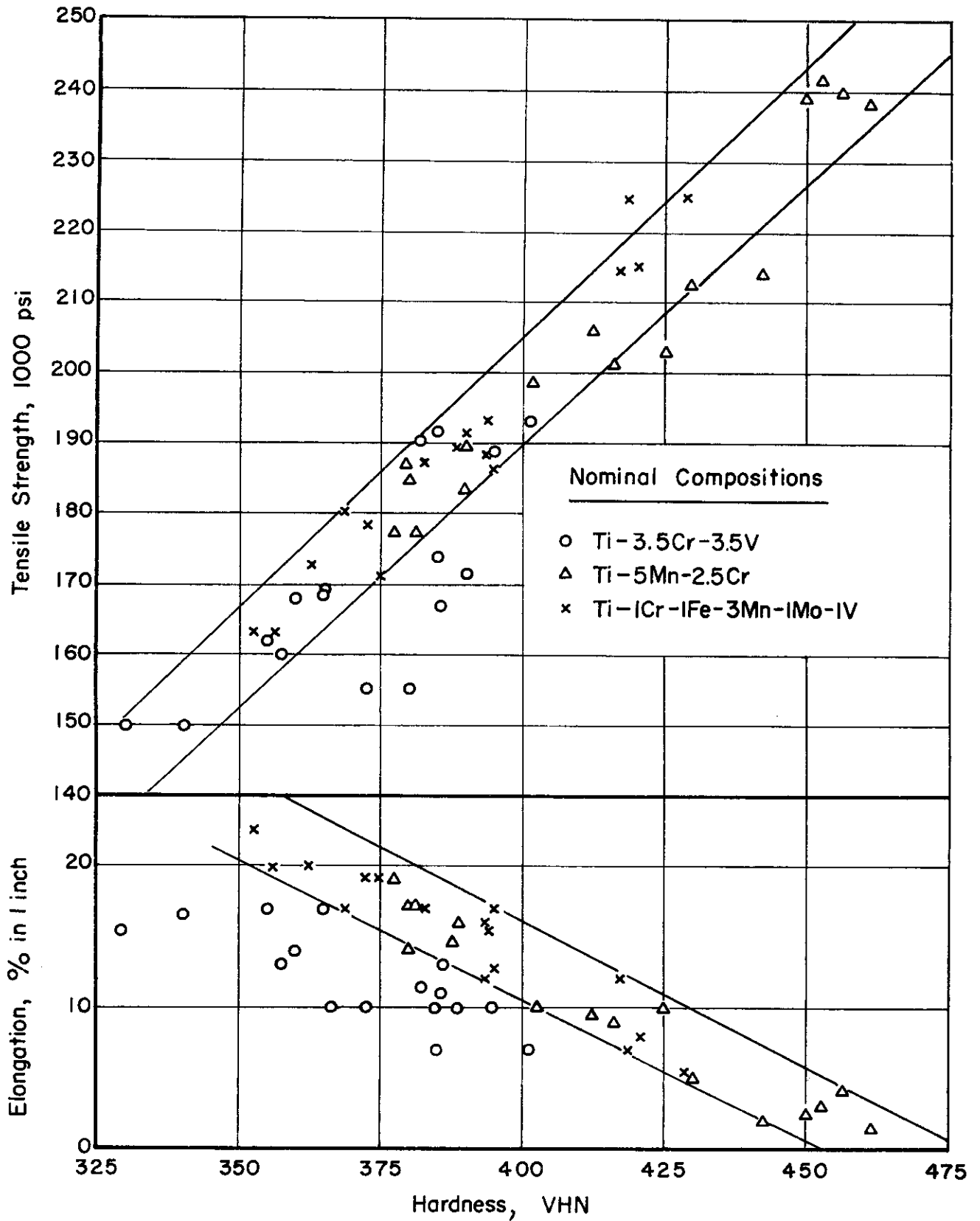(7) Specimen broke in shoulder radius.

FIGURE 17. HARDNESS VERSUS TENSILE STRENGTH AND ELONGATION FOR THREE SELECTED ALLOYS SOLUTION TREATED AT 1300 AND 1400 F AND AGED AT 800 AND 900 F

A-4064

The superior properties resulting from the long-time overaging treatments may be connected with the disappearance of an intermediate phase tentatively designated as ω (omega) which appears during the age hardening of beta-stabilized alloys. More detailed information on this phase is contained in the following sections of this report. Briefly, it was present in titanium-chromium, titanium-iron, and titanium-molybdenum alloys age hardened at temperatures up to 700 F, and in a Ti-8Mn alloy isothermally transformed at 842 F (450 C). In the latter instance, it was present for transformation times up to one hour but disappeared in favor of alpha at some time between one and four hours. Since this phase was observed in all of the aforementioned alloy systems, it is reasonable to assume that it would be present also in age-hardened complex alloys containing two or more of these elements. It has been shown in this and preceding reports that such complex alloys have very low ductility when aged at temperatures up to 700 F and for short times at 800 or 900 F*. Since the metastable omega phase was shown to exist at temperatures up to 700 F and for a period of hours at a temperature of 842 F, it appears that the low ductility previously associated with age hardening may have been due to its presence. The good ductility attained by overaging for long times at 800 to 900 F, then, may be the result of replacement of the omega phase by alpha.

The results described for the long-time overaging treatments were obtained just recently, and an intensive investigation of this type of heat treatment is now under way. This investigation will be continued under the new contract on alloy development. It will involve establishing the temperature and time limits for this type of heat treatment, determining the effect of section size on the resulting properties, and determining the effects of rolling temperature. Thermal-stability tests will be conducted also at temperatures up to 600 F.

Tube-Drawing Tests

In connection with the program for evaluation of the selected alloys by outside companies, specimens of 1/2-inch-round bar stock from five heats of the selected alloys were annealed and sent to Superior Tube Company for tube-drawing tests. They are interested in the use of high-strength titanium alloys for hydraulic tubing in aircraft.

The annealing treatment used for all specimens consisted of heating for one-half hour at 1350 F, furnace cooling in 2 hours to 1150 F, and water quenching. This heat treatment produces the softest structure yet attained in the alloys under investigation. The specimens, each approximately 8 inches long, were machined at Superior Tube Company to remove outside scale, and a 0.187-inch hole was drilled through the center of each.

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*This also is true of the commercial alloy Ti 150 A. Omega phase may also be responsible for the embrittlement of such alloys when they are quenched and aged.

The first drawing tests were conducted at the Norristown plant on October 23, 1952. Prior to drawing, the specimens had been dipped in a special lubricant developed by Superior Tube Company. In addition, a No. 205 chlorinated-oil lubricant was used. Drawing was done on a 10,000-pound draw bench using an 0.186-inch mandrel. Dies providing a reduction in area of 12 to 17 per cent were used for the first drawing operation. No trouble was experienced during the drawing. Only one specimen, that of Heat WT125A, revealed any cracks after drawing; this was the Ti-3.5Cr-3.5V alloy rolled at 1450 F. It was high in tungsten, and some fine cracks had been noted prior to the drawing operation.

Drawing tests have been continued on these specimens at Superior Tube Company. An annealing treatment similiar to the aforementioned one has been used between each drawing operation. The present status of the experimental tubing is shown in Table 18. Cracks have developed on the inside diameters of all the alloys. The depth of these cracks has not been determined. If they are simply surface cracks, drawing will be continued.

The results obtained in these tests thus far are encouraging. As may be seen in Table 18, considerable reduction in wall thickness was attained in most of the alloys before any cracking occurred. According to Mr. H. W. Cooper, of Superior Tube Company, these are the first potentially high-strength alloys which have been cold drawn to any great degree successfully. It is probable that the heat treatment applied to these alloys prior to drawing was not the best for this type of operation. It is expected that further developmental work on heat treatments will result in much better cold drawability.

<u>Forgeability Tests</u>

Two 20-pound ingots of the Ti-1Cr-1Fe-3Mn-1Mo-1V alloy have been sent to the Wyman-Gordon Company, Worcester, Massachusetts, for closed-die forging tests. A small aircraft part will be made in order to evaluate the forgeability of the alloy, and the properties of the forging.

<u>ISOTHERMAL-TRANSFORMATION STUDIES ON BINARY
TITANIUM-MANGANESE ALLOYS</u>

As a continuation of the program for more fundamental studies on titanium alloys, the isothermal-transformation characteristics of three binary titanium-manganese alloys were determined. Previously, time-temperature-transformation relationships were established for three titanium-chromium alloys, as described in WADC-TR-52-249, pages 114

TABLE 18. DATA FOR TUBE-DRAWING TESTS ON
SELECTED ALLOYS

Heat No.	Nominal Composition, %	Original Wall Thickness, inch	Present Wall Thickness, inch	Total Reduction in Area, %
WT42A	3.5Cr, 3.5V	0.125	0.080	35
WT125A	3.5Cr, 3.5V	0.137	0.105	30
WT132A	5Mn, 2.5Cr	0.117	0.072	29
WT136A	1Cr, 1Fe, 3Mn, 1Mo, 1V	0.138	0.115	19
WT86A	1Cr, 4Fe, 1Mn, 1Mo, 1V	0.112	0.085	31

to 151. Information obtained during the earlier work has proved very valuable to the understanding of the basic reactions of titanium alloys after certain heat treatments. It was believed that additional work of this nature on another alloy system would provide further useful information.*

The equilibrium diagram of the titanium-manganese system, using Process A titanium as a base material, shows a eutectoid reaction at about 675 C (1247 F) and a composition of about 20 per cent manganese.[1]** The alloys used in the current investigation were of the hypoeutectiod type, having nominal compositions of 3, 8, and 12 per cent manganese. The expected microstructures for completely transformed specimens of these alloys would be proeutectoid alpha and a eutectoid of alpha and TiMn. However, as will be seen later in this section, the eutectoid transformation was apparently so sluggish that no evidence of this constituent appeared even after relatively long transformation times.

The TTT diagrams for the alloys were determined on the basis of microstructure, X-ray diffraction studies, and hardness.

Materials Used and Experimental Procedure

The alloys were made from Du Pont titanium sponge and high-purity electrolytic manganese. They were arc melted in a water-cooled copper crucible as 3/4-pound ingots, machined to chips, and remelted to promote homogeneity. The ingots were fabricated to 14-gage (0.064-inch) sheet, which was used throughout the investigation. Specimens used for heat treatments were approximately 3/8 by 1/2 by 0.064 inch. A standard beta-izing treatment of 30 minutes at 950 C (1742 F) in a dried-argon atmosphere was applied to all specimens prior to isothermal treatment. The specimens were quenched from the beta-izing furnace into a well-deoxidized molten lead bath held at the desired transformation temperature ±2 C. After holding for a predetermined time, they were quenched into ice water.

After heat treatment, the specimens were cut into two parts, each approximately 1/4 by 3/8 by 0.064 inch. One section was mounted in a room-temperature casting resin for Vickers hardness determinations, and the other section mounted in Bakelite for microscopic examinations. The hardness samples were converted into X-ray diffraction specimens later.

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* It was through the isothermal-transformation study of the titanium-manganese alloys that omega phase was discovered, as will be shown later.

** Superscript numbers in parentheses refer to the references listed at the end of this report.

Metallographic specimens were polished using the standard procedures. Final polishing was done on a high-speed wheel (1750 rpm), using jewelers' rouge as an abrasive. An etching reagent consisting of 1-1/2 per cent HF and 3-1/2 per cent HNO_3 in water was used throughout. Surface-contaminated areas could be detected easily during microscopic examination and avoided in evaluating the microstructures. Vickers hardness determinations were made on polished specimens, using a 10-kg load. Each hardness value reported in this section is the average of at least 3 readings.

X-ray diffraction specimens were prepared by cutting sections approximately 1/16 by 1/16 by 1/2 inch from the hardness specimens, and either chemically etching or electropolishing these sections to slivers about 0.025-inch diameter. The chemical etchant consisted of 15 ml lactic acid, 20 ml HNO_3, and 4 to 10 ml HF. Electrolytic polishing was done in the electrolyte developed at Rem-Cru Titanium Corporation, and described in the Appendix of this report. The two methods of sample preparation were used to check results, particularly when an unknown phase appeared in the patterns of some of the specimens.

In order to obtain diffraction photograms which would be simple to interpret in spite of the coarse-grained nature of the samples, it was decided to use the Debye camera with the sliver samples. X-radiation from a vanadium target was employed without filtration in most of the work. Vanadium radiation was chosen because its characteristic $K\alpha$-wavelength does not excite fluorescence in either the titanium or the manganese in the alloys, and also because it gives maximum dispersion in the X-ray diffraction photograms. A few samples were photographed with filtered copper radiation also.

Eutectoid-Temperature Determination

Since inherent variations in the impurity content of the materials used in this work could affect the eutectoid temperature, its approximate location was checked by the method used in the earlier work on titanium-chromium alloys. Specimens of the 2.91 per cent manganese alloy were heated at 950 C for 30 minutes and quenched in ice water to produce an alpha prime structure. They were then reheated in a molten-lead bath at 675 C (1247 F), 700 C (1292 F), and 725 C (1337 F) for one hour and quenched again. The martensite-type structure of the alpha prime persisted after reheating at 675 C, but, at 700 C, a small amount of beta was observed after the second quench. Still more beta appeared after the treatment at 725 C. These experiments indicated that the eutectoid temperature was very close to the reported value of 675 C.[1]

Titanium-2.91 Per Cent Manganese Alloy

When quenched from the beta field, this alloy undergoes a shear-type transformation to a martensitic structure, as illustrated in Figure 18. The temperature at which this reaction was initiated determined the lower temperature limit for practical isothermal-transformation work. The initiation (M_s) temperature for this reaction was determined, using the modified Greninger-Troiano technique described in WADC-TR-52-249 for the titanium-chromium alloys. Specimens were quenched from the beta field into a lead bath held at various temperatures, allowed to transform for 10 seconds, and then up-quenched into a second lead bath maintained at 650 C (1202 F). They were held for 60 seconds in the second lead bath prior to ice-water quenching. If the first temperature was below the M_s, the martensite-type needles formed could be distinguished readily from the fine Widmanstätten alpha precipitate produced by the transformation at 650 C. The M_s temperature for the 2.91 per cent manganese alloy was placed between 520 C (968 F) and 535 C (995 F). Microstructures of the two specimens initially quenched to these temperatures are shown in Figures 19 and 20.

Isothermal transformations were carried out on this alloy at temperatures of 550 C, 600 C, and 650 C. Proeutectoid alpha separation was initiated at all temperatures in less than 10 seconds. The alpha appeared as a well-defined Widmanstätten structure, which was relatively coarse at all temperatures investigated. The appearance of the structures formed in 10 seconds at 550 and 650 C are shown in Figures 21 and 23. Holding at the transformation temperature for times up to 312 hours simply resulted in some darkening and coalescence of the alpha phase, so that distinct areas of white beta could be observed. No evidence of the titanium-manganese compound or a eutectoid was noted even at the longest transformation time. Figure 22 shows the structure of this alloy transformed for 312 hours at 550 C.

The complete time-temperature-transformation diagram for the 2.91 per cent manganese alloy is presented in Figure 24.

Hardness data for the isothermally transformed specimens are shown graphically in Figure 25. In general, the hardnesses were relatively low at all transformation temperatures. Although the hardness data were rather scattered, there seemed to be a trend for hardnesses to increase slightly with decreasing transformation temperature.

Titanium-7.72 Per Cent Manganese Alloy

This alloy was subjected to an intensive X-ray investigation in connection with the isothermal-transformation work. It was chosen for this investigation because it represents an intermediate alloy in the titanium-manganese system, and also because its composition is very close to that of a commercial alloy (RC130A).

Metallographic Examination

As quenched from the beta-phase region, the 7.72 per cent manganese alloy exhibits a retained-beta structure, as illustrated in Figure 26. The small, black particles shown in this photograph are believed to be an impurity phase, since they appeared indiscriminately in the as-quenched structure and in specimens transformed at all temperatures. In some instances, this phase was present in considerable amounts when X-ray examination revealed no evidence of a second phase. Under these conditions, the alpha phase present in like amounts could be detected easily by X-ray. Isothermal transformation was carried out on this alloy between 650 C (1202 F) and 450 C (842 F).

The initiation of the proeutectoid alpha separation occurred at longer times in this alloy than in the Ti-2.91Mn alloy. Microscopically, alpha was detected first in 30 seconds to 5 minutes at the various transformation temperatures employed. The form of the alpha phase varied considerably with temperature, ranging from a very fine dot-like precipitate at 450 C (Figure 28) to the very coarse Widmanstätten structure formed at 650 C (Figure 34). The initiation of the alpha reactions at temperatures of 450, 550, and 650 C is shown in Figures 27, 30, and 33, respectively. In the temperature range from 450 to 550 C, the initial alpha appears as small dots. The larger particles shown in Figure 30 are believed to be the impurity phase previously mentioned. At 650 C, the initial alpha appears as fine needles along the grain boundaries, as shown in Figure 33.

Longer transformation times at 450 and 500 C result in increased amounts of alpha in the form of a very fine precipitate. This is illustrated in Figures 28 and 29 for the 450 C temperature. After very long holding times at this temperature, a very small amount of another dark-etching phase appears, as may be seen in Figure 29. This may be the titanium-manganese compound or an impurity phase. Identification of the phase could not be made by means of X-ray diffraction.

At 550 C, longer transformation times produced a fine Widmanstätten precipitate of alpha, as shown in Figures 31 and 32. The only effect of increasing time was to increase the particle size of this precipitate. No evidence of a compound was found at this transformation temperature.

Transformation temperatures of 600 and 650 C produced increasingly coarser Widmanstätten alpha, as shown in Figure 34. No evidence of a compound was noted after the longest transformation times (312 hours). The TTT diagram for this alloy, based largely on the metallographic observations, is given in Figure 35.

500X   VHN 374   96169

FIGURE 18. 2.91 PER CENT MANGANESE ALLOY
AS QUENCHED FROM 950 C IN ICE WATER

Structure: Alpha prime
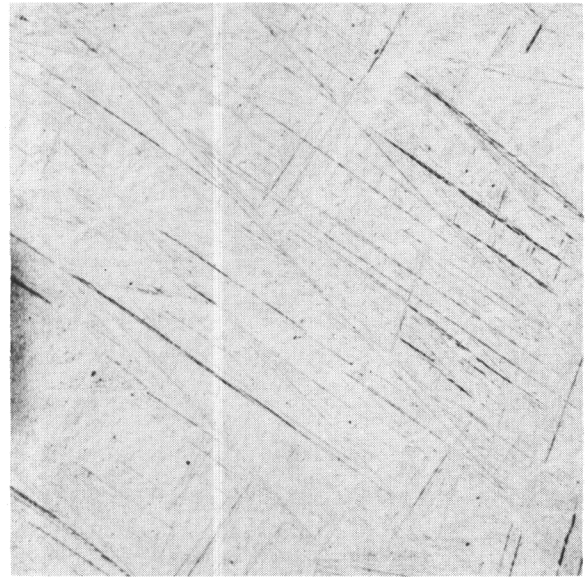
500X   96170

FIGURE 19. 2.91 PER CENT MANGANESE ALLOY;
M_s DETERMINATION AT 520 C

Structure: Alpha prime plates, alpha and beta

500X   96171

FIGURE 20. 2.91 PER CENT MANGANESE ALLOY;
M_s DETERMINATION AT 535 C

Structure: Alpha in beta matrix
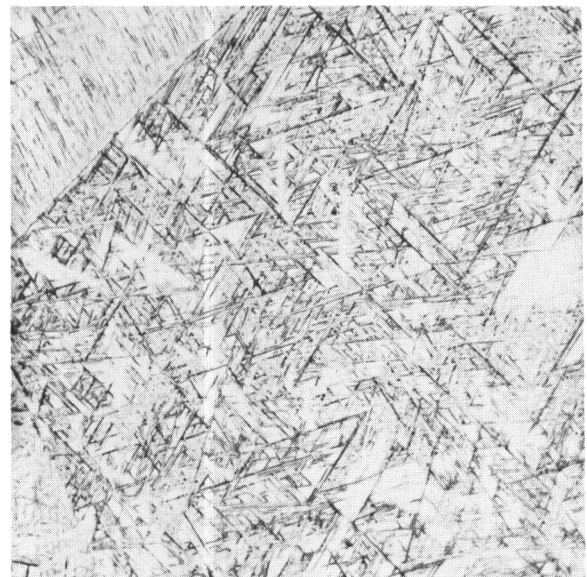
500X   VHN 345   96175

FIGURE 21. 2.91 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED FOR 10 SECONDS
AT 550 C

Structure: Widmanstätten alpha in beta matrix

500X   VHN 275   96176

FIGURE 22. 2.91 PER CENT MANGANESE ALLOY ISO-
THERMALLY TRANSFORMED FOR 312 HOURS AT 550 C

Structure: Widmanstätten alpha in beta matrix

500X   VHN 290   96173

FIGURE 23. 2.91 PER CENT MANGANESE ALLOY ISO-
THERMALLY TRANSFORMED FOR 10 SECONDS AT 650 C

Structure: Widmanstätten alpha in beta matrix
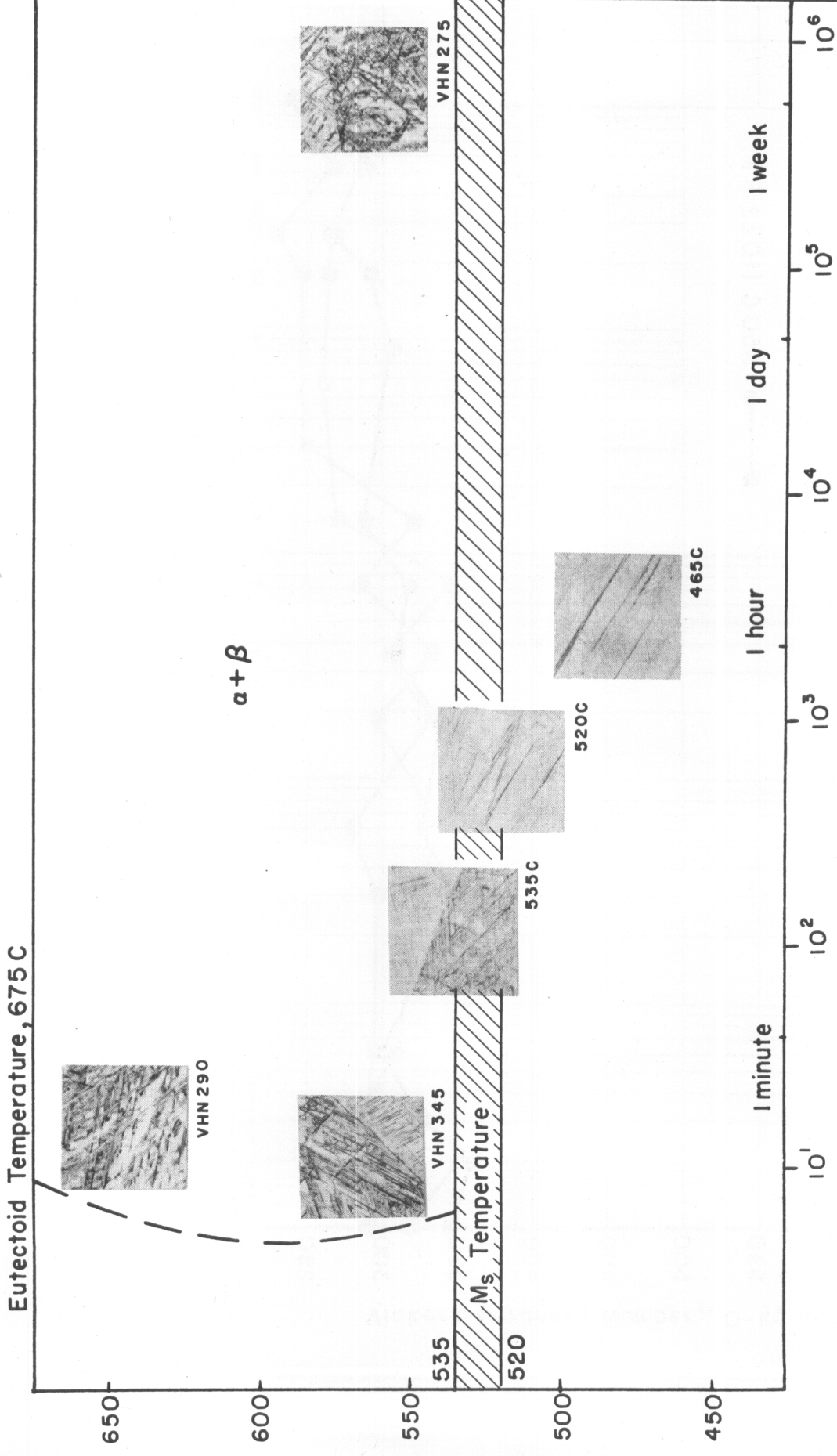
FIGURE 24. ISOTHERMAL-TRANSFORMATION DIAGRAM FOR THE Ti-2.91 Mn ALLOY

A-4065
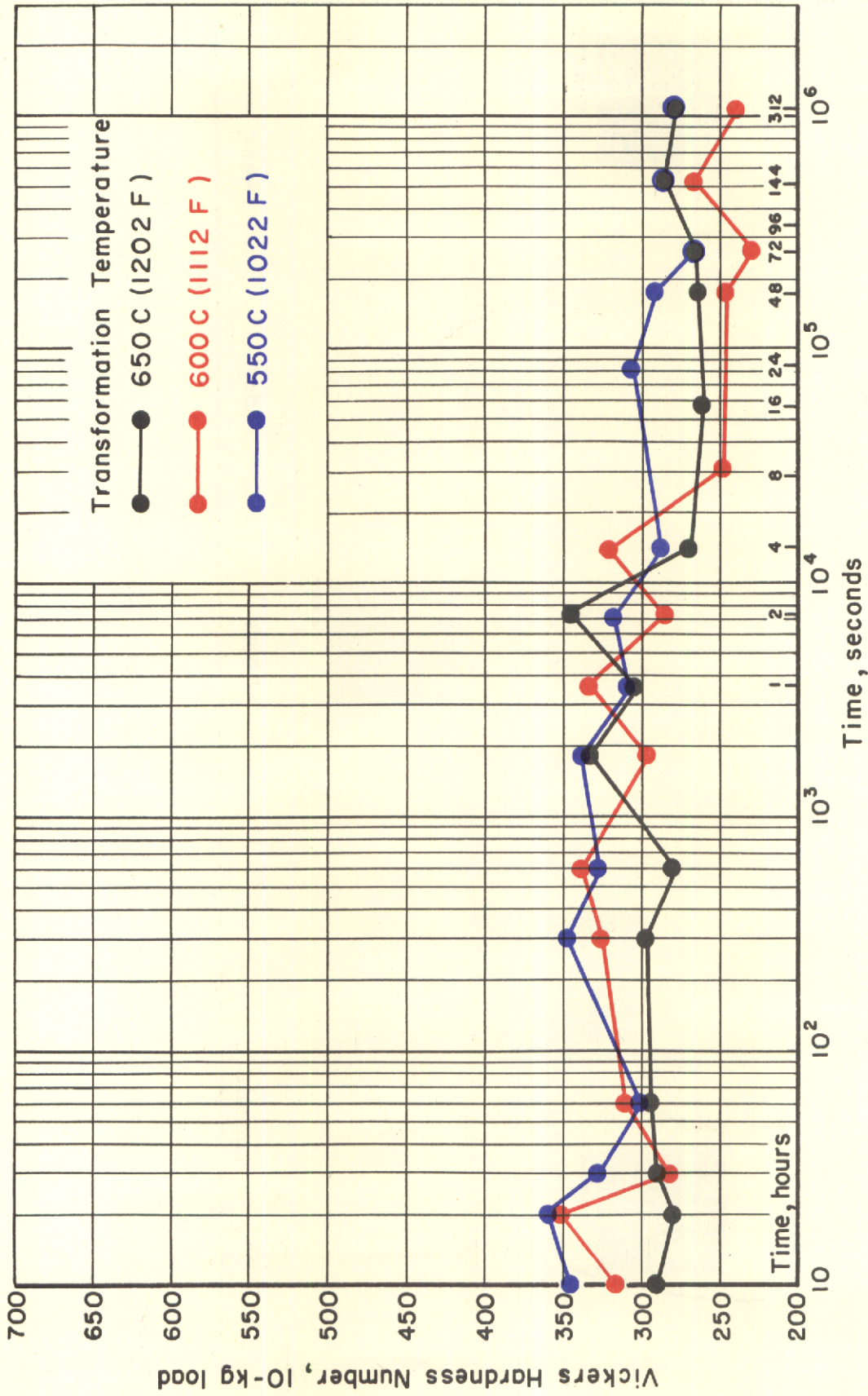
FIGURE 25. HARDNESS VERSUS TIME FOR ISOTHERMALLY TRANSFORMED SPECIMENS OF THE Ti-2.91Mn ALLOY

A-4066
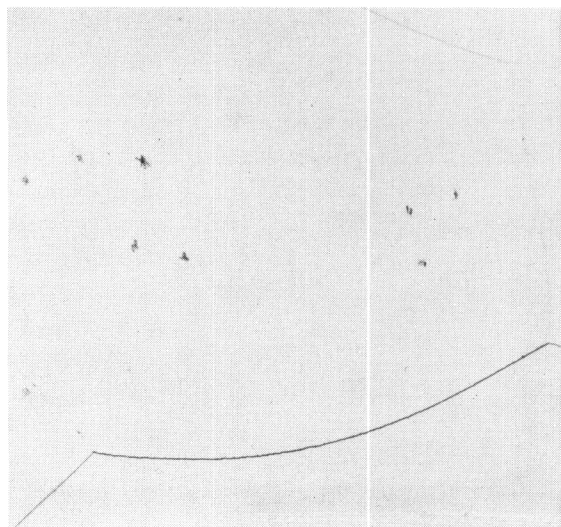
500X   VHN 370   96188

FIGURE 26. 7.72 PER CENT MANGANESE ALLOY
AS QUENCHED IN ICE WATER FROM 950 C

Structure: Retained beta
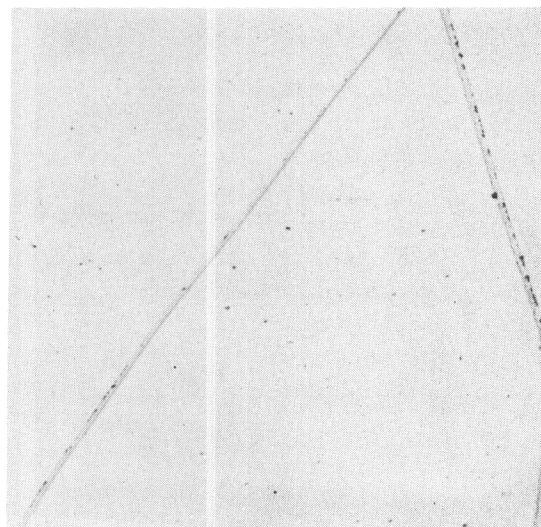
500X   VHN 498   96177

FIGURE 27. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED FOR FIVE MINUTES
AT 450 C

Structure: Alpha in beta matrix; beginning of reaction
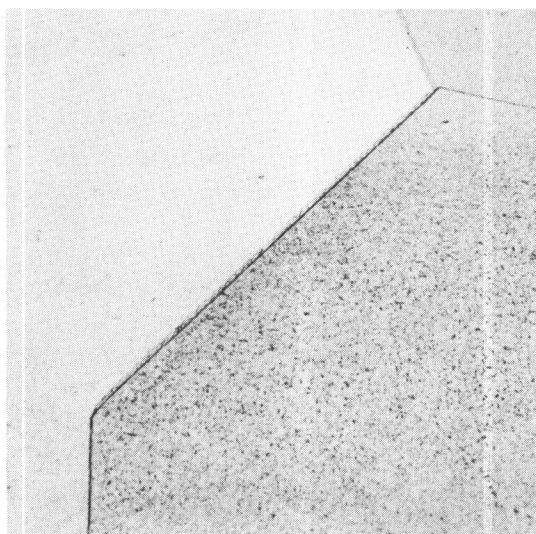
500X   VHN 437   96179

FIGURE 28. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 450 C FOR
168 HOURS

Structure: Alpha in beta matrix
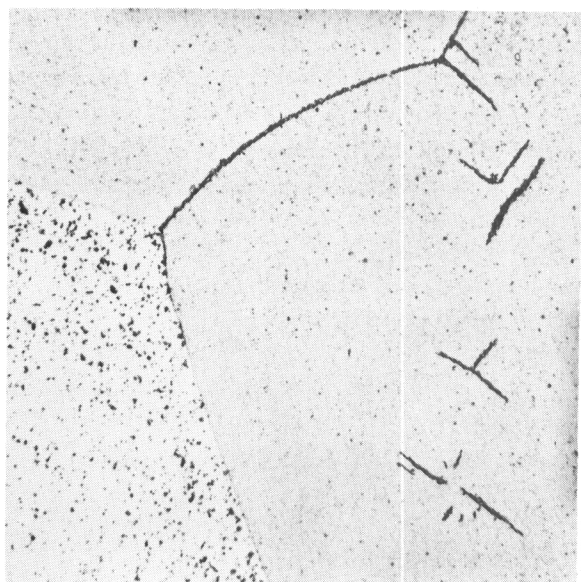
500X   96180

FIGURE 29. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 450 C FOR
408 HOURS

Structure: Alpha in beta matrix
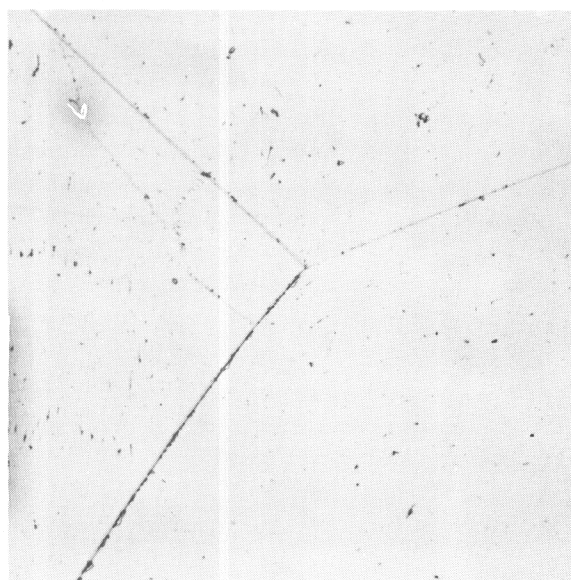
500X   VHN 397   96181

FIGURE 30   7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 550 C for 30
SECONDS

Structure: Alpha in beta matrix; beginning of reaction
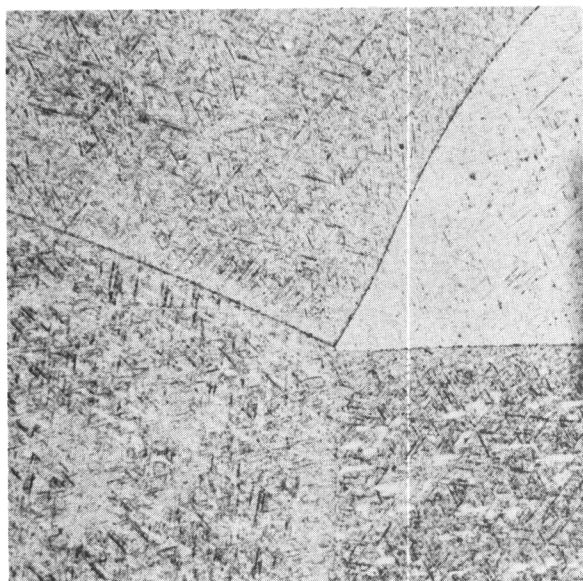
500X   VHN 384   96182

FIGURE 31. 7.72 PER CENT MANGANESE ALLOY ISO-
THERMALLY TRANSFORMED AT 550 C FOR EIGHT
HOURS

Structure: Widmanstätten alpha in beta matrix
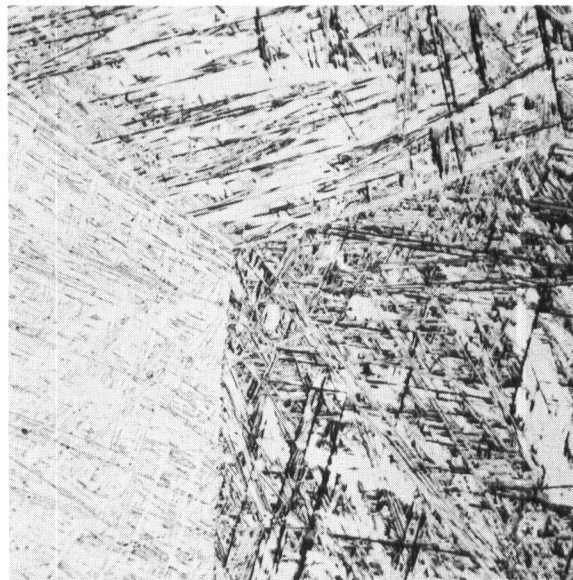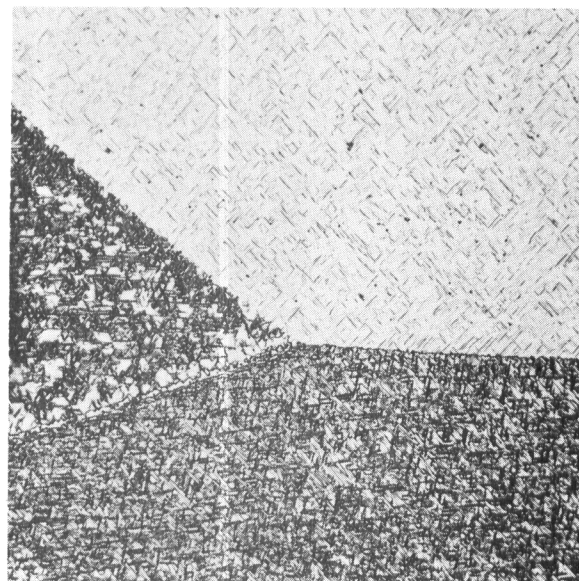

500X   VHN 356   96183

FIGURE 32. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 550 C FOR 312
HOURS

Structure: Widmanstätten alpha in beta matrix

500X   VHN 391   96184

FIGURE 33. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 650 C FOR
60 SECONDS

Structure: Alpha in beta matrix; beginning of
reaction
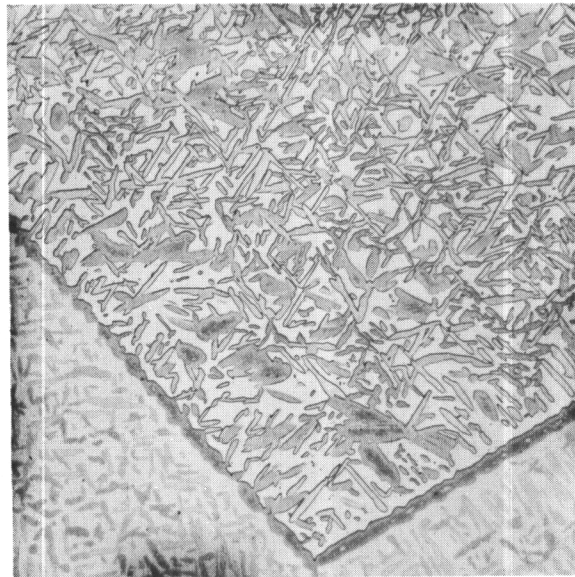
500X   VHN 344   96185

FIGURE 34. 7.72 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 650 C FOR
312 HOURS

Structure: Widmanstätten alpha in beta matrix

FIGURE 35. ISOTHERMAL-TRANSFORMATION DIAGRAM FOR THE Ti-7.72 Mn ALLOY
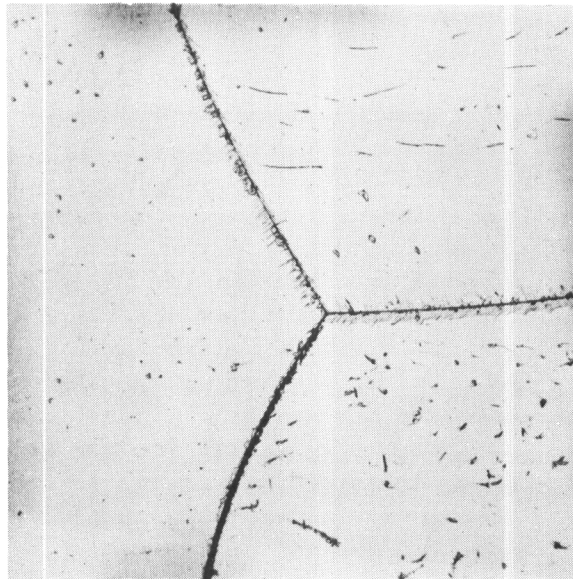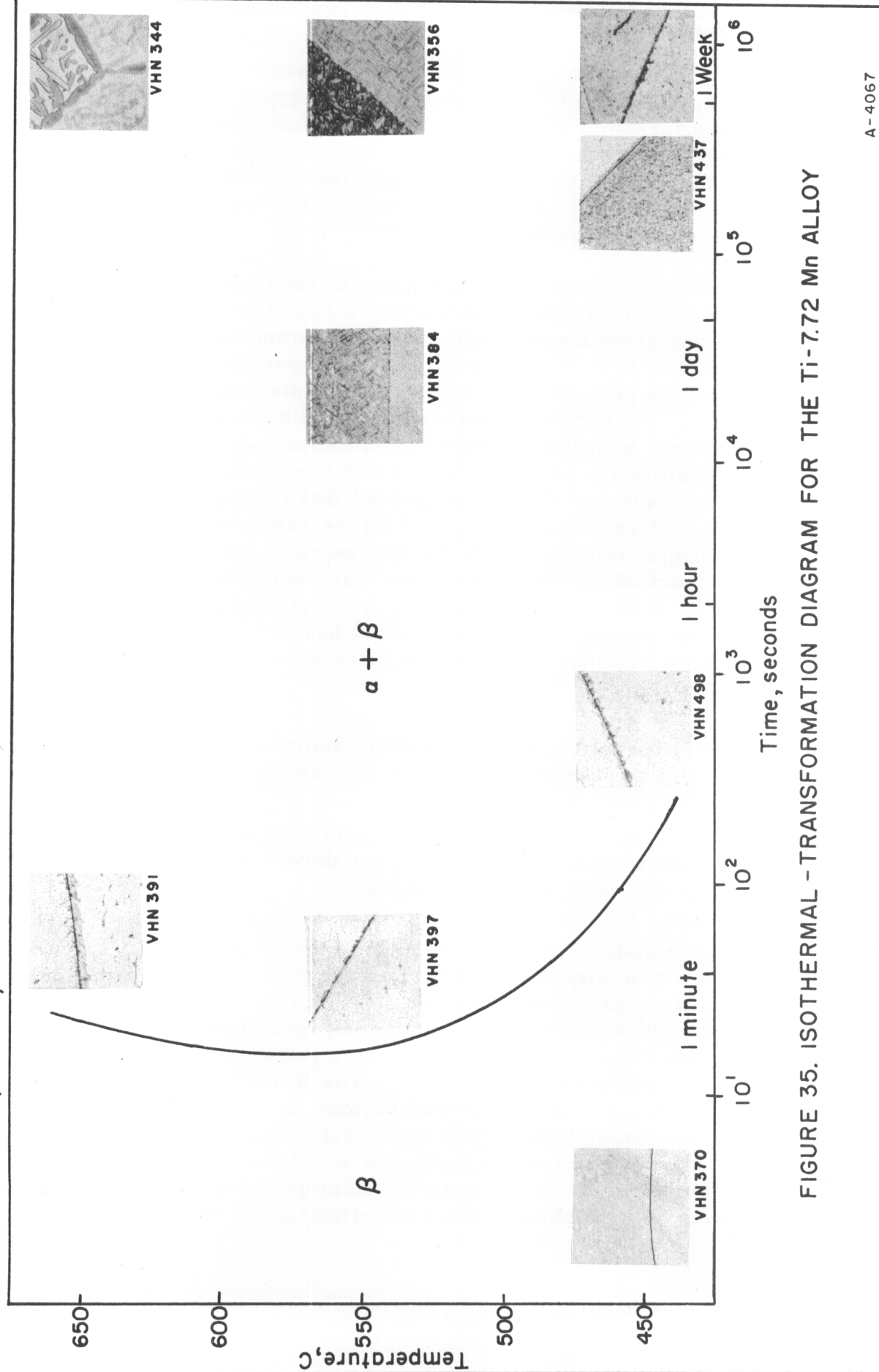
X-Ray Diffraction Studies

A systematic study of the progress of the transformation of this alloy was made by X-ray diffraction. The techniques and types of samples have been described in a foregoing section. Of the specimens transformed at 450 and 550 C, samples were chosen at representative time intervals from 10 seconds to over 300 hours. At 650 C, only specimens transformed from 10 to 60 seconds were examined. The observations made from X-ray photograms of these samples are summarized in Table 19.

The first X-ray diffraction examination with vanadium radiation of some of the samples in which beta phase had not begun to decompose showed a face-centered-cubic phase of lattice constant 4.36 angstroms, and a lattice constant for beta larger by 0.02 to 0.05 angstrom than the expected value. However, when the samples were photographed with copper radiation, the extra phase was not observed and the lattice constant of beta had a reasonable value. The difference in behaviors with the two types of radiation indicated a surface effect, since the penetration of the vanadium radiation would be less than that of the copper. The additional factors suggested that (1) the extra phase was TiH, because of its diffraction pattern, and (2) the expansion of the beta lattice was caused by hydrogen introduced during the chemical polishing of the samples. These conclusions were confirmed when samples showing the extra phase were given an additional electropolish in the alcohol-$AlCl_3 \cdot ZnCl_2$ bath. After the electrolytic polish, the samples which had shown the TiH pattern previously showed no evidence of it, and no more than 0.01-angstrom expansion in the beta phase.

The hydrogen absorption occurred only in samples containing untransformed beta. Most of the samples seemed to be completely unaffected.

It appears now that the electropolishing bath may cause a change in the structure of the surface also. Several samples showed an extra phase and an expanded-beta structure after electropolishing.

At 650 C, no transformation was detectable by X-rays after 60 seconds, although some alpha was observed with the microscope. At 550 C, no transformation was observed at 60 seconds, but, at 5 minutes, alpha phase was present in considerable amounts, and the original beta phase had undergone a change in composition to an average of about 16 per cent manganese, according to lattice-constant measurements. The X-ray photograms showed that the basal (0002) plane of the alpha was parallel to the dodecahedral or (110) plane of the remaining beta. This relationship is illustrated in the portion of the photogram reproduced in Figure 36. If two neighboring spots from a rotating sample occur at the same vertical height on the film as do the 0002 reflections of the alpha and the 110 reflections of the beta phase,
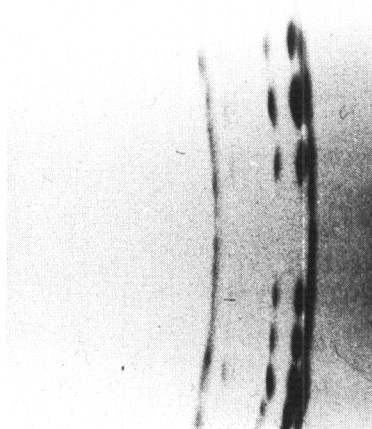
2-3/4X

FIGURE 36. PORTION OF X-RAY DIFFRACTION PHOTOGRAM OF Ti-7.72 Mn ALLOY TRANSFORMED 312 HOURS AT 550 C

Reflections, left to right, are (1010) Kα of alpha, (10$\bar{1}$1) Kβ of alpha, (0002) Kα of alpha, (110) Kα of enriched beta, and (10$\bar{1}$1) Kα of alpha, respectively; vanadium radiation.
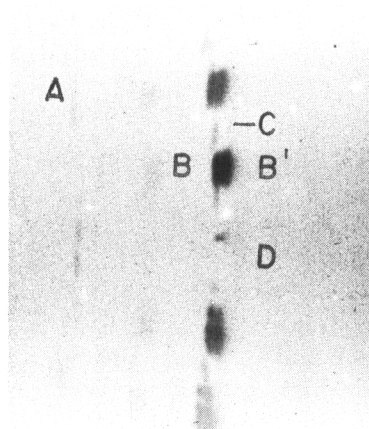
2-3/4X

FIGURE 37. PORTION OF X-RAY DIFFRACTION PHOTOGRAM OF Ti-7.72 Mn ALLOY TRANSFORMED 60 MINUTES AT 450 C

Reflections A and C are the 2.81- and 2.30-Å interplanar spacings of ω-phase, B and B' are the 110 reflections of the beta phase of lattice constants 3.26 and 3.23 Å, respectively, D is a 2.23-Å reflection believed to be the (10$\bar{1}$1) reflection of alpha; vanadium radiation.

TABLE 19. RESULTS OF X-RAY DIFFRACTION EXAMINATION OF ISOTHERMALLY TRANSFORMED Ti-Mn ALLOYS

X-Ray No.	Manganese Content, wt %	Transformation Temp, C	Transformation Time	Intensities of Phase Patterns Observed[1]				Lattice Constant of β-Phase, Å	Deduced Mn Conc of β, wt %
				β	α	ω	Other Phases		
45B	7.72	650	10 sec	VS β_o	0	0	(2) (3)	3.260(4)	-
46A	"	650	20 sec	VS β_o	0	0	(2)	- (5)	-
47A	"	650	30 sec	VS β_o	0	0	0	3.25	8
48A	"	650	60 sec	VS β_o	0	0	0	3.246	8
31A	"	550	10 sec	VS β_o	0	0	(2)	- (5)	-
32A	"	550	30 sec	VS β_o	0	0	0	- (5)	-
44A	"	550	60 sec	VS β_o	0	0	0	3.276?(4)	-
38A	"	550	5 min	VS β_u	MS	0	0	3.22	14
39A	"	550	30 min	VS β_u	S	0	0	~3.21	16
40A	"	550	4 hr	VS β_u	S+	0	0	3.21	16
41A	"	550	48 hr	VS β_u	S+	0	0	~3.21	16
42A	"	550	312 hr	VS β_u	S+	0	0	~3.21	16
27B	"	450	10 sec	VS β_o	0	0	(2) (3)	3.26(4)	-
29A	"	450	30 sec	VS β_o	0	0	(2)	- (5)	-
26A	"	450	5 min	VS β_o	0	0	0	- (5)	-
50A	"	450	10 min	VS β_o	0	F	0	β_o ~3.25	8
36A	"	450	30 min	F, VD β_r / VS β_o / S, VD β_r	0	MF+	0	β_r 3.25-3.22 / β_o 3.26 / β_r 3.26-3.22	8-14 / 5 / 5-14
49A	"	450	60 min	VS β_o / VS, D β_r	VF d	M	0	β_o 3.26 / β_r 3.24-3.20	5 / 10-18
28A	"	450	4 hr	VS β_u	S	0	0	3.199	18
30A	"	450	48 hr	S β_u	MS	0	0	3.184	21
37A	"	450	168 hr	VS β_u	S	0	0	3.188	20
59A	"	450	408 hr	VS β_u	S	0	0	Trace of unknown	3.197	18

Footnotes appear on the following page.

Footnotes for Table 19:

(1) Intensity symbols: S = strong; M = medium; F = faint; V = very; D = diffuse.

   Nomenclature:

   β_o = beta phase of original manganese content
   β_u = beta phase enriched in manganese to its ultimate (essentially eutectoid) composition
   β_r = beta phase of reduced lattice constant between β_o and β_u
   ω = omega phase of unknown structure and composition (see text)

(2) TiH and hydrogen-expanded beta phase were observed in this sample originally but are now believed to have resulted from the chemical polish used.

(3) Electropolishing was found to remove the TiH and most of the hydrogen expansion from this sample.

(4) Lattice constant measured after hydrogenized layer was electropolished off.

(5) Values measured for this sample are not quoted because hydrogen apparently had entered into the beta lattice.

then the planes causing these reflections must be tilted at the same angle to the axis of the camera, but may make any possible angle with respect to each other. If, however, the two reflections appear at the same height when the sample is held stationary, both planes must make the same angles with the axis of the camera and the same angle with the X-ray beam and, therefore, must be parallel to each other. The sample of Figure 36 was rotated during exposure. However, in tests made with stationary samples of titanium-chromium alloys, parallel 0002 alpha and 110 beta reflections were obtained. The circumferential broadening of the spots in Figure 36 represents the range of angular deviation from the truly parallel condition, which is probably due to a slight misfit between the alpha and beta lattices. This crystallographic orientation between the beta and alpha phases is consistent with the Widmanstätten structure observed microscopically in this specimen.

Comparison of the X-ray photograms of the sample transformed 312 hours at 550 C with that of the sample transformed 5 minutes shows only a slight increase in the intensity of the alpha reflections, accompanied by an increase in the apparent manganese content of the beta phase. No evidence of the pattern of the titanium-manganese compound was found.

At 450 C, there was apparently an intermediate step in the decomposition of the original beta into alpha plus beta of near-eutectoid composition. The X-ray photograms showed retained beta after a transformation time of 5 minutes. After 10 minutes, however, the diffraction spots of the beta phase, while remaining sharp, developed streaks in the direction of increasing Bragg angle only. These streaks indicated that the manganese concentration was being increased within local regions in the beta phase. At the same time, reflections from a new phase, tentatively designated omega (ω), made their appearance. At 30 minutes, the intensity of the omega pattern was much increased, as was that of the streaking from the beta diffraction spots. After 60 minutes, the streaks from the beta spots were considerably sharpened, so that each beta spot was doubled, and beta appeared to exist with two discrete lattice constants, 3.26 and about 3.23 angstroms. The pattern of omega phase had increased still further in intensity, and the presence of a small amount of the alpha phase was indicated. A portion of the X-ray photogram of the 60-minute sample is shown in Figure 37. In the specimen transformed for 4 hours, only alpha and beta of approximately eutectoid composition could be detected, and the X-ray diffraction photogram was essentially the same as Figure 36.

The omega phase, then, is apparently an intermediate metastable phase which is nucleated sooner than alpha but disappears as alpha forms. Since it appears simultaneously with local increases in manganese concentration of the beta, it is probably poorer in manganese than the original alloy.

As observed with vanadium radiation, omega appears to fit a cubic lattice with lattice constant of 4.00 angstroms. However, with copper radiation, one or two lines forbidden by this small unit cell appear, so that the lattice constant must be raised to a multiple of 4 angstroms, 8 angstroms apparently being satisfactory. Work on this phase is not complete, and its tentative classification as cubic is subject to revision by further work in progress. Data taken from the diffraction photograms of a typical sample containing the omega phase, as observed with both vanadium and copper Kα-radiation, are given in Table 20.

The exact composition and structure of the omega phase are as yet unknown. However, it has some crystallographic relationship to beta, for each interplanar spacing of beta appears to coincide with an interplanar spacing of omega.

The omega phase may have considerable technological importance, as will be shown elsewhere in this report. Diffuse patterns similar to omega have been found recently in age-hardened, beta-stabilized alloys of other binary systems. In these patterns, it appears that omega is definitely pseudocubic, rather than cubic.

It is possible that the omega phase is an unstable phase of pure titanium made metastable by interstitial impurities. Since hydrogen is the only one of the interstitial elements which can be removed from the alloys (by vacuum annealing), experiments are under way to determine whether this element is a factor in the formation of the omega phase.

Hardness Tests

Hardnesses obtained on the isothermally transformed specimens are presented graphically in Figure 38   An interesting correlation between the presence of the omega phase as detected by X-rays and the hardness of the specimens may be observed from this figure. It may be noted that the hardness of the specimens transfomed at 450 C increases steadily with increasing transformation time to a maximum of about 535 VHN after 1 hour. The hardness then decreases sharply to a value of about 475 VHN after 4 hours' transformation   The omega phase was detected in these specimens at transformation times of 10 minutes to 1 hour, and was absent at times of 4 hours or longer. Thus, it appears possible that the high hardness level attained in this alloy was due, in part at least, to the presence of the omega phase. The specimens transformed at 500 and 550 C also show a rise in hardness during the first hour, although no omega phase was detected in the latter specimens. However, the magnitude of the hardness increase is much less than that
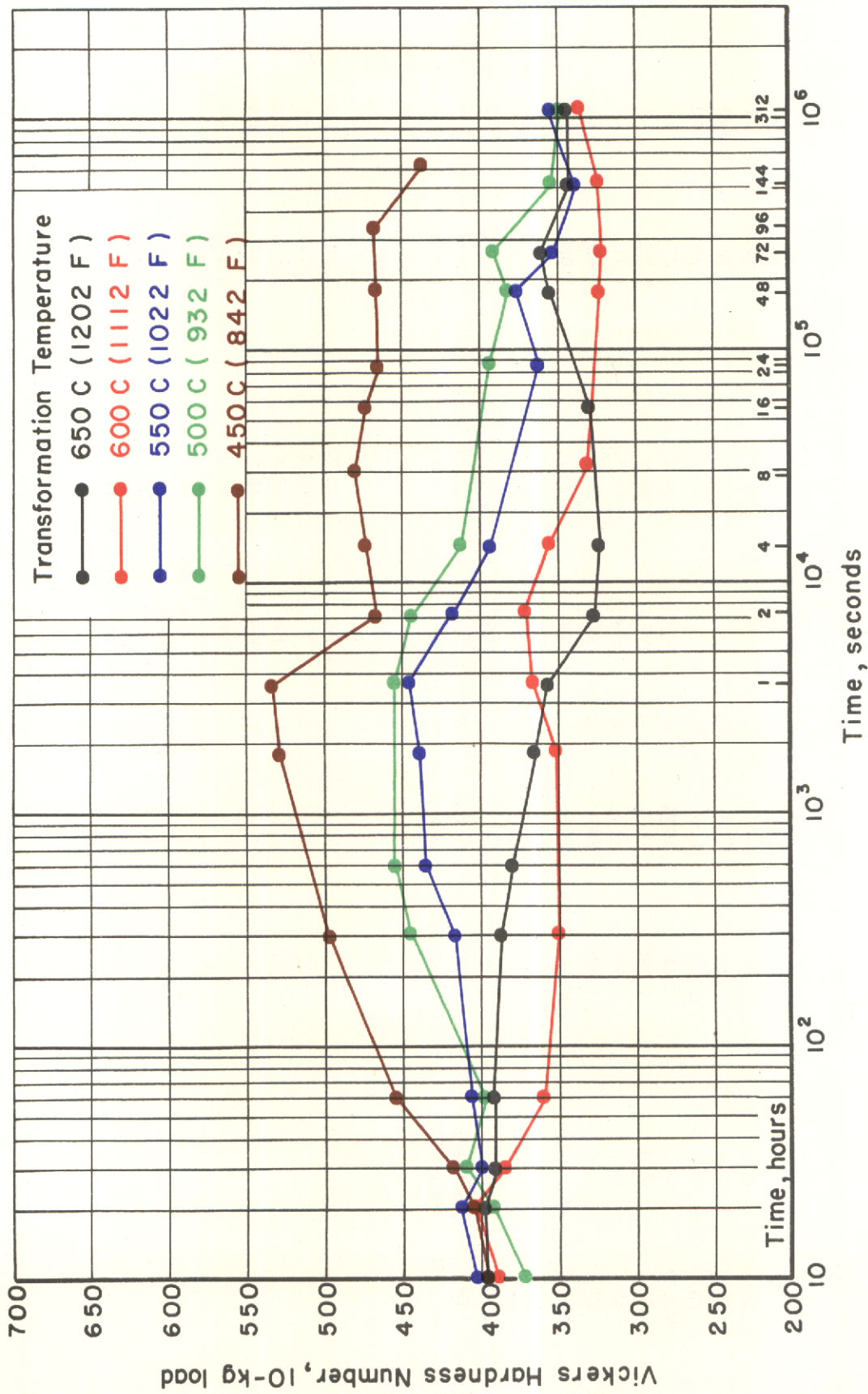
FIGURE 38. HARDNESS VERSUS TIME FOR ISOTHERMALLY TRANSFORMED SPECIMENS OF THE Ti-7.72Mn ALLOY

A-4068

TABLE 20. ANALYSIS OF DIFFRACTION PATTERNS[1] OF THE Ti-7.72Mn ALLOY ISOTHERMALLY TRANSFORMED 60 MINUTES AT 842 F (450 C)

Unfiltered Vanadium Radiation

Phase[3]	I[2]	Bragg Angle, θ,°	Interplanar Spacing, Å[4]	NKL
ω	MF+	26.4	2.82	--
$\beta_o + \beta_r$	F, D	30.1	KB 2.28	110
ω	M	33.0	2.30	--
β_o	VS	33.0	2.30	110
β_r	VS	33.4	2.27	110
ω	VF, D	34.1	2.23	10T1
ω	F	44.6	1.783	--
ω	M	50.2	1.630	--
β_o	MS	50.2	1.630	200
β_r	MF, D	50.2-51.4	1.63-1.60	200
ω	VVF	53.7	KB 1.417	--
ω	M+	62.5	1.412	--
ω	F	70.1	1.332	--
β_o	M+	70.1	1.332	211
β_r	MS, VD	71.1-72.9	1.324-1.310	211

Copper Radiation With Nickel Filter

Phase[3]	I[2]	Bragg Angle, θ,°	Interplanar Spacing, Å[4]	NKL
ω	VVF+	15.0	2.79	--
$\beta_r + \beta_o$	VF	17.8	KB 2.28	110
ω	F	19.7	2.28	--
β_o	s	19.7	2.28	110
β_r	s	19.9	2.26	110
ω	VVF	~24.3	~1.87	--
ω	VF	25.7	1.77	--
ω	VVF	26.7	1.71	--
ω	MF	28.3	1.620	--
β_o	M+	28.3	1.620	200
β_r	MFd	28.7	1.600	200
ω	VVF	29.8	1.547	--
ω	MF	33.2	1.402	--
ω	F	35.5	1.322	--
β_o	MF+	35.5	1.322	211
β_r	MF+	36.0	1.308	211
ω	M	39.9	1.200	--
β_o	MF	42.0	1.150	220
β_r	F+	42.7	1.133	220
ω	F	48.4	1.026	--
β_o	F+	48.4	1.026	310
β_r	VF+	49.3	1.013	310
ω	VF	50.2	1.000	--
ω	F	52.8	0.965	--
	VVF+	54.1	0.949	--
β_o	F	55.0	0.939	222
β_r	F	56.0	0.927	222
ω	F	57.3	0.913	--
ω	VF	60.0	0.888	--
β_o	F+	62.15	0.869	321

TABLE 20. (Continued)

	Unfiltered Vanadium Radiation					Copper Radiation With Nickel Filter			
Phase(3)	I(2)	Bragg Angle, θ,°	Interplanar Spacing, Å(4)	NKL	Phase(3)	I(2)	Bragg Angle, θ,°	Interplanar Spacing, Å(4)	NKL
					β_r	VF+	63.3	0.860	321
					ω	VF+	64.5	0.850	--
					ω	F	75.9	0.793	--

(1) Made in 5.73-mm Debye camera, Straumanis setting.

(2) I is average intensity of each spot and does not consider the effect of the multiplicity factor on total line intensity. For symbols, see Table 19.

Nomenclature:

(3) β_o = beta phase of original manganese content.

β_r = beta phase of reduced lattice constant, indicating enrichment in manganese.

ω = omega phase of unknown structure and composition.

(4) Interplanar spacings not corrected absorption.

observed in the specimens transformed at 450 C. Thus, if the increase in hardness were connected with the formation of omega, this phase may have been present in too small an amount to be detectable by X-ray diffraction.
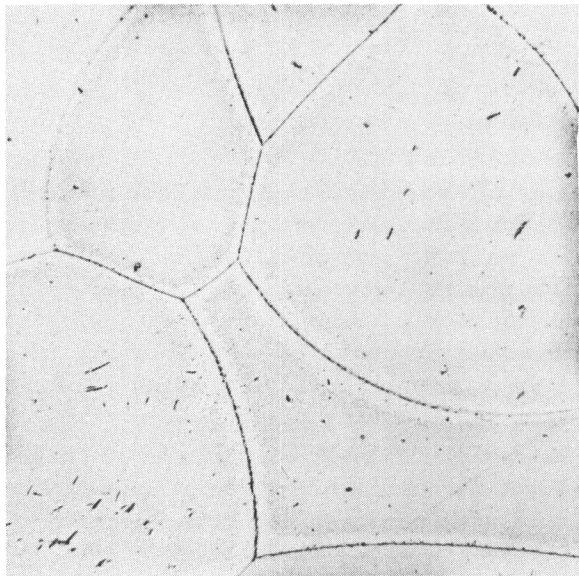

Titanium-12.3 Per Cent Manganese Alloy

The transformation characteristics of this alloy were very similar to those of the Ti-7.72Mn alloy. The time for the initiation of the alpha rejection was increased by the higher alloy content. The microstructures observed at the various transformation temperatures were very similar to those of the previously discussed alloy, except that, at longer transformation times at the higher temperatures, relatively more beta phase was present. Representative microstructures for this alloy are presented in Figures 39 through 44, inclusive. A complete time-temperature-transformation diagram for this alloy is presented in Figure 45.

Hardness curves are presented in Figure 46. It may be noted that this alloy shows a rise in hardness at all transformation temperatures except 650 C somewhat similar to those shown for the 7.72 per cent manganese alloy at 450 to 550 C. The time for initiation of this rise in hardness is longer in the 12.3 per cent manganese alloy, as would be expected from the sluggish nature of its transformation process. The omega phase may be responsible for the high hardnesses, but this has not been verified by X-ray work as yet. On the basis of the excellent tensile properties currently being obtained in complex alloys by overaging treatments, it is believed that the high-manganese alloy could also develop good properties if aged long enough to produce softening.

A STUDY OF THE HARDENING MECHANISM IN BETA-STABILIZED TITANIUM ALLOYS

It has been recognized for some time that the high strength and hardness attained in many beta-stabilized titanium alloys was primarily due to a submicroscopic reaction in the retained beta present. High hardnesses can be produced in these alloys by cooling at a suitable rate from the temperature range in which the high-temperature beta phase is stable, or by cooling rapidly from this range and reheating to lower temperatures. Such behavior is typical of coherency- or precipitation-hardening types of reaction in other systems. In titanium alloys, it was believed generally that this coherency-hardening reaction involved the precipitation of the alpha phase from the beta. However, no actual proof of this postulated mechanism has been offered up to the present time.

500X   96186

FIGURE 39. 12.3 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 450 C FOR
30 MINUTES

Structure: Alpha in beta matrix; beginning of
reaction

500X   VHN 500   96187

FIGURE 40. 12.3 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 450 C FOR 168
HOURS

Structure: Alpha in beta matrix
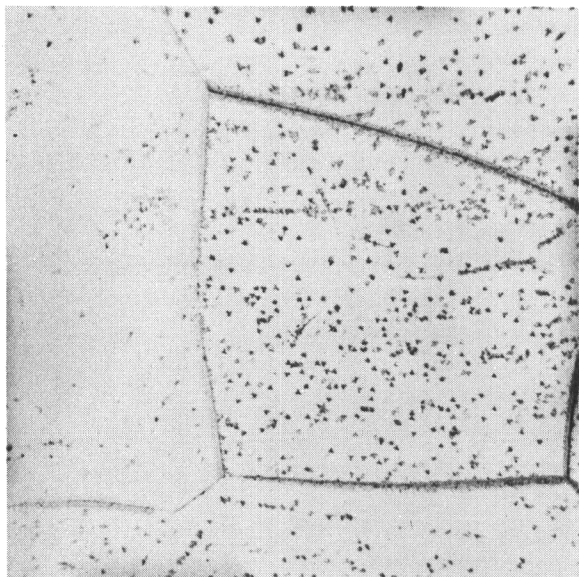
500X   VHN 347   96189

FIGURE 41. 12.3 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED FOR FIVE MINUTES
AT 550 C

Structure: Alpha in beta matrix; beginning of reaction
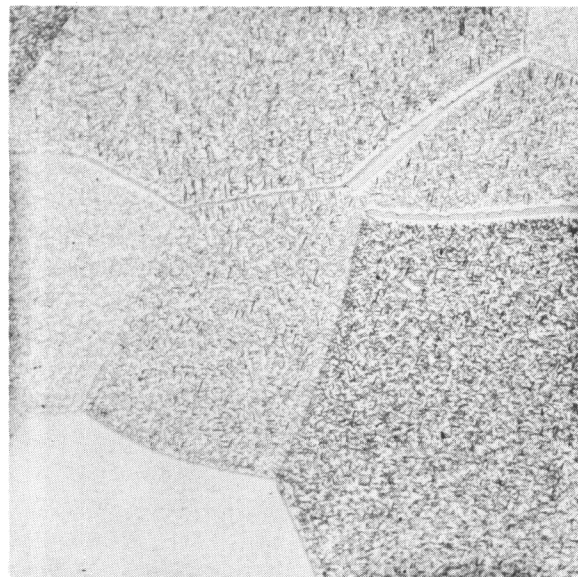
500X   VHN 364   96191

FIGURE 42. 12.3 PER CENT MANGANESE ALLOY
ISOTHERMALLY TRANSFORMED AT 550 C FOR
312 HOURS

Structure: Alpha in beta matrix
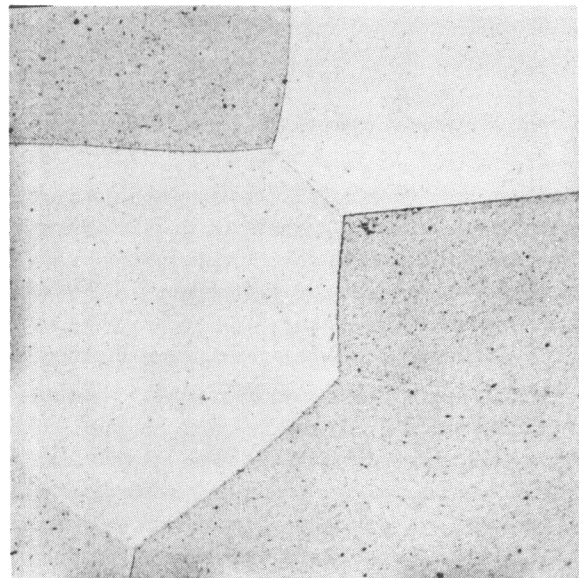
500X   VHN 345   96192

FIGURE 43.   12. 3 PER CENT MANGANESE ALLOY ISO-
THERMALLY TRANSFORMED AT 650 C FOR 10 MINUTES

Structure: Alpha in beta matrix; beginning of reaction

500X   VHN 355   96193

FIGURE 44.   12. 3 PER CENT MANGANESE ALLOY ISO-
THERMALLY TRANSFORMED AT 650 C FOR 312 HOURS

Structure: Widmanstätten alpha in beta matrix
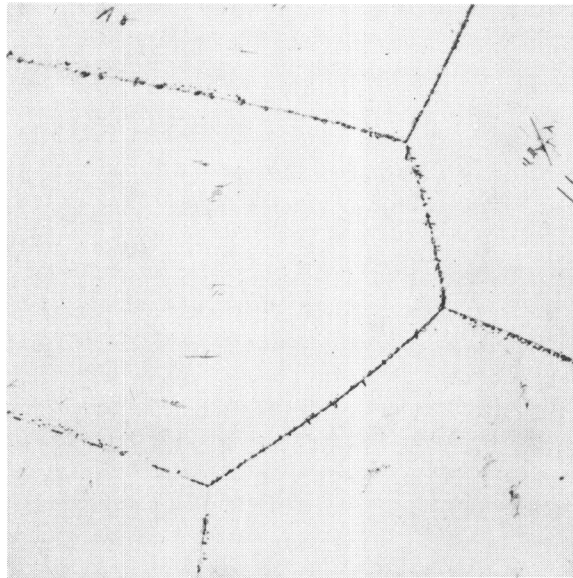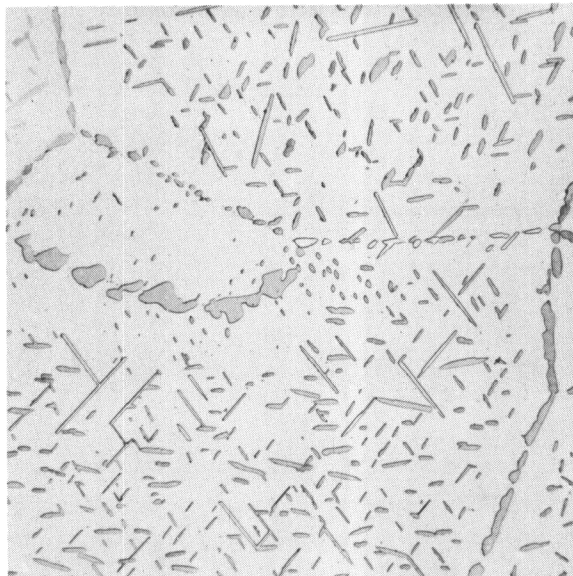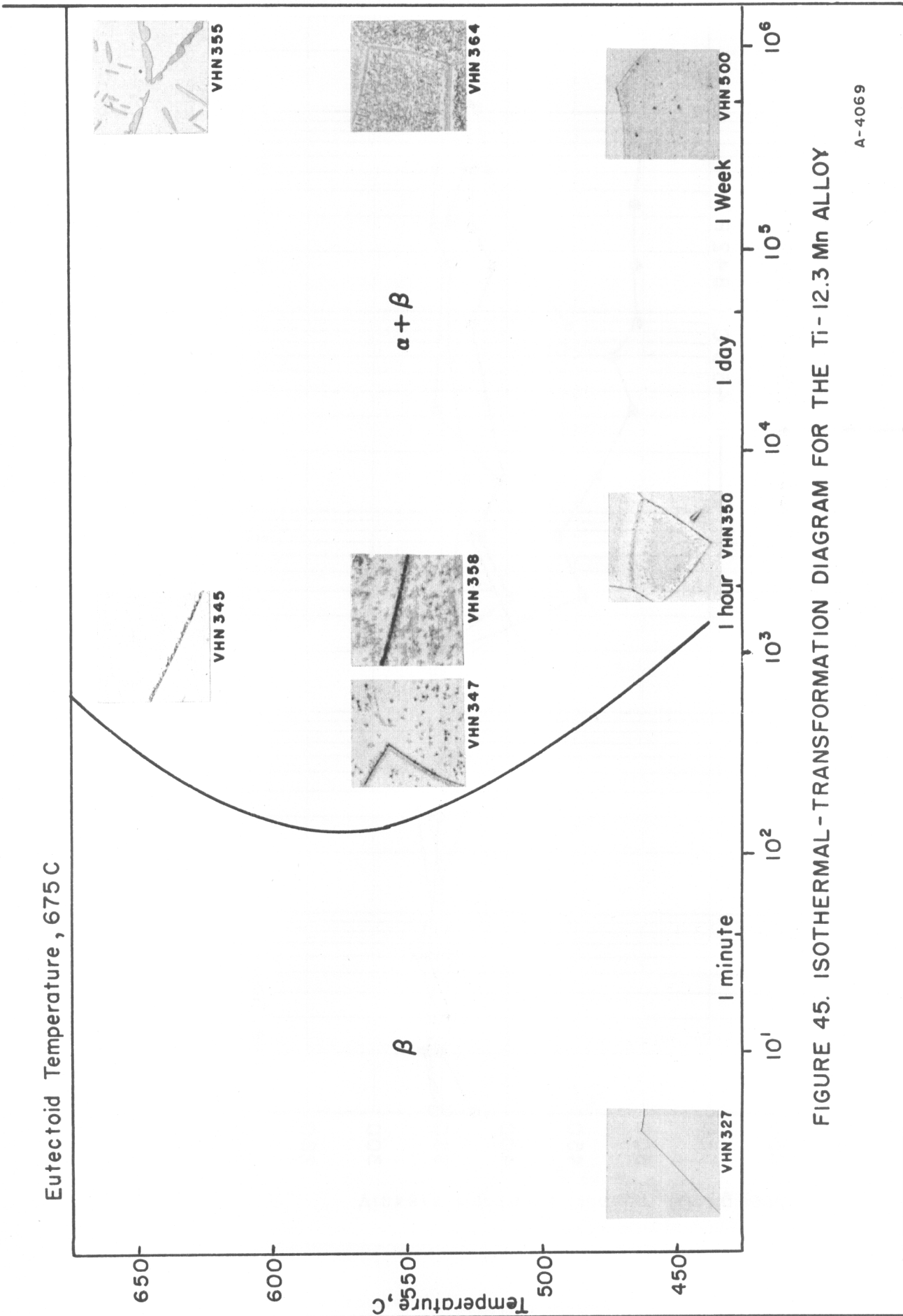
FIGURE 45. ISOTHERMAL-TRANSFORMATION DIAGRAM FOR THE Ti-12.3 Mn ALLOY

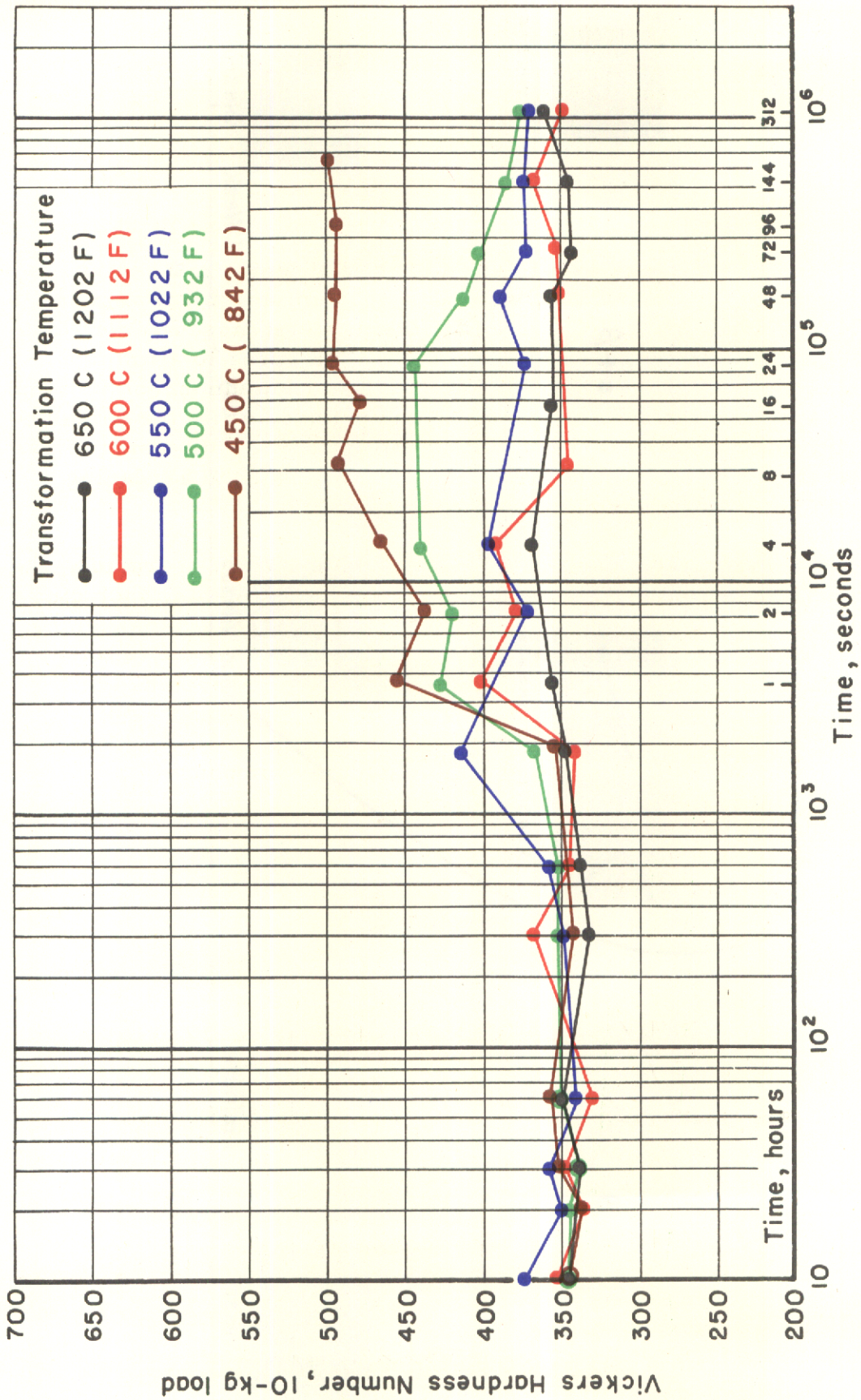Eutectoid Temperature, 675 C

A-4069

FIGURE 46. HARDNESS VERSUS TIME FOR ISOTHERMALLY TRANSFORMED SPECIMENS OF THE Ti-12.3Mn ALLOY

A-4070

Since the current alloy-development program included an extensive study of heat treatment, a better understanding of the basic hardening mechanism was highly desirable. In particular, it was felt that more knowledge of this hardening mechanism would lead to methods of improving the ductility of high-strength alloys. A number of heat treatments had been developed which produced high strength in many alloys, but, at the very high strength levels (200,000 psi), the ductility of such alloys decreased to almost zero.

Consequently, a limited investigation of the hardening mechanism was undertaken, using binary alloys of the titanium-chromium, titanium-iron, and titanium-molybdenum systems. The binary alloys were chosen rather than the more complex alloys currently being investigated, in order to eliminate as many variables as possible in the initial work. The titanium-chromium and titanium-iron systems contain intermetallic compounds, while the titanium-molybdenum system is of the terminal-solid-solution type. By using alloys of the latter system, it was hoped to prove conclusively that the hardening mechanism did not involve any compound formation.

The alloys were to be investigated in both the unhardened (rapidly quenched) condition and after various age-hardening treatments, including overaging until a visible precipitate appeared. The progress of hardening was to be followed by electron microscopy and various X-ray diffraction techniques. The X-ray diffraction techniques to be applied included the following:

1. Debye photograms of polycrystalline samples for phase identification.

2. Transmission Laué photograms on single crystals of very large-grained specimens to detect coherency by the methods used by Guinier[2], Preston[3], and others.

3. Photograms made in a Weissenberg camera on single crystals to provide more exact identification of any precipitate, and information on matrix-precipitate relationships.

Work on this investigation was begun rather late in the period covered by this report, and is only partially completed. The results achieved to date are presented in the following sections.

Materials Used

The materials used in this investigation were in the form of 14-gage (0.064-inch) hot-rolled sheet. The alloys had been double melted as 3/4- to 1-pound ingots, forged at 1750 to 1800 F, and rolled to sheet at 1450 F. The heat numbers and nominal compositions are given on the following page.

Heat No.	Nominal Alloy Content, wt %
WS152A	8Cr
WS256A	15Cr
WT99A	4Fe
WT101A	5Fe
WT95A	10Mo

The Ti-8Cr alloy had been used previously in the isothermal studies described in WADC TR-52-249. The remaining alloys had been used in exploratory work in alloy development.
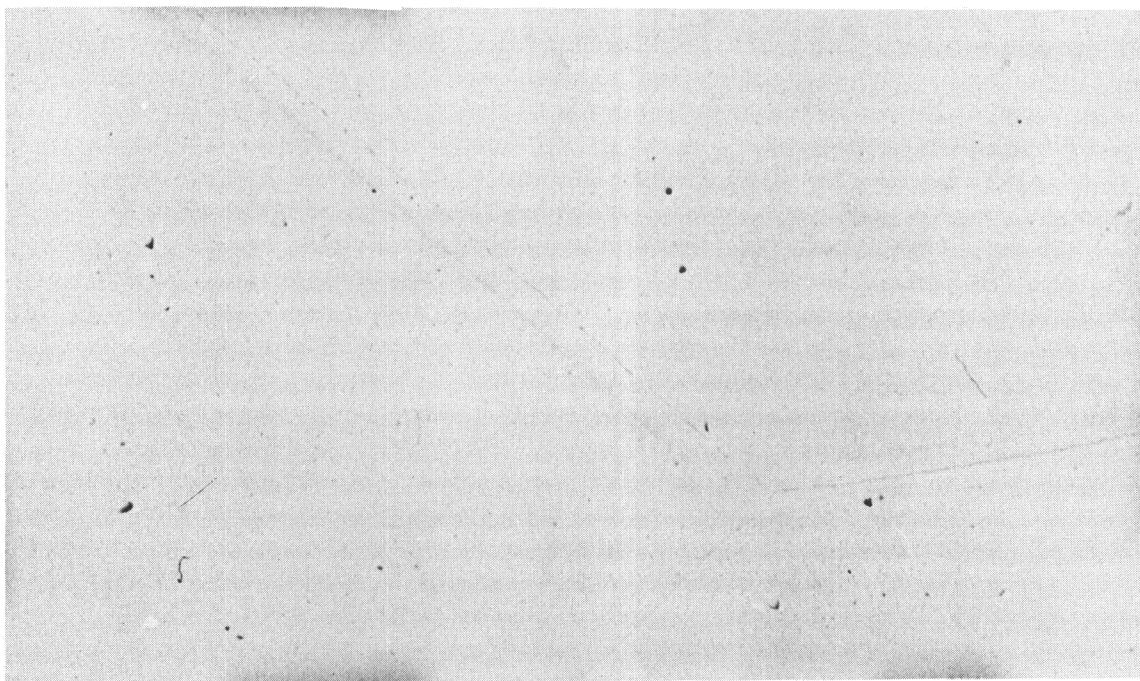
Electron Microscopy

Specimen Preparation

Only the Ti-8Cr, Ti-5Fe, and the Ti-10Mo alloys were used in this study. Sheet sections 1/2 by 1/2 by 0.064 inch of each alloy were heated in a dried-argon atmosphere at 1700 F (927 C) for one-half hour and quenched in iced brine. They were then aged for periods of 1 and 5 hours at 500 F (260 C), and 1, 2, and 4 hours at 700 F (371 C). The aged specimens were mounted in a room-temperature-setting resin, given a metallographic polish, and subjected to Vickers hardness tests on their cross sections. Average hardnesses so obtained are presented in Table 21. The specimens were then remounted in the room-temperature-setting resin with one of the 1/2 by 1/2-inch faces exposed. To remove surface contamination, approximately 0.015 inch was removed from the surface of each specimen by hand grinding on wet silicon carbide paper. The ground specimens were then electropolished in the Rem-Cru electrolyte described in the Appendix to this report, etched, and immediately covered with Zapon lacquer to prevent any corrosion effects from exposure to the air.

Both Formvar positive and silica positive replicas were prepared from thick negative Zapon replicas stripped from the surface of the specimens. The Formvar replicas were formed by flowing a dilute solution of Formvar over the Zapon and allowing to dry. Silica was vapor deposited on the Zapon to form the silica replicas. In both cases, the Zapon was dissolved away from the replicas in amyl acetate. Then Formvar replicas were shadowed with platinum at an angle of 1 to 5. The silica replicas were unshadowed.

Results

Thus far, only the specimens quenched from 1700 F and aged to maximum hardness at 700 F have been examined. The Ti-8Cr alloy, as quenched from 1700 F, shows slight evidence of a precipitate at 5000 diameters' magnification, as shown in Figure 47. Increasing the magnification

500X   VHN 331   E-3569C

FIGURE 47.   ELECTRON MICROGRAPH OF Ti-8Cr ALLOY
AS QUENCHED FROM 1700 F INTO ICED BRINE

Formvar replica; little evidence of precipitate
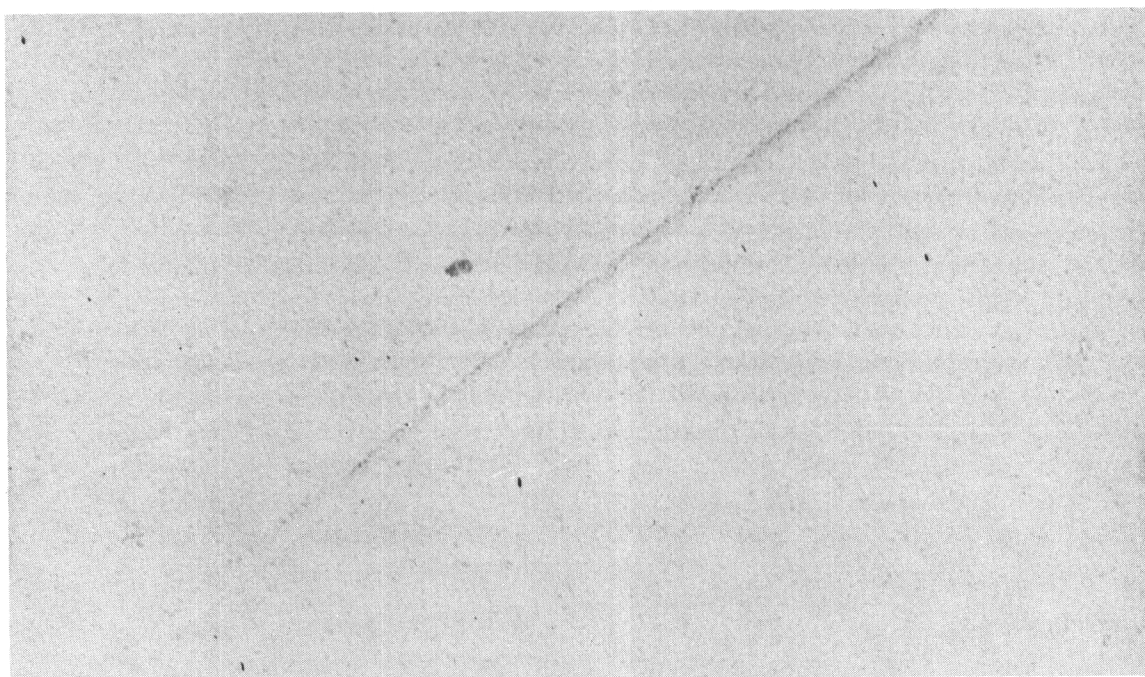
20,000X   VHN 331   E-3631C

FIGURE 48.   ELECTRON MICROGRAPH OF THE SAME SPECIMEN
AS SHOWN IN FIGURE 47 AT HIGHER MAGNIFICATION AND
USING SILICA REPLICA

Grain boundary shown is believed to contain a fine precipitate; widely
scattered particles of precipitate are also barely visible within the grains.

to 20,000 diameters, however, reveals what is apparently a very fine precipitate concentrated in the grain boundary. The silica replica revealed this structure somewhat better than the Formvar. Figure 48 is a reproduction of the structure revealed by the silica replica.

After aging for 4 hours at 700 F, the 8 per cent chromium alloy showed a very fine, general precipitate at 5000 diameters, as shown in Figure 49. At higher magnification (20,000X), this precipitate is revealed more clearly, as shown in Figures 50 and 51 for the Formvar and silica replicas, respectively.

Examination of these structures reveals that the precipitate particles generally are arranged in short, irregular rows, which frequently appear connected, as if they were the jagged edges of short platelets. This is somewhat more evident in Figure 50, which was made from a Formvar replica. This micrograph shows a tendency for the precipitate rows to be somewhat parallel in a given part of the field. The apparent difference in particle size shown by the two replica techniques is not a serious discrepancy, and may be explained readily by consideration of the differences in the replicas employed[4]. It is of interest to note that a very similar microstructure, showing apparent alignment of particles, has been observed in the study of coherency hardening of 24S-T aluminum alloy[5].

The Ti-10Mo alloy had structures very similar to those of the chromium alloy in both the quenched and the aged conditions, as shown in Figures 52 and 53. There seemed to be less tendency for the precipitate particles in the aged specimens to form in rows, as they did in the Ti-8Cr alloy.

Initial work on the Ti-5Fe alloy revealed no precipitate in either the quenched or aged conditions. However, this may be due to etching technique, rather than to an actual absence of the precipitate. Work is being continued on this alloy.

The X-ray diffraction studies described in the following section indicate that the precipitate observed in the titanium-chromium and titanium-molybdenum alloys may be the new omega phase.

X-Ray Diffraction

Specimen Preparation and Heat Treatment

The various X-ray techniques being applied in this investigation require specimens having different grain sizes. In attempting to produce single grains large enough for the transmission Laué and Weissenberg techniques, specimens of the alloys in the preceding section were heated for one-half hour at 1900 F and quenched in iced brine. Microscopic examination of these specimens revealed, however, that the grain size was of the order of 1 mm or less, and was too small for single-crystal work. Consequently, these specimens were used for phase-identification

work, using a Deybe camera of 57.3-mm diameter. In some instances, the specimens used for electron microscopy were also employed for phase-identification work. An attempt was made to produce the larger grain sizes needed for the single-crystal work using higher temperatures and longer times. A summary of the results obtained is given below:

Temperature	Time, hr	Approximate Diam of Average Grain, mm
2000 F (1090 C)	1	0.8
1950 F (1066 C)	4	1.0
1922 F (1050 C)	24	> 1
2012 F (1100 C)	20	1.5-2.0

The last treatment produced grains large enough for the single-crystal work, although larger grains would have been desirable.

For the Debye-camera work, sliver specimens were prepared from the quenched sheet sections by chemical etching. An attempt was made to produce thin specimens for the single-crystal work by electropolishing quenched sheet sections to very small thicknesses. However, the process was difficult to control, and had a disadvantage in that the edges of the specimens were rounded. Thus, no thin, tapered sections could be obtained. However, by a combination of electropolishing and chemical etching, thicknesses of the order of 0.008 inch were obtained on a few specimens. The work on these specimens is still in a very early stage.

In order to eliminate as much as possible the effects of inhomogeneity in the materials used, the initial X-ray work was done on the same specimen in both the quenched and the aged conditions. After photograms were obtained from the as-quenched specimens, they were aged for 2 hours at 500 F in a silicone-oil bath. Then additional photograms were taken of the same area of the specimen. In addition to this, Debye patterns were also obtained from electron-microscopy specimens of the Ti-8Cr and Ti-10Mo alloys in the aged condition (4 hours at 700 F). Results of the X-ray diffraction examinations completed to date are given in Table 22. The hardnesses of the specimens are included for ready comparison. In the case of the specimens aged for 2 hours at 500 F, the hardnesses given were estimated from those obtained on the electron-microscope specimens shown in Table 21.

It is evident from Table 22 that a new phase appears after aging in all of the alloys except the Ti-15Cr alloy. Evidence of this phase also was detected in the as-quenched specimen of the Ti-4Fe alloy, which, it will be noted, had a high hardness in the quenched condition. This phase, tentatively
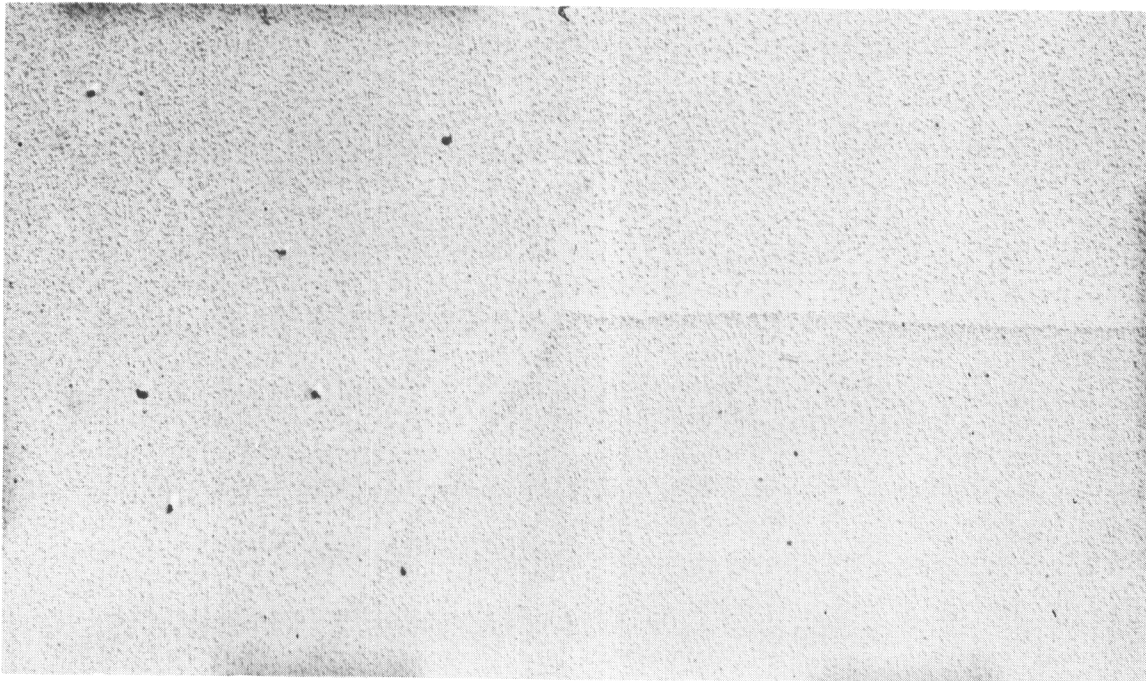
5000X   VHN 529   E-3568F

FIGURE 49.   ELECTRON MICROGRAPH OF Ti-8Cr ALLOY QUENCHED
AND AGED FOR FOUR HOURS AT 700 F

Shadowed Formvar replica; a general fine precipitate is clearly visible
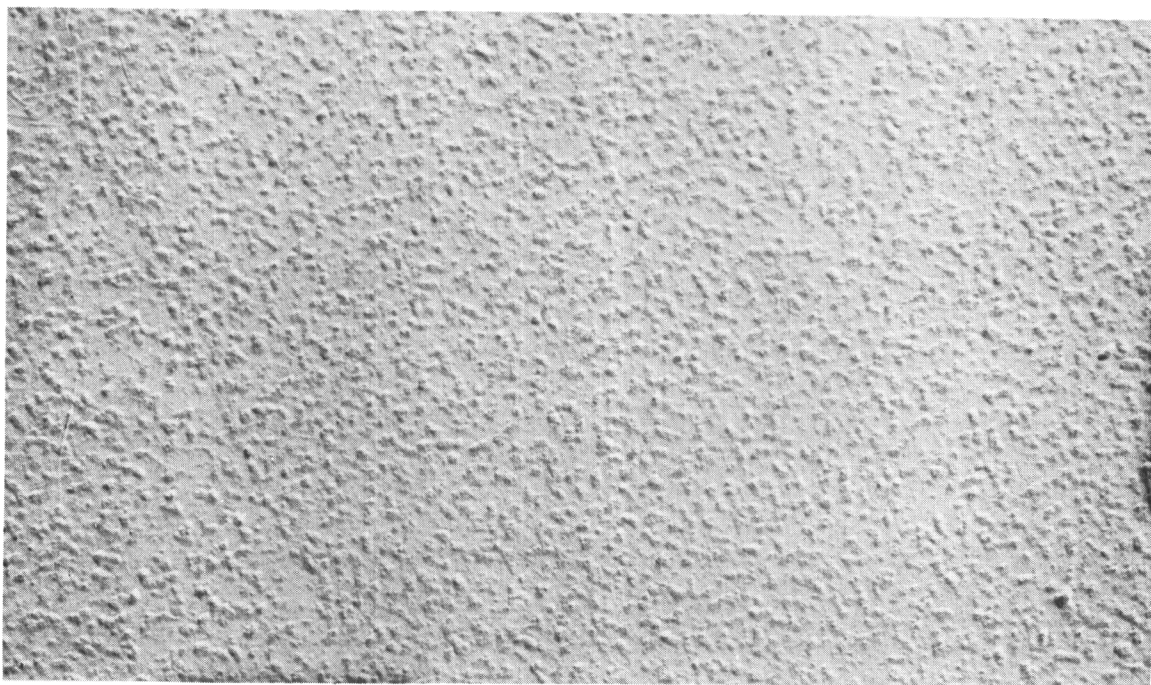
20,000X   VHN 529   E-3582B

FIGURE 50.   ELECTRON MICROGRAPH OF THE SAME SPECIMEN
SHOWN IN FIGURE 49 AT HIGHER MAGNIFICATION

Note apparent alignment of precipitate particles in short rows
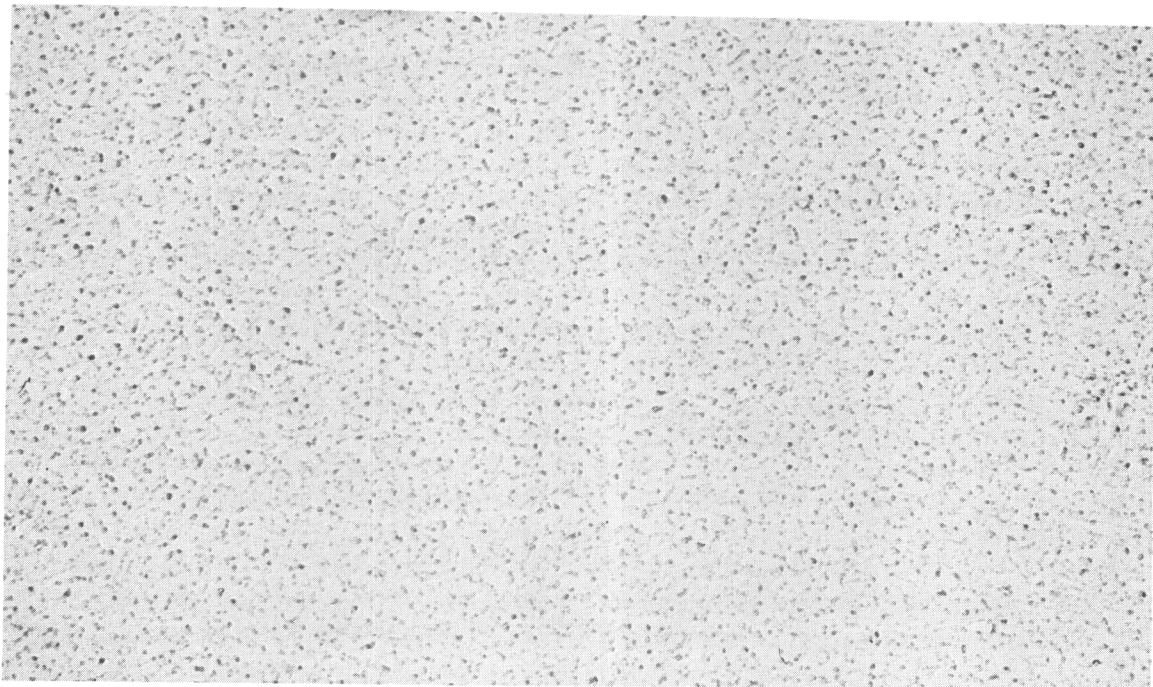
20,000X   VHN 529   E-3577D

FIGURE 51. ELECTRON MICROGRAPH OF THE SAME SPECIMEN SHOWN
IN FIGURES 49 AND 50, EXCEPT SILICA REPLICA WAS USED

Note apparent difference in particle size due to different replica
technique
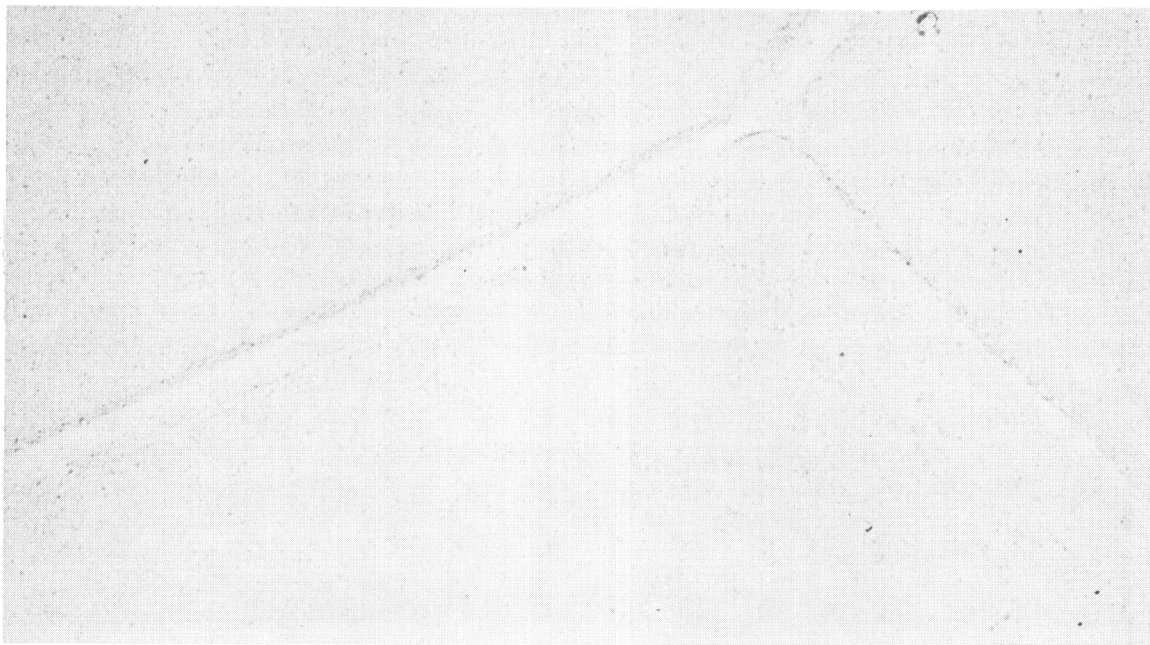
20,000X   VHN 317   E-3633B

FIGURE 52. ELECTRON MICROGRAPH OF Ti-10Mo ALLOY AS
QUENCHED IN ICED BRINE

Silica replica; fine precipitate at grain boundaries and widely
scattered through the grains

20,000X   VHN 504   E-3614-B

FIGURE 53. ELECTRON MICROGRAPH OF Ti-10Mo ALLOY QUENCHED
AND AGED FOUR HOURS AT 700 F

General fine precipitate present

TABLE 21. HARDNESS DATA ON HEAT-TREATED BETA-STABILIZED TITANIUM ALLOYS USED FOR ELECTRON MICROSCOPY

Heat No.	Nominal Alloy Content, weight per cent	Vickers Hardness Number (10-Kg Load)[1]					
		Aged at 500 F			Aged at 700 F		
		1 hr	5 hr	0 hr	1 hr	2 hr	4 hr
WS152A	8.0Cr	446	467	331	503	--	529
WT101A	5.0Fe	501	511	465	570	574	584
WT95A	10.0Mo	428	442	317	489	504	504

(1) Solution treated in argon at 1700 F for 1/2 hour; quenched in iced brine; aged in argon at temperatures and for times indicated.

TABLE 22.   HARDNESS AND X-RAY DIFFRACTION DATA[1] FOR QUENCHED AND AGED Ti-Cr, Ti-Fe, AND Ti-Mo ALLOYS

Nominal Composition, %	Quenching Temp, F	Quenching Medium	Aging Treatment	Hardness, VHN	Reductive Intensities of Phases[2]		
					β	α	ω
8Cr	1900	Iced brine	None	359	--	--	--
	1742	Liquid nitrogen	None	405	VS	0	MFd
	1900	Iced brine	2 hr at 500 F	450[3]	VS	0	VFd
	1700	Ditto	4 hr at 700 F	529	VS, Sh + D	0	M
4Fe	1900	"	None	534	VS	0	Fd
	1900	"	2 hr at 500 F	--	VS	0	Md
5Fe	1900	"	None	503	VS[4]	0	0
	1900	"	2 hr at 500 F	505[3]	VS	0	VF vd
10Mo	1900	"	None	339	S	0	0
	1700	"	4 hr at 700 F	504	VS	0	VF sh

(1) Photograms made in a 57.3-mm diameter Debye camera.
(2) Symbols: S = strong
              M= medium
              F = faint
              V = very
              D = diffuse
              Sh = sharp
(3) Estimated from Table 21.
(4) Beta-phase spots distorted.

named "omega", appears to be isomorphous with the intermediate phase detected in the Ti-8Mn alloy isothermally transformed at 450 C, described in the preceding section of this report. The structure and composition of the phase are as yet unknown. The interplanar spacings of the omega phase in the hardened alloys were very close to those shown in Table 20 of the preceding section. In the present relatively coarse-grained specimens, the reflections of the omega phase are discrete spots, as are the beta-phase reflections, showing that the omega lattice has a definite crystallographic orientation with the parent beta lattice. In the samples aged at 500 F, the reflection pattern of omega was very diffuse. The omega phase reported in the Ti-8Cr alloy quenched in liquid nitrogen was discovered only recently in diffraction patterns made earlier from oscillating-wedge samples. It had not been recognized as a new phase until the pattern of omega was well known, because of confusion with beta-phase reflections and the K-beta reflections of the beta phase.

The Debye photogram of the Ti-8Cr alloy aged at 700 F is unique among this group of samples. Not only is the pattern of omega rather sharp, but strong localized enrichment of the beta phase in chromium was indicated by streaking from the beta spots. The same phenomenon was observed in the Ti-8Mn alloy isothermally transformed at 842 F. The omega-phase pattern in the aged Ti-10Mo alloy was also sharp, but the beta-phase pattern was not streaked, indicating that localized enrichment had not occurred. However, it is conceivable that the enriched regions are present, but not detectable, owing to low periodicity.

Some Laué transmission patterns were obtained from both the aged and the unaged samples of the Ti-8Cr alloy. Copper radiation was used and the distance from sample to film was 3 cm. The unaged sample chemically polished to a thickness of about 0.015 inch, was examined first. The Laué pattern of this sample had round spots. Then the sample was chemically etched to a thickness of about 0.008 inch and examined again. After this further treatment, the Laué spots obtained always were elongated, the principal spots extending in a radial direction. In order to determine whether the streaking observed was a result of the chemical etching or of an aging phenomenon, a Ti-15Cr alloy, quenched from 1900 F and chemically etched to the required thickness, was examined. The hardness of this alloy is believed to be derived solely from solid-solution effects, and not from the transformation type of hardening. The Ti-15Cr alloy also gave radially elongated spots, indicating that the streaking observed in the Ti-8Cr alloy may have been caused by the chemical etching employed. Because of the observation of a TiH pattern and expansion of the beta phase in Debye photograms of some sliver samples described elsewhere, it is suspected that the entry of hydrogen could have caused this distortion of the crystals. Thus, for future work, a method of sample preparation which will not allow hydrogen penetration will be used.

The aged sample of the Ti-8Cr alloy was photographed repeatedly at various orientations having a mirror plane of symmetry in a search for the

principal poles for that grain. However, the orientation was never perfect enough to distinguish matrix from nonmatrix spots.

One specimen, that of the Ti-4Fe alloy quenched from 1900 F, has been examined in the Weissenberg camera. A sliver specimen of this alloy was etched until a particularly large grain was situated on the tip. This grain had its (311) direction parallel to the axis of the sliver, and could not be adjusted to give rotation about a principal axis. No pattern other than that of a perfect beta grain was obtained.

Discussion

From this work and the isothermal-transformation studies, it appears that a hitherto unsuspected phase, which has been named omega tentatively, is involved in the hardening mechanism of beta-stabilized titanium alloys. X-ray work on isothermally transformed titanium-manganese alloys indicates that this phase is a transition phase in the transformation of beta to alpha. The omega phase observed by X-rays may be the same phase as shown by the electron microscope to exist as a fine precipitate in age-hardened chromium and molybdenum alloys. Although a precipitate was not detected by electron microscopy in titanium-iron alloys, X-ray diffraction indicated the presence of omega phase in hardened alloys of this system. That this phase can be produced during continuous cooling from the beta field was indicated by the X-ray results obtained on the brine-quenched Ti-4Fe alloy and a Ti-8Cr alloy quenched in liquid nitrogen.

Whether this phase causes hardening by coherency with the beta matrix or with a subsequent alpha precipitate has not been established as yet. If the omega phase is the precipitate seen with the electron microscope, its hardening effect may be due entirely to its fine dispersion in the retained-beta phase. It is also possible that there is additional hardening induced by the formation of coherent alpha nuclei in the beta matrix, but this has not been disclosed yet. Whether the omega phase exists in overaged specimens which still retain considerable hardness also remains to be investigated. Further work along these lines is being conducted under the new contract on alloy development.

WELDABILITY STUDIES

One of the properties being considered in the development of titanium-base alloys is weldability. For the purpose of obtaining information concerning their welding characteristics, a group of alloys, which ranged from intermediate to high strengths and included both the alpha-beta and the metastable-beta structures, was selected for investigation. A preliminary test on a metastable-beta Ti-15Cr alloy (described in WADC-TR-52-249,

pages 162-168) produced a weld which had a minimum bend radius of 3T as welded. The results of this test prompted further work on other alloys.

The investigation was not designed to determine completely the welding characteristics of these alloys, but was conducted to obtain preliminary information concerning the effects of the welding cycle on the ductility of the heat-affected zone and the weld metal of arc-welded butt joints. The effects of a postweld heat treatment on the welded joints were studied also.

Welding Procedure

Inert-gas-shielded tungsten-arc welds were made in the specimens in a controlled-atmosphere welding chamber with an inert atmosphere. The test specimens were 1/16 inch thick, 5 inches long, and 1 inch wide. Filler rods approximately 1/8 inch wide were sheared from each of the alloy sheet materials for use in making the welds. The metastable-beta alloys were welded in the as-rolled condition, while the alpha-beta alloys were given a stabilizing heat treatment which consisted of heating to 1350 F for one-half hour, furnace cooling at a rate of approximately 3 F per minute to 1150 F, followed by air cooling to room temperature.

Before welding, the specimens were grit blasted, etched in a 4 per cent hydrofluoric acid - 10 per cent nitric acid - 86 per cent water solution, and wire brushed. This cleaning method appeared to remove surface films and left the sheet bright.

The specimens were placed in a jig to form a square butt joint with no root opening. The jig consisted of a mild-steel backup bar with a 1/4 x 1/16-inch groove on which the specimens were held in place by copper bars and C-clamps. The jigs were then placed in the controlled-atmosphere chamber, which was purged by evacuating to a pressure of approximately 50 microns and filled with helium.

Single-pass square-butt-joint welds were made in the specimens with the welding conditions listed below:

1. Direct current, straight polarity

2. 70 amperes

3. 18 to 22 volts

4. 1/16-inch thoriated-tungsten electrode

5. 7 inches of arc travel per minute

Test Procedures

The welds were inspected for appearance and uniformity. All welds were bright and shiny at the face and root and appeared to be entirely free of any surface films. During welding, a black powder formed along the edges of the weld and on the copper hold-down bars. This deposit was not analyzed.

Then the 5-inch weld specimens were cut in half; one-half was tested as welded, while the other half was given an alpha-beta heat treatment. This heat treatment consisted of heating to 1350 F for one-half hour and furnace cooling at a rate of approximately 3 degrees F per minute to 1150 F, from which temperature the specimens were water quenched. The heat treatment was made in a stainless steel retort which was sealed and had argon passing through it during heating. When the temperature of 1150 F was reached, the retort was removed from the furnace, the seal broken, and the specimens quenched in water.

The ability of the base metal, heat-affected zone, and weld metal to deform plastically together was tested by making longitudinal bend tests on the welds. The welds were bent to an angle of 90 degrees around dies of progressively decreasing radii until failure occurred, and the smallest radii around which the specimen could be bent without failing were recorded as the minimum bend radius of the weld. Failure was considered to have occurred when an open defect greater than 1/16 inch in any direction was observed.

Test Results

Figures 54 and 55 show longitudinal bend specimens which were tested and illustrate the degree of bending which was reached when failure occurred.

Table 23 lists the physical properties of the alloys tested and the minimum bend radii of the specimens in the as-welded and heat-treated conditions. It was found that generally the alpha-beta alloys had very little ductility in the as-welded condition, but that the ductility of the welded specimen could be improved by heat treatment. The beta alloys, with the exception of the 8 per cent molybdenum - 6 per cent iron alloy, were brittle in both the as welded and heat treated conditions. The Ti-8Mo-6Fe alloy had some ductility as-welded but was embrittled by the heat treatment. From examination of the fractures, it appeared that most of the alloys failed in the weld metal.
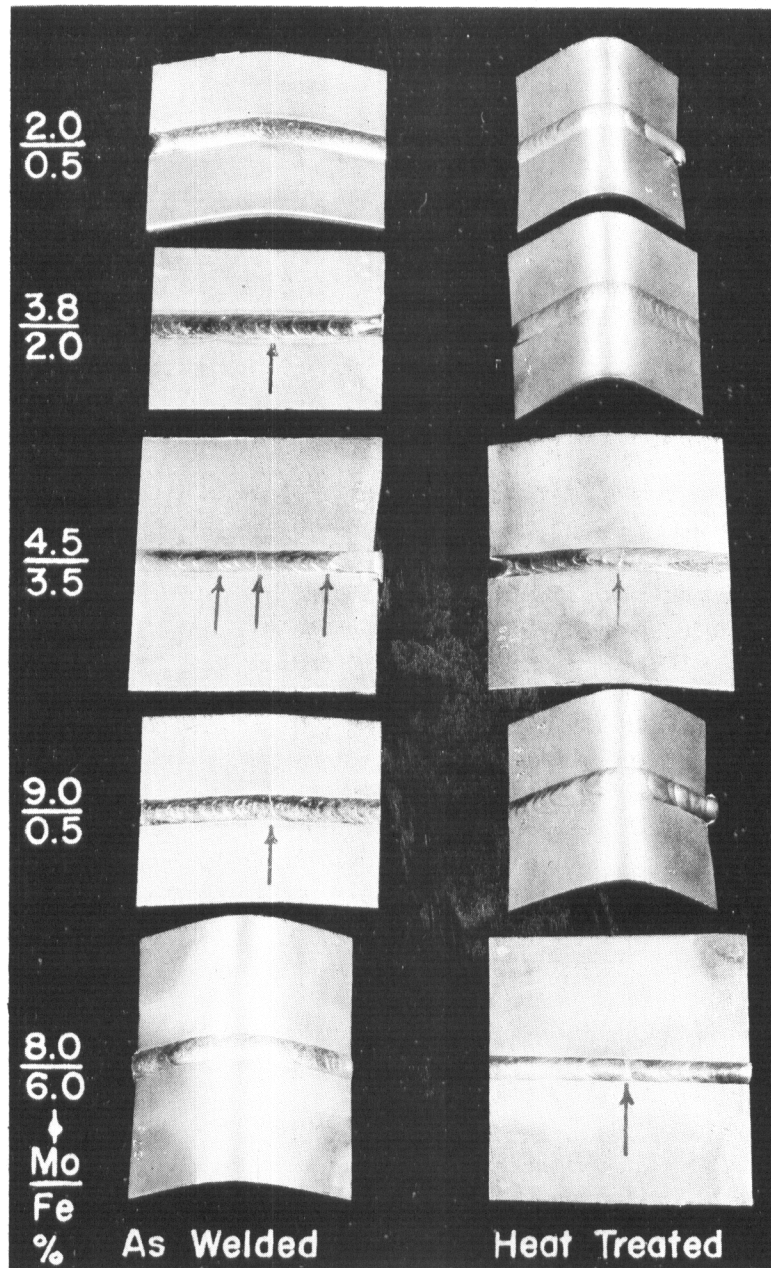
FIGURE 54. PHOTOGRAPH SHOWING EFFECTS OF ADDITIONS OF
MOLYBDENUM AND IRON ON DUCTILITY OF WELDS IN TITANIUM
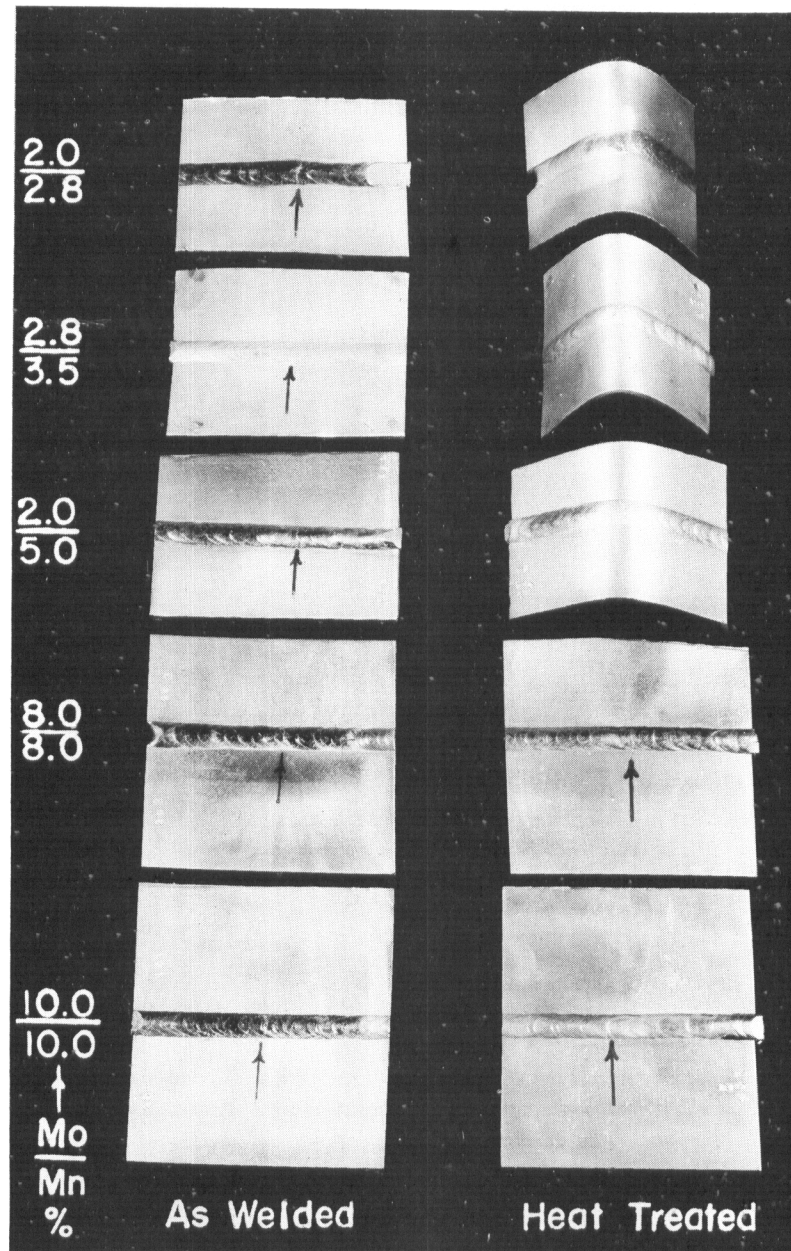
94452

FIGURE 55. PHOTOGRAPH SHOWING EFFECTS OF ADDITIONS OF MOLYBDENUM
AND MANGANESE ON DUCTILITY OF WELDS IN TITANIUM

TABLE 23. PROPERTIES OF WELDED AND UNWELDED TITANIUM ALLOYS

Composition, per cent beta stabilizer	Alloy Type	Ultimate Tensile Strength, Hot-Rolled Sheet, psi	Minimum Bend Radius, Unwelded Hot-Rolled Sheet(1)	Minimum Bend Radius, As-Welded Sheet(2)	Minimum Bend Radius After Postweld Heat Treatment(2,3)
2.75 Mn, 2.0 Mo	a-b	166,300	4T	>24T	6T
3.5 Mn, 2.75 Mo	a-b	180,200	3T	>24T	4T
5.0 Mn, 2.0 Mo	a-b	200,000	4T	>24T	12T
8.0 Mn, 8.0 Mo	b	150,900	1.5T	>24T	>24T
10.0 Mn, 10.0 Mo	b	151,800	1.5T	(4)	>24T
0.5 Fe, 2.0 Mo	a-b	125,300	1.5T	>24T	4T
2.0 Fe, 3.75 Mo	a-b	172,200	3T	>24T	6T
3.5 Fe, 4.5 Mo	a-b	204,000	8T	>24T	>24T
0.5 Fe, 9.0 Mo	a-b	152,400	3T	>24T	6T
6.0 Fe, 8.0 Mo	b	155,900	1.5T	8T	>24T
3.5 V, 1.5 Fe	a-b	146,600	1.5T	24T	6T
2.0 V, 2.0 Mn, 1.0 Fe	a-b	156,300	3T	>24T	3T
5.0 V, 2.0 Mn	a-b	165,700	3T	>24T	6T

(1) Bend in direction of rolling.
(2) Longitudinal bends in direction of rolling.
(3) Alpha-beta heat treatment. Heated to 1350 F for 1/2 hour, furnace cooled at a rate of 3 F per minute to 1150 F, and water quenched to room temperature.
(4) Cracked before testing.

Microstructure and Hardness

Cross sections of all welded specimens in both the as-welded and heat-treated conditions were examined microscopically. In all cases, the weld metal consisted of very coarse, columnar grains. Immediately adjacent to the weld metal was an area of large, equiaxed grains which decreased in size with increasing distance from the weld until they blended into the original base-metal structure. The weld metal showed evidence of alloy segregation in varying degrees in all of the alloys. This segregation was usually more apparent in the heat-treated specimens, due to variations in the amount of alpha precipitated during the heat treatment. A typical over-all view of the weld and adjacent heat-affected zone, as welded, is presented in Figure 56. The appearance of the same specimen after heat treatment is shown in Figure 57. Note the striated structure in the weld metal caused by freezing segregation. The black area in the weld in Figure 57 was a hole, apparently from a gas bubble formed during welding. These holes were observed in several of the welds.

The microstructures of the weld metals differed somewhat in the different types of alloy. In the intermediate-strength, alpha-beta alloys, it consisted of a fine alpha precipitate in a beta matrix, as shown in Figure 58 for the Ti-2Mn-2V-1Fe alloy. The weld metal of the high-strength, alpha-beta and the metastable-beta alloys was essentially all retained beta, as illustrated in Figure 59 for the Ti-5Mn-2Mo alloy. After heat treatment, all of the alloys had a fine alpha precipitate in the beta matrix present in the weld metal. Alloy segregation was particularly noticeable in the heat-treated specimens. It varied considerably for different alloys from no detectable segregation in the Ti-2Mn-2V-1Fe alloy (Figure 60) to the pronounced segregation illustrated for the Ti-5Mn-2Mo alloy in Figure 61. It may be significant that the alloy showing the least evidence of segregation (Ti-2Mn-2V-1Fe) had the highest bend ductility of the group (Table 23).

Hardness surveys were made on the metallographic specimens using a Knoop indenter with a 500-gram load. Impressions were made at 0.5-mm intervals, starting at the center of the weld and extending into the base metal. In the heat-affected zone, where hardness changed rapidly with increasing distance from the weld, shorter intervals were used. The hardness results for alloys representative of the types of alloy used in these experiments are plotted in Figures 62, 63, and 64.

It is evident from the figures that the weld metal and the heat-affected zone adjacent to it in both types of alpha-beta alloy develop relatively high hardnesses in the as-welded condition. As would be expected, the high-strength alloys had somewhat higher hardnesses than the intermediate-strength types. The high hardness was probably the result of hardening of the beta phase during cooling, and was a contributing factor, at least, to the lack of ductility of these alloys as welded.
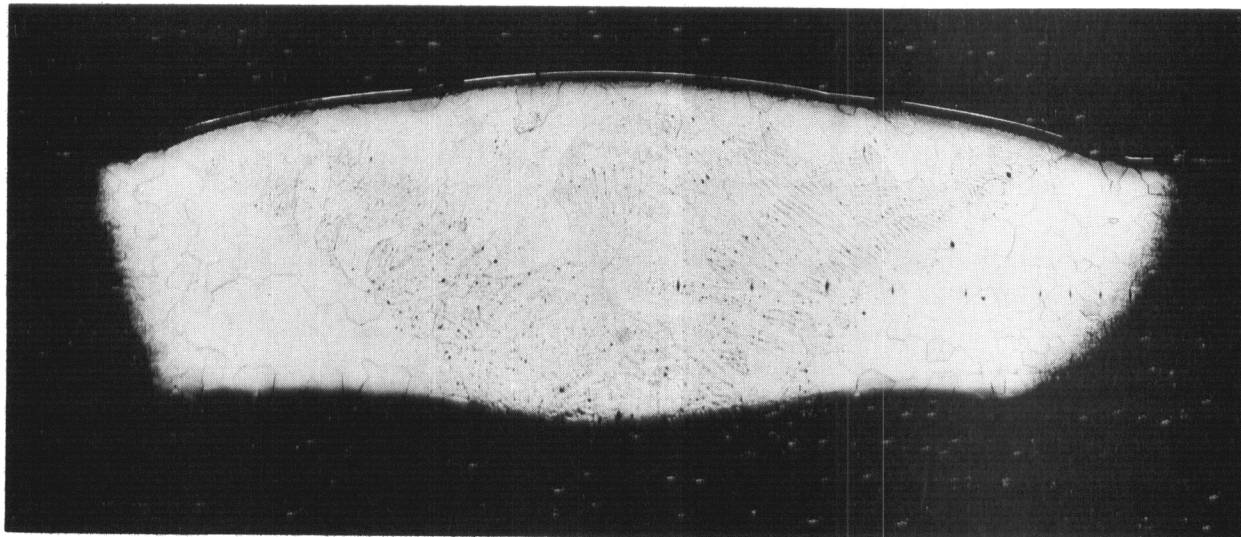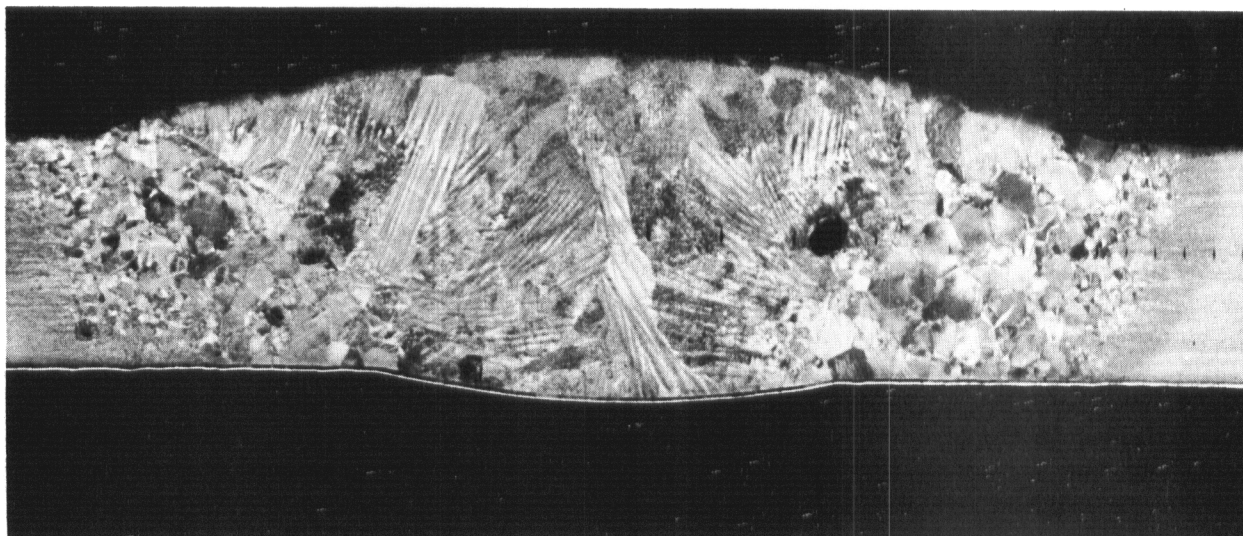
20X   96030

FIGURE 56.   Ti-5Mn-2Mo ALLOY, AS WELDED

Structures of weld-metal and heat-affected zones
are typical of all alloys used in this investigation

20X   96029

FIGURE 57.   Ti-5Mn-2Mo ALLOY, AS WELDED AND HEAT TREATED

Note striated appearance of weld metal from alloy segregation
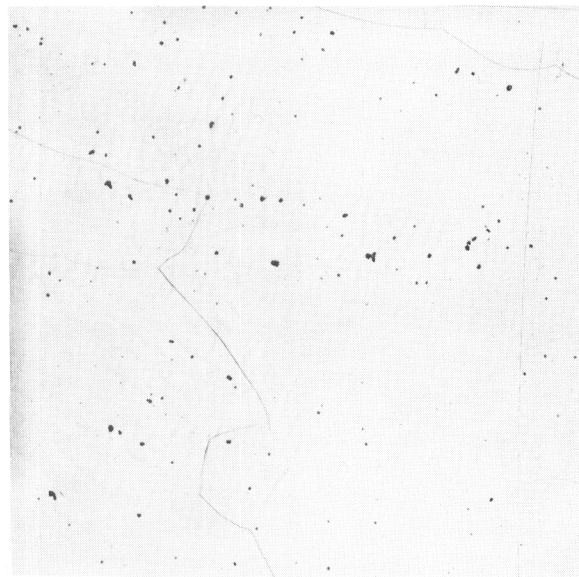
500X   96022

FIGURE 58.   WELD METAL OF Ti-2Mn-2V-1Fe
ALLOY, AS WELDED

Structure: Fine alpha precipitate in beta
matrix; very little freezing segregation

500X   96023

FIGURE 59.   WELD METAL OF Ti-5Mn-2Mo ALLOY,
AS WELDED

Structure: Retained beta; note evidence of strong
freezing segregation.
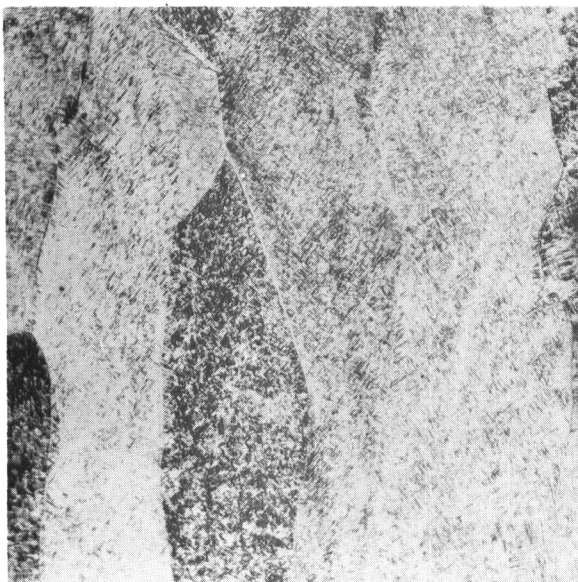
500X   96020

FIGURE 60.   Ti-2Mn-2V-1Fe ALLOY AS WELDED
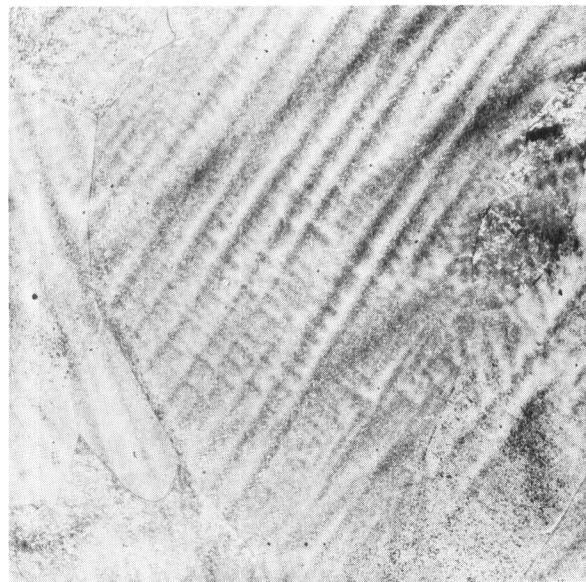AND HEAT TREATED

Structure: Widmanstätten alpha in beta matrix

500X   96018

FIGURE 61.   Ti-5Mn-2Mo ALLOY AS WELDED AND
HEAT TREATED

Structure: Fine alpha precipitate in beta matrix

FIGURE 62. REPRESENTATIVE HARDNESS OF ARC-WELDED AND HEAT-TREATED INTERMEDIATE-STRENGTH TITANIUM ALLOYS OF ALPHA-BETA TYPE
Sheet Thickness— 0.064 inch

A-4071

FIGURE 63. REPRESENTATIVE HARDNESSES OF ARC-WELDED AND HEAT-TREATED INTERMEDIATE-STRENGTH TITANIUM ALLOYS OF DUCTILE METASTABLE-BETA TYPE
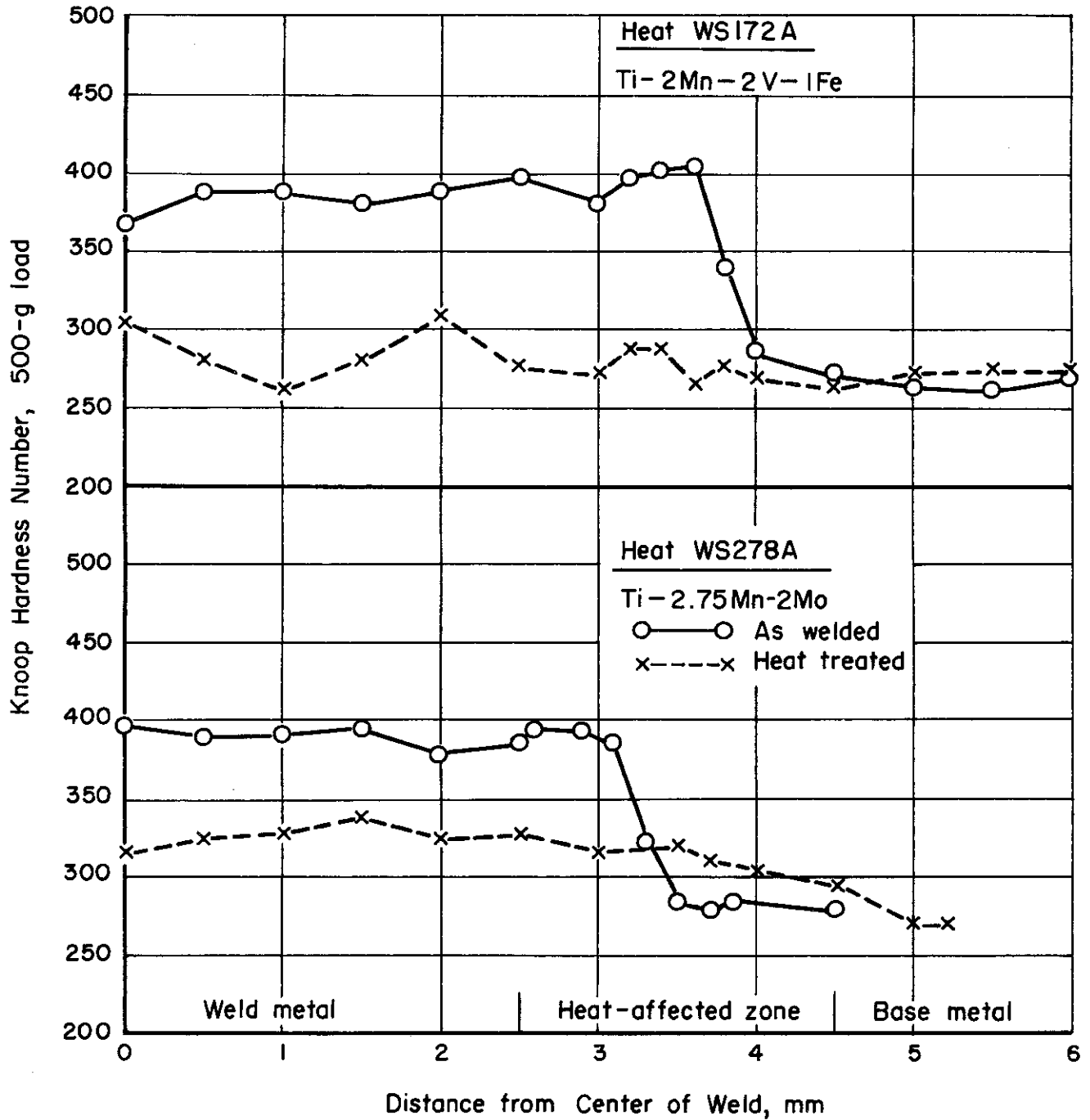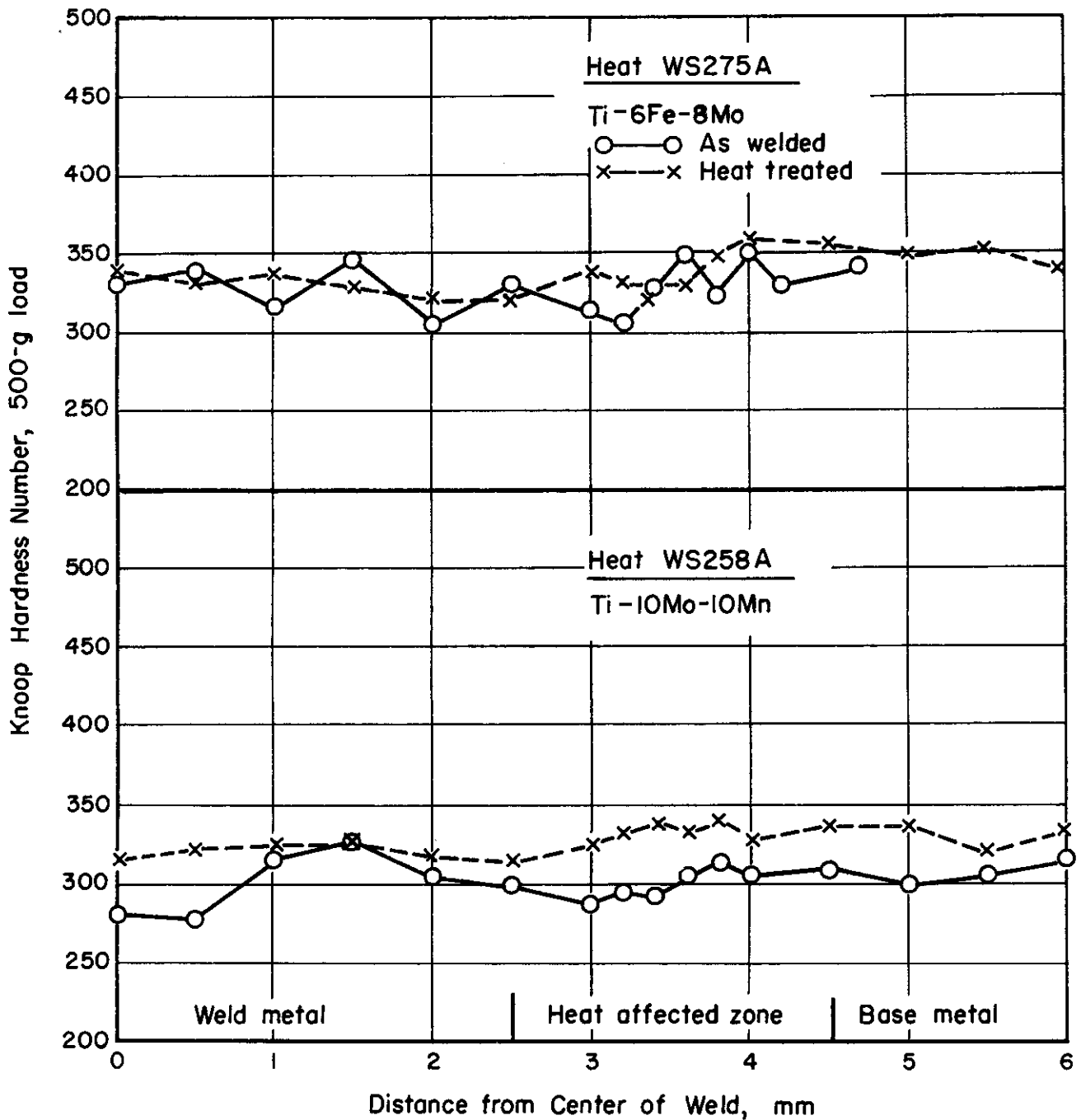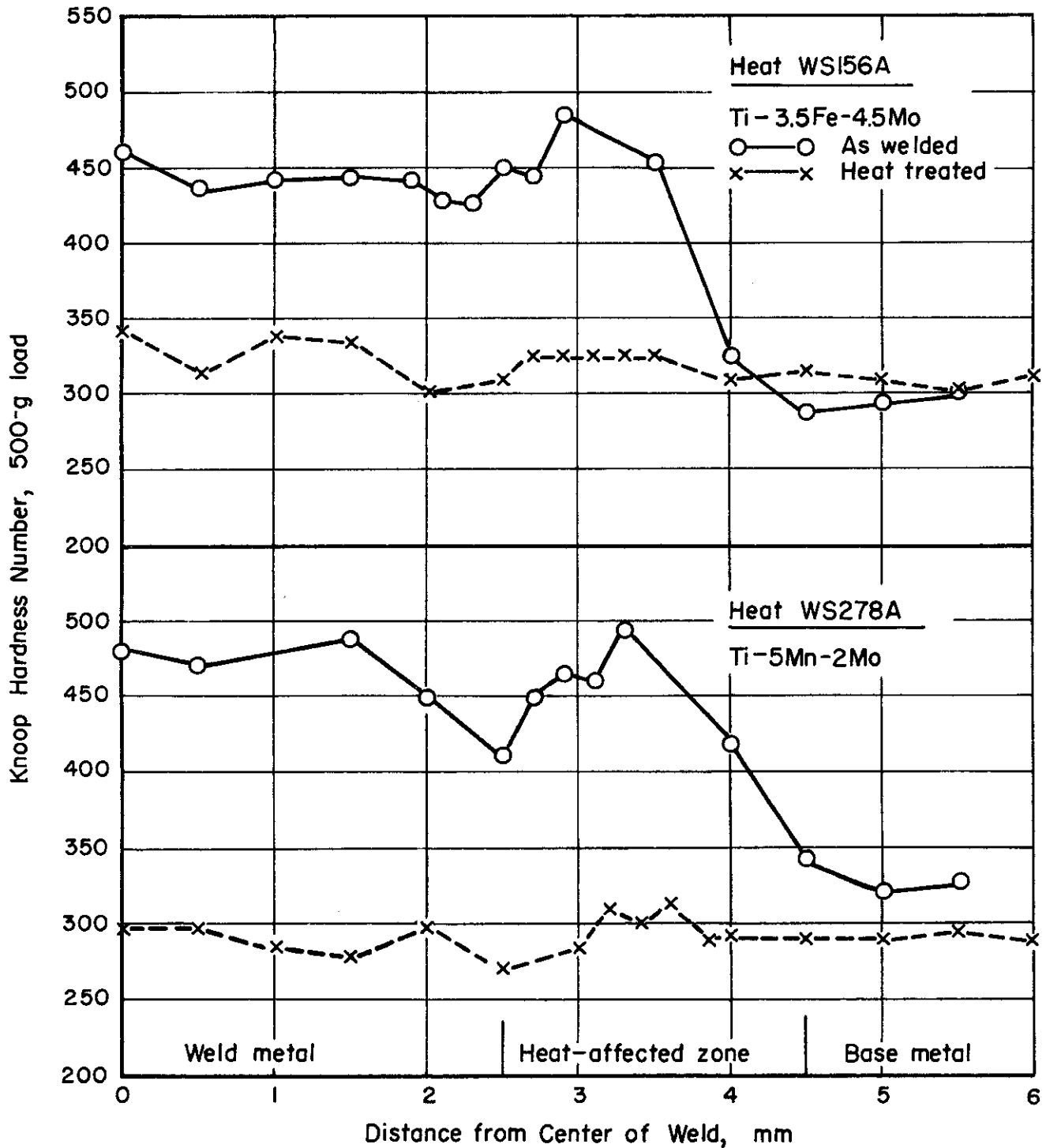Sheet Thickness — 0.064 inch

A-4072

FIGURE 64. REPRESENTATIVE HARDNESSES OF ARC-WELDED AND HEAT-TREATED HIGH-STRENGTH TITANIUM ALLOYS OF ALPHA-BETA TYPE
Sheet Thickness — 0.064 inch

A-4073

Heat treatment reduced the hardness of the weld metal to values only slightly above that of the base metal, in most cases. The ductility was improved also (Table 23), except in the case of the Ti-3.5Fe-4.5Mo alloy. The lack of ductility of this alloy after heat treatment was not commensurate with the hardness of the weld metal (Figure 64), and must be attributed to other factors, possibly segregation in the weld.

In contrast to the alpha-beta alloys, the hardness of the weld metal in the metastable-beta alloys was essentially the same as that of the base metal. Heat treatment, if anything, raised the hardness slightly. The lack of ductility in the heat-treated specimens of these alloys again does not correlate with hardness. A possible explanation of the lack of ductility of these alloys is contained in the discussion of this section.

Discussion

Previous work under this contract has shown that alpha-beta alloys may be hardened and embrittled by cooling from the alpha-beta or beta-phase fields at a suitable rate. This rate varies somewhat with alloy content. All of the alloys used in this study were found to have alloy segregation in their welds. It is probable, therefore, that certain areas in the welds had the critical alloy content for embrittlement both during cooling after welding and during the cooling employed in the heat treatment. These regions might not have sufficient area to raise the over-all hardness of the weld metal, but would serve as loci for initiation of fracture during bending. It is possible that improved heat treatments, such as the overaging treatments described in a preceding section of this report, might "level out" the hardnesses in the segregated areas of the weld and produce improved ductility. This and other types of postweld heat treatment will be tried in the welding studies planned for the new contract.

EXTRUSION OF TITANIUM

Arc Melting of Unalloyed Titanium Ingots

During the period covered by this report, a total of 820 pounds of unalloyed titanium was arc melted in ingot form for preparation into billets to be extruded at Metal Trims, Incorporated, of Youngstown, Ohio. These alloy billets were shipped to Metal Trims on August 7, 1952, and will be used for the initial tests to determine the proper extrusion temperature, pressure, lubricants, and die materials required for titanium.

A series of twenty-eight 20-pound ingots was melted, using extruded melting stock prepared from titanium-sponge fines of less than 6-mesh size. Sponge in this condition was unsuitable for melting on the alloy-development program because (1) it contained a high oxygen and nitrogen content, and (2) it adhered to and alloyed with the electrode when fed into the arc furnace, resulting in tungsten contamination of the ingot. Over a period of four years, approximately 700 pounds of fine sponge had accumulated at Battelle. Since high quality is not essential in the initial extrusion tests, it was decided to salvage this sponge and process it for arc melting. The fines were compacted into briquettes and extruded to bar stock at the Revere Copper and Brass Company of Detroit, Michigan, as described in the Summary Report for the period May 19, 1951, to May 18, 1952. The extruded bars were hot rolled to sheet at 1450 F, grit blasted, and then pickled in a sulfuric-hydrofluoric acid solution to remove the adherent scale, and sheared into chips for melting stock.

Eight 20-pound and two 50-pound ingots were prepared from high-grade Process A titanium sponge. These melts were used, aside from the preparation of extrusion billets, to evaluate the effect of premelting treatments on the sponge, described later in this report.

Originally it was intended to melt all the titanium into 50-pound ingots in the 6-inch-diameter arc furnace. However, it was difficult to obtain good fusion of the ingots and, on forging, deep cracks developed at the poorly fused sections. This would result in a high loss of metal in processing the ingots into extrusion billets. In order to avoid this loss, it was decided to melt the remaining stock into 20-pound heats in the 4-inch-diameter arc furnace in which good fusion of the ingot could be attained.

The ingots were melted in a water-cooled copper crucible, using a thoriated-tungsten electrode and a 99.97 per cent argon atmosphere. The extruded melting stock used to prepare these initial extrusion billets contained an excessive amount of volatile matter which, on melting, caused spattering of the titanium bath to the electrode tip. However, with the exception of ten ingots melted from extruded stock originally of minus 20-mesh sponge and three Process A ingots, the tungsten content due to electrode erosion was below 0.20 weight per cent. Although this amount of contamination is somewhat higher than usual, it should not affect the extrusion tests, since physical properties will not be determined on the initial material.

Fabrication of Ingots Into Extrusion Billets

The two 50-pound ingots were forged at 1700 F by drawing to a 4-3/4-inch round. The 20-pound ingots were forged at 1700 F by upsetting to a minimum 4-3/4-inch round. During forging, Ingots 30 and 51 developed deep cracks and could not be fabricated to round stock. These two ingots will be

TABLE 24.   DATA ON MELTING AND FABRICATION OF
EXTRUSION BILLETS

Billet	Material	Weight, lb	Length, in. [1]	Tungsten [2] per cent	Avg [3] BHN
12-2 [4]	Alloy scrap	15-1/4	6-1/8	No data	341
12-3 [4]	Ditto	14-1/2	5-3/4	Ditto	321
13-1	Process A	16-1/2	6-1/2	> 0.2	185
13-2	Ditto	16-1/2	6-1/2	> 0.2	180
14	Extruded	15	6	> 0.2	331
15	Process A	14	5-5/8	0.1	192
16	Ditto	13-1/2	5-1/2	0.03	165
24	"	15	6	0.17	181
25	Extruded	15	6	> 0.2	336
26	"	14	5-5/8	> 0.2	342
27	Process A	15-1/2	6-1/4	0.14	185
28	Ditto	15-1/2	6-1/4	> 0.2	216
29-1	"	17	6-7/8	0.14	228
29-2	"	17-3/4	7-1/4	0.14	220
30 [5]	Extruded	-	-	0.11	-
31	"	14-1/2	5-3/4	> 0.2	230
32	"	15	6	> 0.2	229
33	"	14-1/4	5-3/4	0.1	171
34	"	14-1/4	5-3/4	0.18	202
35	"	14-3/4	5-7/8	> 0.2	230
36	"	13-1/2	5-1/2	0.11	241
37	"	14-1/2	5-3/4	> 0.2	228
38	"	15	6	0.16	226
39	"	15	6	0.08	212
40	"	15	6	0.14	207
41	"	14-3/4	5-7/8	0.05	226
42	"	14-1/2	5-5/8	0.07	204
43	"	13-3/4	5-1/2	0.1	202
44	"	14-1/2	5-3/4	0.08	209
45	"	14-1/2	5-3/4	0.15	209
46	"	14-1/2	5-3/4	0.08	248
47	"	14-3/4	5-7/8	0.07	266
48	"	14-3/4	5-7/8	> 0.2	255
49	"	15-1/4	6-1/8	0.12	275
50	"	14-3/4	5-7/8	> 0.2	289
51 [5]	"	-	-	> 0.2	-
52	Process A	15	6-1/8	0.18	174
53	Ditto	15	6	0.16	197
54	"	14-3/4	5-7/8	> 0.2	179
55	Extruded	13-3/4	5-1/2	0.1	265

TABLE 24.   (Continued)

Billet	Material	Weight, lb	Length, in. [1]	Tungsten[2] per cent	Avg[3] BHN
56	Extruded	14-1/2	5-3/4	0.09	266
57	Ditto	13-1/4	5-3/8	0.13	281

(1) Length is the only variable dimension; diameter is standard 4-7/16 inches.

(2) Tungsten contamination reported is based on original ingot weight.

(3) Average of two readings on top and bottom of billet using a 3000-kg load.

(4) Ingot 12 was melted from titanium-alloy scrap. Billets 12-2 and 12-3 had unsuitable surfaces and were remelted. They were to be part of the original shipment to Metal Trims.

(5) Cracked during forging. These ingots will be remelted and fabricated to usable billets.

remelted at a later date. The remaining rounds were machined into extrusion billets 4-7/16 inches in diameter by approximately 6 inches long. The forty machined billets weighed a total of 593 pounds; the total wieght of machined billet prepared to date is 750 pounds. Data on the melting and fabrication of the billets are presented in Table 24.

Treatment of Process A Sponge for Arc Melting

Process A titanium sponge in the as-received condition is unsuitable for arc melting because of its high volatile content, mainly magnesium chloride and absorbed moisture. On melting, the volatiles boil out of the bath, causing spattering of the molten titanium to the electrode tip. This titanium alloys with the tungsten and causes erosion of the electrode, resulting in tungsten contamination in the form of small inclusions in the ingot. It has been found necessary, therefore, to treat the sponge for removal of the volatiles prior to melting.

The standard method at Battelle is to leach the sponge in a solvent for removal of the magnesium chloride, followed by a vacuum heat treatment to remove the entrapped solvent and further reduce the volatile content.

The two principal solvents for magnesium chloride are hot water and methanol. Hot-water leaching is the more desirable because of its low comparative cost. However, a series of treatments using a hot-water leach and vacuum treatments at 700 F and 1250 F showed little improvement in the melting characteristics of the sponge when compared with the as-received material. When leached in methanol, followed by vacuum treatment, the sponge exhibited a marked improvement in melting quality. Spatter on the electrode tip was reduced to the point that the tungsten contamination could be held to a low value.

Additional leaching tests were conducted to determine the number of successive leaches and the optimum soaking time required to produce a non-spattering melting stock. The leaching was performed using agitation in several tests to determine if the soaking time could be shortened. The tests made were as follows:

1. One 24-hour still leach.

2. One 72-hour still leach.

3. Five 10-minute agitated leaches.

4. Five 30-minute agitated leaches.

5. One 72-hour still and five 10-minute agitated leaches.

6. Three 24-hour still leaches.

7. Two 72-hour and three 24-hour leaches, each agitated
   10 minutes.

8. Two 24-hour and one 72-hour still leaches.

These treatments will be evaluated by comparing the amount of tungsten contamination when melting the sponge into 20-pound ingots for extrusion purposes.

The use of mechanical vibration, in place of agitation, is being investigated on a 2-pound sample of sponge in order to reduce the amount of fines created during leaching. Another 2-pound sample is being leached in a solution of methanol containing 10 per cent hydrochloric acid, to be follwed by two successive leaches in methanol to determine whether removal of the magnesium chloride is improved by a slightly acidic leach.

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January 5, 1952

APPENDIX

APPENDIX

ARC MELTING OF EXPERIMENTAL TITANIUM-BASE ALLOYS

The melting procedures used with the 2-1/4-inch and 4-inch furnaces are the same as reported in WADC-TR-52-249.

The work completed or in process during the period from May 19, 1952, to December 7, 1952, is described in this phase of the report. Table 25 shows the status of the work.

TABLE 25. INGOTS COMPLETED AND IN PROCESS

Completed

1 - 10 pound - 2.5Cr, 5.0Mn
1 - 10 pound - 5.0Mn, 2.0Mo
1 - 15 pound - 3.5Cr, 3.5V
3 - 20 pound - 3.5Cr, 3.5V
2 - 20 pound - 2.5Cr, 5.0Mn
4 - 20 pound - 1.0Cr, 1.0V, 1.0Fe, 3.0Mn, 1.0Mo
1 - 20 pound - 1.0Cr, 1.0V, 4.0Fe, 1.0Mn, 1.0Mo
1 - 20 pound - 3.5Mn, 3.5Cr

In Process

1 - 10 pound - 2.5Cr, 5.0Mn
1 - 20 pound - 2.5Cr, 5.0Mn
2 - 20 pound - 1.0Cr, 1.0V, 4.0Fe, 1.0Mn, 1.0Mo
2 - 20 pound - 3.5Mn, 3.5Cr
1 - 20 pound - 15.0Cr

New Equipment

A second 4-inch arc-melting furnace has been completed and was put into operation during the last month.

ELECTROPOLISHING OF TITANIUM
AND TITANIUM ALLOYS

The mechanical polishing of titanium is a laborious and time-consuming operation, due to the formation of a disturbed layer on the surface of the specimen during preliminary grinding operations. A number of etching and repolishing operations are necessary to remove this disturbed layer. Previous attempts to obviate this difficulty by electrolytic polishing all involved the use of electrolytes containing perchloric acid. Such electrolytes would not be suitable for the work under this contract, since the majority of specimens were of thin (0.064-inch) sheet which required mounting in organic resins for examination. It is well known that perchloric acid in contact with organic materials may form an explosive mixture. Consequently, the use of electrolytic polishing on this contract required the development of a non-explosive electrolyte.

At a symposium on "Surface Treatment of Titanium", held at Watertown Arsenal recently, an electrolyte developed at Armour Research Foundation for zinc plating of titanium was described. It was stated that this electrolyte, consisting basically of a 6 to 10 per cent solution of HF in ethylene glycol, appeared to have some polishing action on titanium. Attempts to polish metallographic specimens in this solution were unsuccessful. However, the addition of about 5 per cent concentrated H_2SO_4 to the Armour solution resulted in reasonably good polishing action on several specimens of titanium alloys. Some staining and etching still occurred during the polishing operation in this electrolyte. On the assumption that the water introduced into the solution with the HF was responsible for the staining and etching effects, an anhydrous solution was prepared by dissolving BaF_2 in the ethylene glycol and adding an excess of H_2SO_4. HF was formed by the reaction

$$H_2SO_4 + BaF_2 \longrightarrow 2HF + BaSO_4 \; .$$
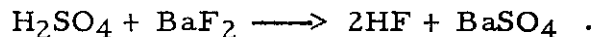
Excellent results were obtained with this electrolyte, using a 45-volt "B" battery as a power source and a stainless steel cathode. Considerable heating occurred during polishing in this electrolyte. This heating was undesirable in view of the information obtained on the low-temperature aging of titanium alloys (Seventeenth Progress Report, dated March 18, 1952). Substitution of glycerine for the ethylene glycol reduced the heating considerably and produced an excellent polish, but applied voltages of 70 to 90 volts were necessary. The composition of the final electrolyte was as follows:

<div style="text-align:center">

80 cc glycerine
5 grams BaF_2
5 cc concentrated H_2SO_4

</div>

The specimens to be polished were wet ground through 600-grit silicon carbide paper according to standard procedures. They were then polished by immersion in the electrolyte for 1-1/2 to 2 minutes. Electrical contact was made with the specimen by drilling through the side of the Bakelite mount, tapping, and inserting a brass screw. If a number of specimens were to be polished in the same mount, electrical contact was made between specimens by laying copper wires across their backs before mounting. This is illustrated schematically in Figure 65. Mild agitation of the specimen seemed to improve the quality of the polish.

A circuit diagram for the electrolytic cell is shown in Figure 66. A versatile d-c power source was provided for the cell by hooking a selenium oxide, power-type rectifier to the output of a 5-ampere Variac autotransformer, which, in turn, was connected to the 110-volt outlet. By this means, applied voltage to the cell could be varied from 0 to 100 volts, and currents up to 6 amperes were possible.

Representative microstructures of four alloy specimens having widely differing microstructures, electropolished in the BaF_2 - H_2SO_4 solution and chemically etched, are shown in Figures 67 to 72. An applied voltage of 90 volts was used and the polishing time was 1-1/2 minutes. Current density was about 2 amp/sq in.

The BaF_2 - H_2SO_4 electrolyte was found to have two major disadvantages:

1. Some heating still occurred during polishing.

2. Of a number of specimens in a single mount, some specimens would be polished, while others remained virtually unaffected.

After this electrolyte had been in use for a short time, information was obtained on an electrolyte developed by the Rem-Cru Titanium Corporation consisting of the following:

> 90 cc ethyl alcohol (absolute)
> 10 cc n-butyl alcohol
> 6 grams $AlCl_3$
> 25 grams $ZnCl_2$

The salts are dissolved in the alcohol mixture in the order given. Care must be taken to cool the solution during mixing of the salts, since considerable heat is evolved.
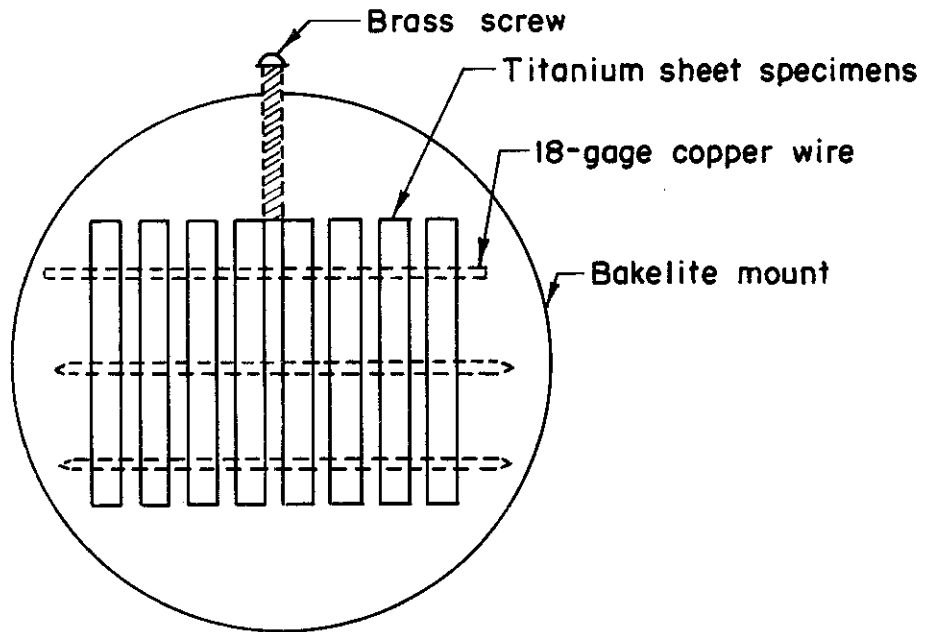
FIGURE 65. SCHEMATIC REPRESENTATION OF INTERNAL AND EXTERNAL CONNECTIONS FOR ELECTROPOLISHING SHEET SPECIMENS OF TITANIUM ALLOYS
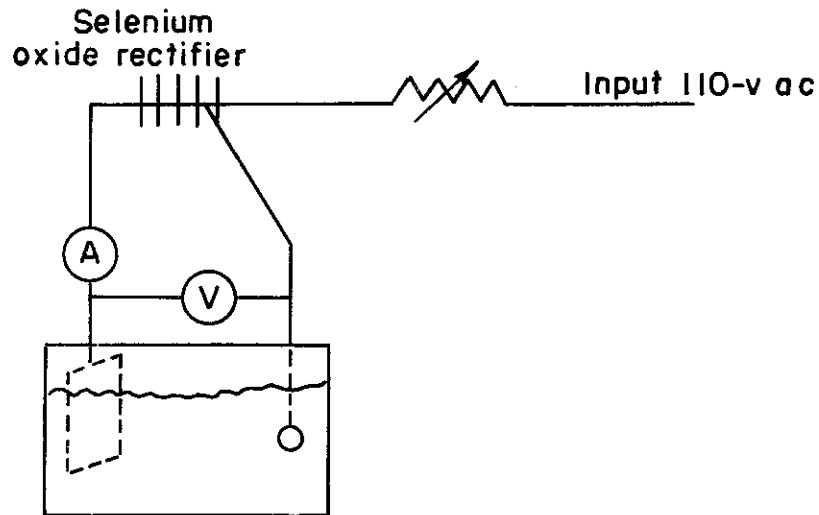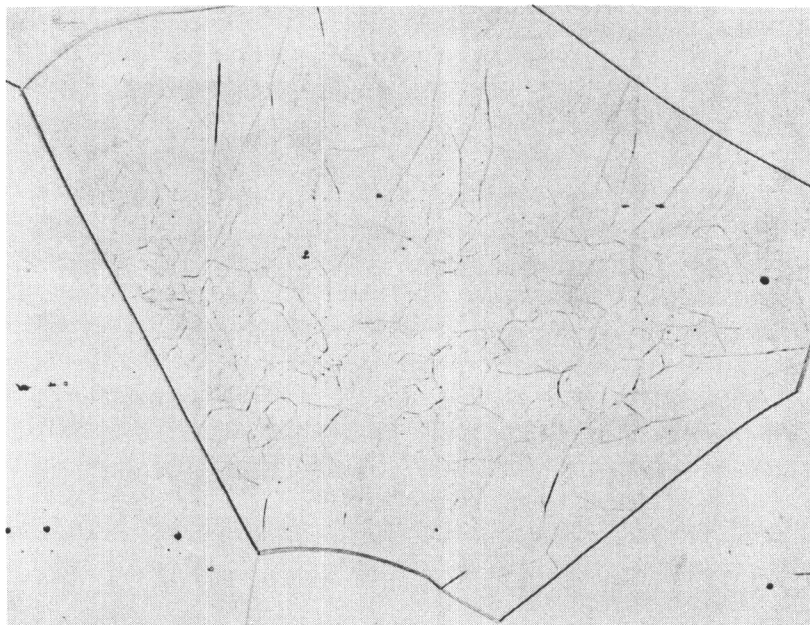

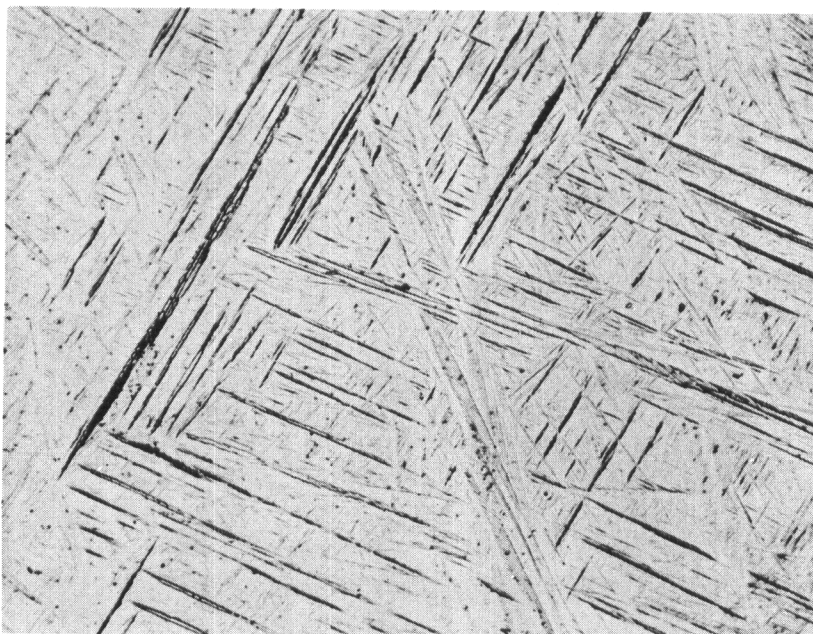FIGURE 66. SCHEMATIC REPRESENTATION OF CIRCUIT FOR ELECTROPOLISHING TITANIUM ALLOYS

A-4074

250X   92645

FIGURE 67.   Ti-7.5Cr ALLOY HEATED AT 1742 F (950 C) FOR 1/2 HOUR
              AND ICE WATER QUENCHED

Electropolished in the BaF_2 - H_2SO_4-glycerine electrolyte and etched in 1% HNO_3-
1% HF-1% $NaNO_2$ in water. Note subboundary structure in retained-beta grain.

500X   92643

FIGURE 68.   Ti-4.14Cr ALLOY HEATED AT 1742 F (950 C) FOR 1/2 HOUR
              AND ICE WATER QUENCHED

Polished and etched as in Figure 177; alpha prime structure.
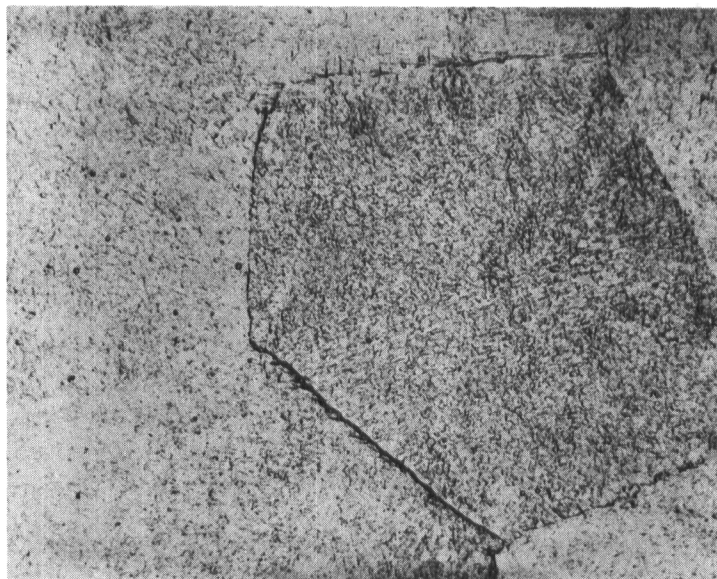
500X   92644

FIGURE 69. Ti-7.54Cr ALLOY ISOTHERMALLY TRANSFORMED FOR 72
HOURS AT 1202 F (650 C)

Electropolished as described in Figure 67 and stain etched in 2% HF - water solution

Structure: Alpha particles in retained-beta matrix
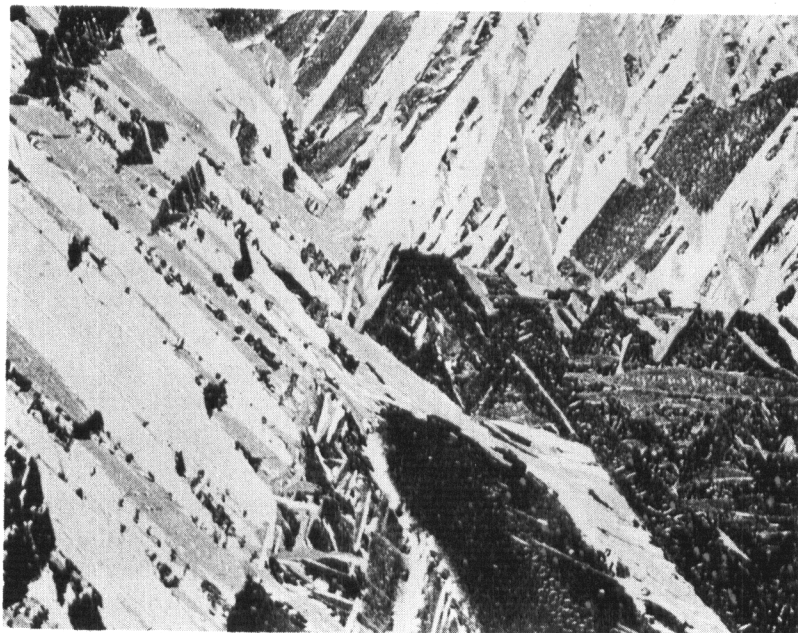

500X   92646

FIGURE 70. COMPLEX Ti-1Cr-4Fe-1Mn-1Mo-1V ALLOY AS HOT ROLLED
AT 1600 F

Electropolished and etched as in Figure 67

Structure: fine alpha precipitate in beta matrix.
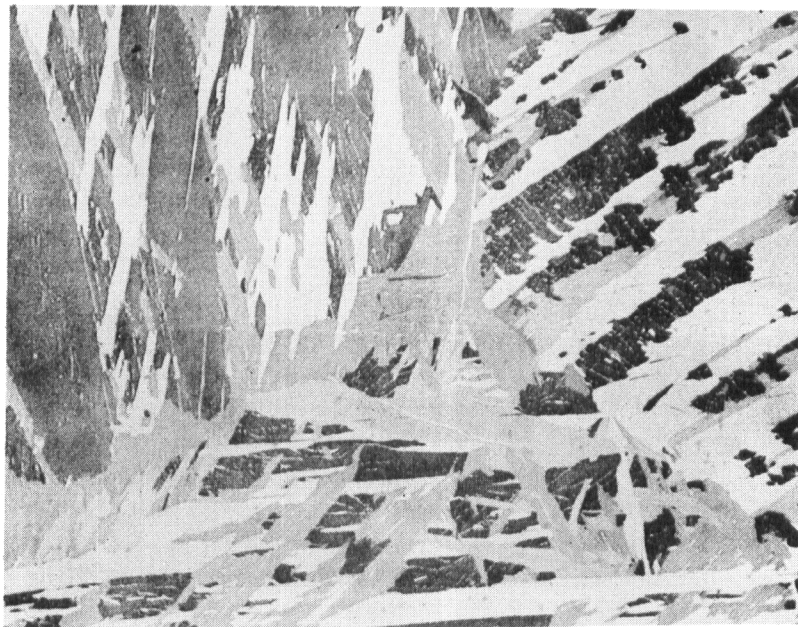
500X   93432

FIGURE 71.   UNALLOYED PROCESS A TITANIUM WATER QUENCHED FROM
1700 F

Electropolished using Rem-Cru electrolyte and etched in 2% HF - water solution

500X   93431

FIGURE 72.   UNALLOYED PROCESS A TITANIUM WATER QUENCHED
FROM 1700 F

Electropolished using Rem-Cru electrolyte and etched in 2% HF - water solution

This solution has been used to polish a large number of specimens with very satisfactory results. Best results were obtained at an applied voltage of 30 volts and a current density of about 0.4 amp/sq in. Polishing time was 3 to 6 minutes.

The microstructures of two unalloyed Process "A" titanium specimens, water quenched from 1700 F and electropolished using this electrolyte, are shown in Figures 71 and 72.

The Rem-Cru electrolyte has two distinct advantages over the glycerine-base electrolyte developed on this project. Heating effects are nil in the former, and all specimens in a multiple-specimen mount are satisfactorily polished. The quality of the polish produced by the glycerine-base electrolyte is slightly better, but this factor is outweighed by the other two factors.

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(2) Guinier, A., Compt. rend., Vol 204, 1937, p 1115, and Vol 206, 1938, p 1641; Nature, Vol 142, 1938, p 569.

(3) Preston, G. D., "The Diffraction of X-Rays by Age Hardening Aluminum-Copper Alloys", Proc. Roy. Soc., A., Vol 167, 1938, pp 526-538.

(4) Schwartz, C. M., Inst. of Metals Symposium on "Metallurgical Applications of the Electron Microscope", 1949, p 85 (Monograph and Report Series No. 8).

(5) Geisler, A. H., "Precipitation From Solid Solutions of Metals", Symposium on Phase Transformations in Solids, John Wiley and Sons, New York, 1951, p 496.

Data from which this report has been prepared are contained in BMI Laboratory Notebooks Nos. 6722, 6741, and 7585.

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