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THE FABRICATION OF ARC-MELTED INGOTS OF TITANIUM AND TITANIUM-MANGANESE ALLOYS INTO PLATE

BY R. W. HUBER, V. C. PETERSEN, AND R. C. WILEY

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UNITED STATES DEPARTMENT OF THE INTERIOR
Douglas McKay, Secretary
BUREAU OF MINES
J. J. Forbes, Director

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THE FABRICATION OF ARC-MELTED INGOTS OF TITANIUM AND TITANIUM-MANGANESE ALLOYS INTO PLATE

by

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SUMMARY

Three arc-melted ingots, two of a titanium-7 percent manganese alloy and the third of unalloyed titanium, were forged and rolled into 3/4-inch plate; comparative data were obtained on these processes. The finished plate, part of which was used by the Naval Bureau of Ordnance, was tested for mechanical and other properties. These properties were correlated with sponge purity, forging technique, rolling temperature, and cooling rate from the rolling temperature. For the alloy material, transformation data were obtained, and their age-hardening characteristics were studied. The temperature-impact relationship was established for rolled sections from all three ingots.

INTRODUCTION

Arc-melted ingots of unalloyed titanium and a titanium-manganese alloy were fabricated into plate under closely controlled conditions by personnel of the Metals and Alloys Section, Physical Metallurgy Branch, Bureau of Mines, College Park, Md. This program was cosponsored by the Naval Bureau of Ordnance. Bureau of Mines interest lay in correlating the processing procedures with the mechanical properties of the finished plate.

Considerable data have been published recently on constitution diagrams and properties of titanium-manganese alloys in general.3/4/5/6/ However, little if any correlation between processing schedules and end results has been published, particularly with respect to the present commercial production of the proprietary titanium-manganese alloys.

Several factors in the production of titanium and titanium alloys have a pronounced influence on the physical and mechanical properties of the finished product: (1) The degree and nature of impurities in the base materials, (2) the amount of impurities introduced during the melting cycle and their distribution, (3) the homogeneity of the alloy, (4) forging, (5) rolling, and (6) final heat-treating cycles. All of these items are important from a metallurgical standpoint in producing the desired end properties in the metal.

This report describes in detail the various steps in fabricating the materials into finished plate, presents comparative mechanical tests, and reviews the response of the titanium-manganese alloy to various heat treatments. An attempt is made to correlate all factors with the tests on the finished plate.

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ACKNOWLEDGMENTS

The authors wish to acknowledge the assistance given them by the personnel of the Republic Steel Co., Canton, Ohio, who melted the ingots; also to the personnel of the Naval Gun Factory, particularly Harold Bernstein, under whose direction the ingots were forged.

MATERIALS

For this work 3 ingots, each weighing about 300 pounds, were prepared by arc-melting selected titanium sponge. The control ingot, unalloyed titanium, was manufactured from sponge of 125-130 Brinell hardness produced by the Bureau of Mines plant in Boulder City, Nev. The composition of the other 2 ingots was titanium-7 percent manganese, which is a well-known and widely used commercial alloy. The 2 alloy ingots varied in the quality of the sponge used to produce them, 1 having been manufactured from low-hardness (110-115 Brinell) and the other from medium-hardness (140 Brinell) sponge. In this way, not only could the manganese alloy itself be studied but also the effect of sponge purity, which plays a large role in all titanium metallurgy. The sponge for the low-hardness alloy ingot was produced by the Bureau of Mines, and that for the medium-hardness ingot came from the regular production of the du Pont de Nemours Co., Newport, Del. Electrolytic manganese was used for alloy additions.

MANUFACTURING OPERATIONS

Melting

The three ingots were arc-melted at the titanium plant of the Republic Steel Co. in Canton, Ohio, by Republic Steel Co. personnel in a water-cooled copper crucible 12 inches in diameter. In height the ingots produced ranged from 16 to 18 inches and in weight from 280 to 340 pounds. The ingots were melted under an inert-gas atmosphere by direct-current arc from a water-cooled, thoriated tungsten electrode 1 inch in diameter. The electrode was fixed to the centrally located shaft at an angle so as to describe a circular path 6 inches in diameter during melting. The electrode rotation was regulated in accordance with the buildup in the melt, and the rate of electrode retraction was governed by an automatic arc-voltage control.

Before evacuation approximately 10 pounds of sponge was placed in the bottom of the crucible for arc initiation. The system was closed and evacuated to a pressure of about 100 microns, and then backfilled to atmospheric pressure with an atmosphere containing 80 percent helium and 20 percent argon. A continuous flow of this gaseous mixture was maintained throughout the melting cycle. After backfilling, the arc was initiated on the loose sponge (referred to as the heel of the ingot), which was melted before the feed mechanism was started.

All sponge (sized to -1/2, + 1/8 inch) was vacuum-dried for 6 to 12 hours, then loaded into feed bins, which supplied the vibratory feed mechanism. For the alloy ingots, the manganese was also added to the feed mechanism, which fed both materials in the proper proportion to the crucible during the melting process. The rate of feed for the first hour of melting was set at 20 to 40 pounds of material per hour. After 50 pounds had been melted, the rate of feed was increased periodically until it had reached the maximum rate of 70 to 80 pounds of material per hour.

The total elapsed time for melting ranged from 6 to 8 hours, depending upon the difficulties encountered during the operation. During melting, some electrode was

lost by spatter from the melt, which alloyed with and washed away the electrode. As a check on this weight loss, the electrode was weighed before and after melting.

The operating current used, once the melt was underway, ranged from 2,500 to 2,700 amperes, with a potential of approximately 60 volts. To supply the power, 10 d.c. generators were used. These were normally operated at 60 percent of their rated capacity.

Although the thoriated tungsten electrode is preferred rather than the carbon electrode, because of carbon pickup in the melt, use of a thoriated-tungsten electrode requires more experience on the part of the melter, as the arc is more difficult to control. The unalloyed ingot had considerable sponge buildup along the side wall due to the inexperience of the melter in operating with a thoriated-tungsten electrode. This left a very porous, incompletely melted surface on the ingot wall, which resulted in a greater loss in machining to provide a surface suitable for forging.

The top of the arc-melting furnace was equipped with two 5-1/2- by 3/4-inch Pyrex sight glasses. This enabled the melter to observe closely the progress of the melt and maintain control of the arc at all times and if necessary to request a slower or faster rate of feed. The sight glasses were equipped on the inner surface with a wiper, which removed the black, sooty substance that was continuously deposited. Temperature indicators on the control board showed the temperature of the cooling water circulated around the crucible, the top jacket, and the bottom cover plate.

After melting, the ingot was allowed to cool 30 to 45 minutes before removal from the copper crucible. It was not difficult to strip the ingots because of ingot shrinkage and lack of a wetting action or adhesion between molten titanium and copper.

Surface Preparation of the Ingots Before Forging

After having been weighed in the as-cast condition, the ingots were skinned to a depth of approximately 1/8 inch to remove the somewhat irregular crust on the ingot wall and the heels of the ingots were removed. Machining the ingot surface eliminates any defects in the skin and helps to insure a good surface free of cracks in the forged product. Some irregularities on the sides of the commercially pure titanium ingot were filled in by heli-arc welding. All sharp corners were given a radius by grinding.

The ingot designation was as follows:

Ingot No.	Type	Sponge hardness,1/ BHN	Weight, lb. (as melted)	Finished diameter (inches)	Finished height (in.)
1	Ti-7 percent Mn	140	280	11.0	15.0
2	Ti-7 percent Mn	110-115	300	11.5	18.0
3	Unalloyed Ti	125-130	340	11.0	16.5

1/ Determined on buttons arc melted from representative sponge samples.

Forging

The ingots were forged at the U. S. Naval Gun Factory by personnel of the gun factory.

The ingots were preheated in an electric furnace for approximately 6 hours at 650° C. (1,200° F.) and then brought up to 980° C. (1,800° F.) in 3 hours. They were then upset approximately 25 percent in an 800-ton hydraulic press and pressed laterally into an octagonal section with a distance across the flats of approximately 7 inches. The ingots were reheated when deemed necessary. Figures 1 and 2 are photographs, showing, respectively, the upsetting and lateral working of the ingots.

After this preliminary forging, the billets were halved transversely, one half to be finished by press forging (on the same press as above) and the other half to be finished by hammer forging in a 6,000-pound steam hammer. Thus, forging conditions could be compared. For finish forging the billets were heated to 980° C. (1,800° F.) (time to heat - about 8 hr.) and both the hammer- and press-forged billets were taken down to slabs 2-1/2 inches thick and 8 inches wide. The length of the slabs thus produced ranged from 27 to 34 inches. All surface end bursts encountered in the initial stages of forging were ground out before forging was continued.

All the forged billets were radiographed to locate areas containing tungsten segregation caused by losses from the arc-melting electrode.

Rolling

Forged billets were rolled into plate by Bureau of Mines personnel at College Park, Md.

The forged billets were cut into sections 6 inches long, providing a number of pieces 6 by 8 by 2-1/2 inches for rolling into 3/4-inch plate. The sections were so cut as to avoid, as far as possible, the tungsten segregations revealed by radiography. They were cut with a hydraulic-feed, liquid-cooled power saw equipped with high-speed steel blades. It took 1-1/2 hours to saw 1 section of an alloy billet; this compared favorably to a 1-hour cutting time for a section of the unalloyed ingot.

Two of the cut sections were selected from each forged billet for rolling into 3/4-inch plate; sections most free from tungsten segregation were chosen. Table 1 shows the identification of these sections and the rolling temperatures employed. It was felt that the greatest amount of information would be obtained if 2 rolling temperatures were employed, 1 above the alpha-beta transformation and the other below. For an alloy containing 7 percent manganese, the transformation temperature occurs at about 800° C. (1,470° F.); rolling temperatures of 850° C. (in the all-beta field) and 750° C. (in the alpha-beta field) were therefore selected for the manganese-alloy sections. Temperatures of over 850° C. were not employed because excessive beta grain growth might occur, with consequent loss of ductility in the finished plate. For the unalloyed material, the transformation temperature is in the vicinity of 900° C. (1,650° F.), so rolling temperatures of 950° and 850° C. were chosen for this material.

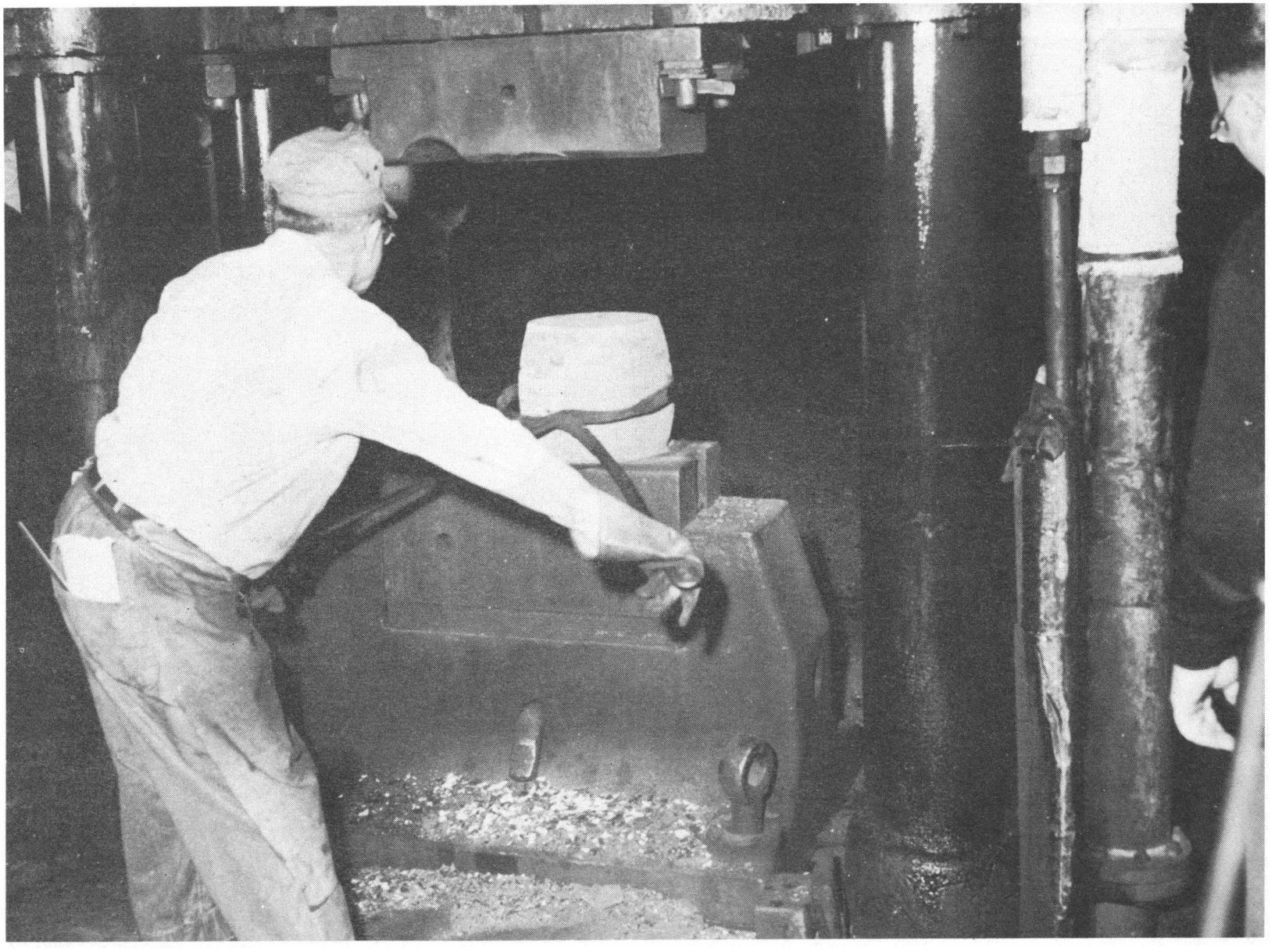


Figure 1. - Upsetting a titanium-alloy ingot.

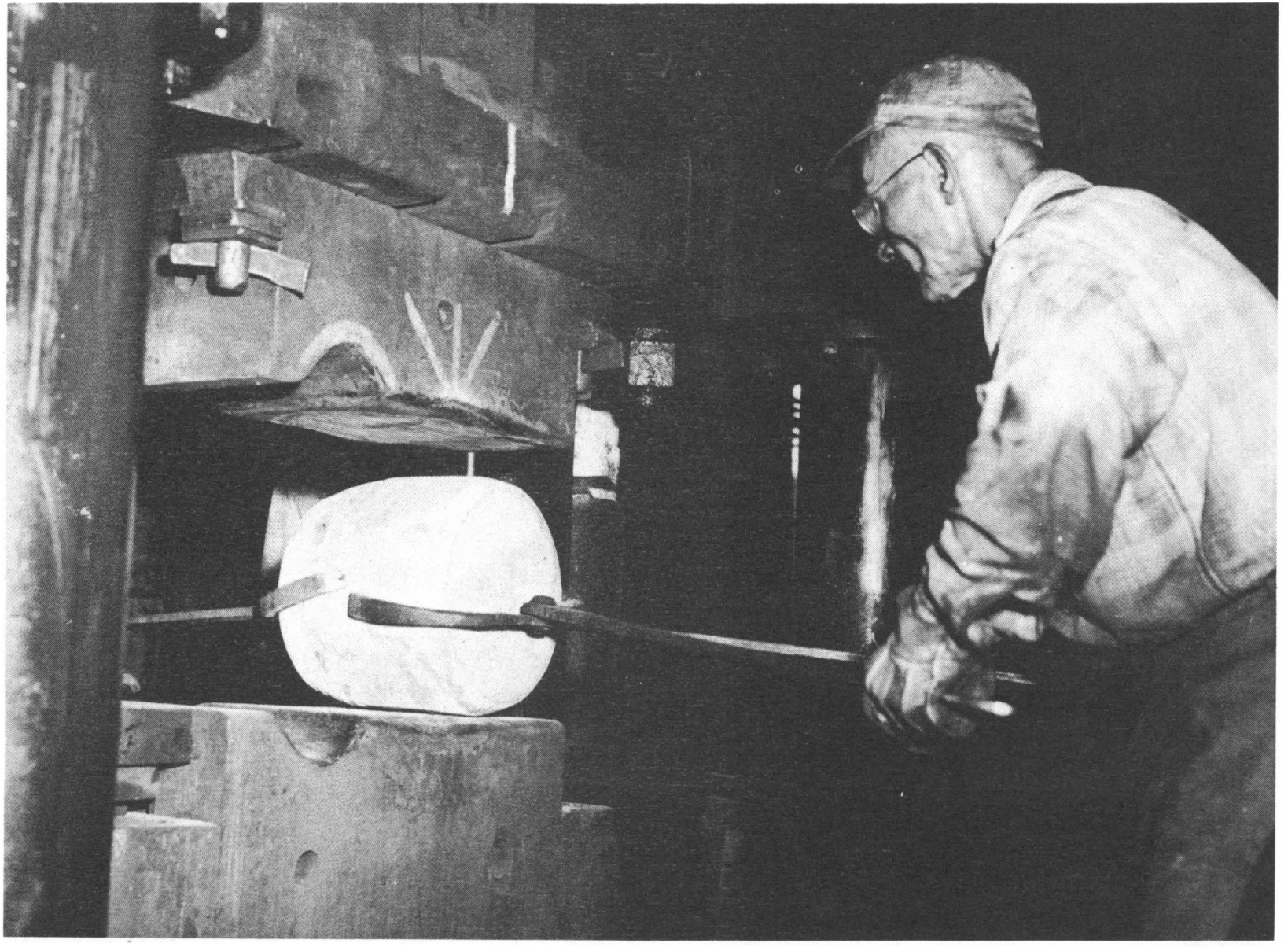


Figure 2. - Forging a titanium-alloy ingot.

TABLE 1. - Identification of sections and rolling temperatures

Section designation	From ingot	Material	Method of forging	Rolling temperature °C.	Rolling temperature °F.
H1A	1	Mn alloy - medium - hardness sponge	Hammer	850	1,560
H1B	1	do.	do.	750	1,380
P1A	1	do.	Press	850	1,560
P1B	1	do.	do.	750	1,380
H2A	2	Mn alloy - low - hardness sponge	Hammer	850	1,560
H2B	2	do.	do.	750	1,380
P2A	2	do.	Press	850	1,560
P2B	2	do.	do.	750	1,380
H3A	3	Unalloyed titanium	Hammer	950	1,740
H3B	3	do.	do.	850	1,560
P3A	3	do.	Press	950	1,740
P3B	3	do.	do.	850	1,560

The forged surface was not removed from the sections before rolling. All sections were preheated for 1 hour at 1,200° F. and then held at the desired rolling temperature for 1/2 hour before rolling commenced. Electric resistance furnaces were used for heating.

Manganese alloy sections heated to 850° C. (H1A, P1A, H2A, P2A) were rolled from 2-1/2 to 1-1/8 inches in thickness, with reductions of 0.100 inch per pass; they were reheated every other pass. From a thickness of 1-1/8 inches down to the finished thickness of 3/4 inch, only 1 pass of 0.100 inch was taken between reheats.

Manganese-alloy sections heated to 750° C. (H1B, P1B, H2B, P2B) were similarly rolled, except that the reduction per pass was 0.050 inch instead of 0.100 inch. These small reductions were necessary to prevent excessive roll pressures with subsequent stalling of the mill or roll breakage.

Sections of unalloyed titanium heated to 950° C. (H3A, P3A) were reduced 0.200 inch per pass down to a thickness of 1-1/2 inches, with a reheat after each pass. From 1-1/2 inches to 3/4 inch the reductions were limited to 0.120 inch per pass, again with a reheat after each pass.

Sections of unalloyed titanium heated to 850° C. (H3B, P3B) were reduced 0.120 inch per pass for the entire rolling cycle down to 3/4 inch thickness, with a reheat after each pass.

After the last rolling pass each section was reheated to its proper rolling temperature and then cooled in the furnace by cutting off the power. Some variation undoubtedly existed among the cooling cycles; however, the rate of cooling was slow enough to minimize differences between sections.

The final size of the rolled sections was 3/4 by 6-1/2 by 27 inches. These were cut into pieces 8 inches long; several of these were milled to remove 0.060 inch from the rolled surfaces and thus provide pices 5/8 by 6 by 8 inches for work not covered in this report. It was deemed necessary to remove stock to a depth of 0.060 inch, as ascertained by metallographic and microhardness surveys, to eliminate surface irregularities and contamination incurred in forging and rolling.

The remaining 3/4-inch material (from the center section of the rolled plate) was reserved for mechanical tests and heat-treating experiments.

One section of the press-forged No. 2 billet (low sponge hardness) was reserved for rolling at 650° C. (1,200° F.); it was felt that this low temperature might produce metal of good mechanical properties. The 5-1/2-inch section was held 1 hour at 650° C. before rolling was begun. Because of the low working temperature involved, reductions were limited to 0.020 inch per pass, with reheating after each pass. Final thickness was 0.520 inch. This section was identified as P2D.

Experiments were also conducted on the effect of water quenching from various rolling temperatures to compare resulting mechanical properties with those of the slow-cooled material. For these experiments, specimens measuring 1-1/2 by 2 by 3 inches were cut from billet H1 (hammer-forged - medium sponge hardness) and from billet P2 (press-forged - low sponge hardness). X-ray diffraction and metallographic studies (see sections relating to these) had established the transus temperatures of billet H1 as about 785° C. (1,445° F.), and of billet P2 as about 740° C. (1,365° F.). Specimens from billet H1 were rolled at 3 temperatures below the transus, 730°, 745°, and 760° C., and 3 temperatures above the transus, 815°, 835°, and 845° C. Due to paucity of material, only 3 rolling temperatures were used for the specimens from billet P2 - these were 730°, 745°, and 760° C. - the first being below, the second slightly above, and the last well above the transus.

The sections were rolled from 1-1/4 to 1/2 inch thick; reductions were 0.050 inch per pass, with 1 pass per reheat. When the finish dimension (1/2 inch thick) was reached, the specimens were returned to the furnace, held 1/2 hour at the rolling temperature, and water-quenched. Direction of rolling was normal to forging direction.

TESTING PROCEDURE AND RESULTS

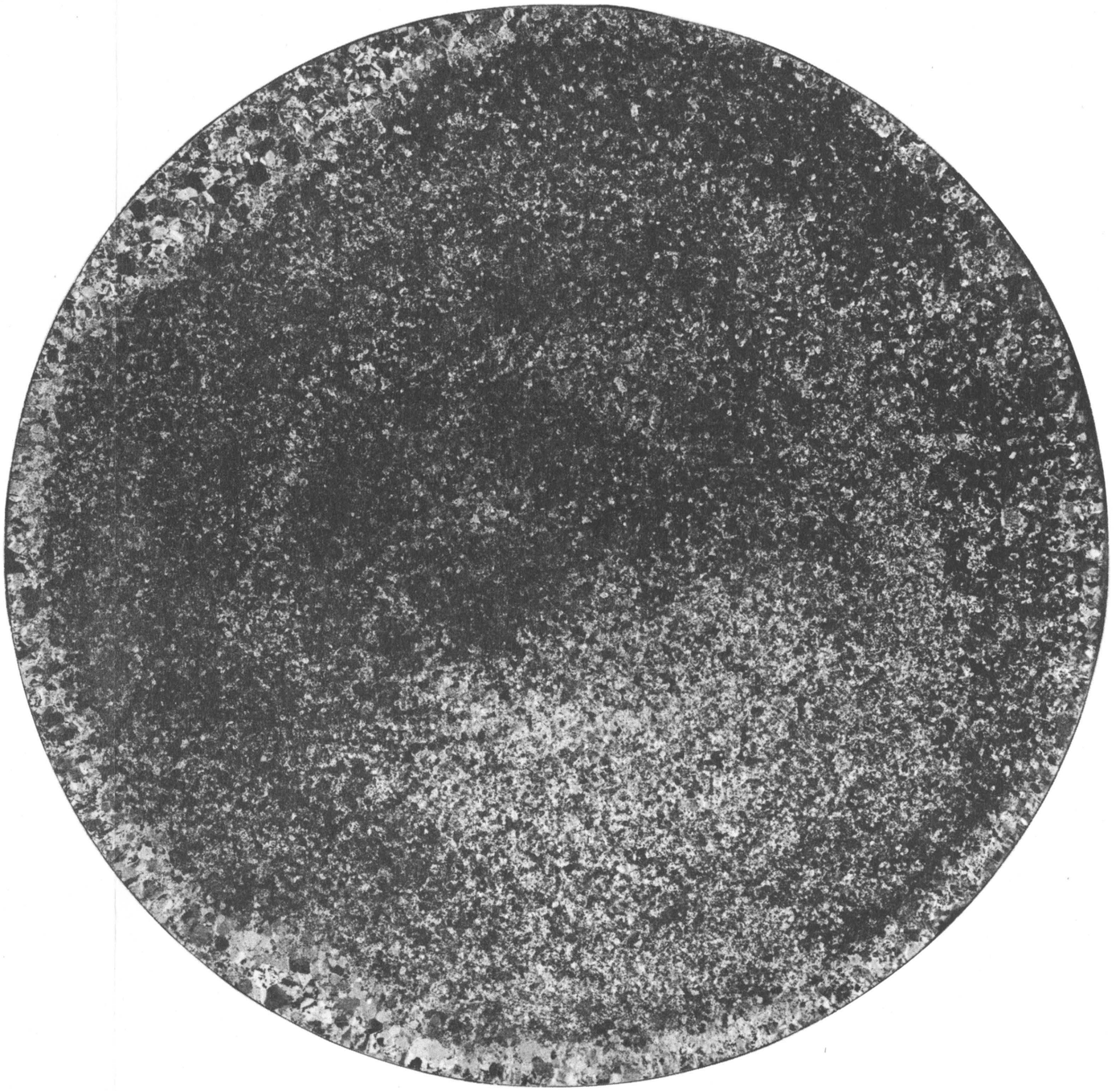
Chemical Analysis

In accord with general commercial practice, 1/8 inch was removed from the circumference of ingot 1 (manganese alloy - medium sponge hardness) and then a 1/16-inch cut was taken; the cut was interrupted every inch of length, and the chips were taken for chemical analysis. In this way, alloy distribution at the outer surface and from top to bottom of the ingot could be charted. Results of this study can be found in table 2. Except for the manganese content, the ingot is uniform chemically. Even with the manganese, the uniformity is about as good as could be expected. Lack of melting at the outside of the ingot causes greater heterogeneity there than at the center of the ingot. The low manganese content at the bottom of the ingot is also understandable, since melting is initiated by striking the arc on a mass of sponge at the bottom of the crucible. This sponge, much of which becomes part of the ingot, dilutes the manganese content. In general, the ingot is considered fairly homogeneous.

During machining of tensile and impact specimens, chips were taken from the cleaned metal for chemical analyses. Table 3 lists the resulting compositions. It can be noted from the table that ingot 1 is uniform in composition but that the two halves of ingot 2 (the low-hardness sponge ingot) vary considerably in manganese content, the press-forged billet showing the higher manganese. Ingots 2 and 3 also show high tungsten concentrations, because of loss of tungsten from the electrode during arc melting. As stated previously, (p. 4), attempts were made in testing to avoid those areas that showed heavy segregations of tungsten.

2/3 X

Figure 3. - Polished cross section of heel of ingot 1.



2/3 X

Figure 4. - Etched cross section of heel of ingot 1. Kellers etchant.

TABLE 2. - Distribution of alloys in ingot 1

Distance from top of ingot (inch)	Chemical composition, percent			
	C	Mn	Fe	N
0 - 1-1/4	0.050	7.78	0.07	0.008
1-1/4 - 2-1/2	.050	7.90	.09	.009
2-1/2 - 3-1/2	.022	7.86	.08	.010
3-1/2 - 4-1/2	.040	9.41	.07	.008
4-1/2 - 5-1/2	.044	8.62	.07	.008
5-1/2 - 6-1/2	.050	8.34	.08	.010
6-1/2 - 7-1/2	.054	8.25	.06	.008
7-1/2 - 8-1/2	.048	7.84	.06	.009
8-1/2 - 9-1/2	.056	7.79	.06	.007
9-1/2 - 10-1/2	.048	7.67	.06	.005
10-1/2 - 11-1/2	.048	8.14	.06	.010
11-1/2 - 12-1/2	.048	7.84	.06	.011
12-1/2 - 13-1/2	.038	7.09	.05	.011
13-1/2 - 14-1/2	.036	7.11	.05	.015
14-1/2 - 15-1/2	.042	6.70	.06	.015
15-1/2 - 16-1/2	.050	5.57	.06	.021

TABLE 3. - Chemical analysis of rolled sections

Section designation	Chemical composition, percent					
	C	Mn	Fe	N	Cl	W
H1A	0.066	7.45	0.045	0.030	0.021	0.044
H1B	.066	7.46	.067	.031	.019	.045
P1A	.056	7.48	.074	.019	.016	.058
P1B	.061	7.50	.051	.018	.016	.060
H2A	.038	8.01	.040	.049	.017	.032
H2B	.026	7.71	.042	.039	.032	.050
P2A	.032	8.96	.083	.026	.017	.500
P2B	.032	9.05	.042	.038	.012	.510
H3A	.050		.024	.030	.026	.012
H3B	.068		.060	.014	.024	.260
P3A	.064		.028	.029	.022	.450
P3B	.062		.024	.021	.020	.022 - .600

Macrostructure

A polished section of ingot 1, cut 2 inches from the butt end, is presented in figure 3 at approximately 0.7 diameter. The band of porosity observed approximately two thirds of the distance from the ingot center was found, under magnification, to be highly reflecting, smooth-walled gas pockets. The pattern of porosity caused by gas evolution would be expected to vary in degree and distribution in other ingot sections. The porosity is not considered significant, as subsequent working would obliterate it. The macrostructure of this section after etching in aqueous 3 percent hydrofluoric acid - 6 percent nitric acid is shown in figure 4. The grain size is uniform and equiaxed, except for one area of large grains at A; this was probably occasioned by local overheating by the arc.

Cross sections from the forged slabs (ingot 1) had the appearance shown in figures 5 and 6, the former illustrating the press forging and the latter the hammer forging. Smaller, more uniform grains and more even working occurred from hammer forging than from press forging. Sections from ingot 2 contained like structures.

The macrostructures of the rolled sections from ingot 1 are shown in figure 7 (press-forged billet) and figure 8 (hammer-forged billet). It can be seen from the figures that hot working above the transus produces uniform and equiaxed grains, whereas an elongated fiber structure is present when sections are worked below the transus. Local recrystallization, as seen in the central zone of H1B, (fig. 8), is indicative of nonuniform deformation during rolling. This phenomenon is most pronounced with light reductions and is associated with roll friction.

Mechanical Tests

The center section of each rolled plate was reserved for mechanical test specimens, which included 4 tensile specimens, 2 longitudinal and 2 transverse with respect to the rolling direction, and 4 Charpy-impact specimens, 2 longitudinal and 2 transverse. Due to an improper cooling procedure with sections P2A and H2A, the test on these sections had to be repeated without enough stock for all test bars. The tensile specimens were standard 0.505-inch diameter with threaded ends, and the Charpy specimens were standard size (0.394-inch square) with the V-notch cut perpendicular to the rolling plane (that is, through the plate thickness). Vickers hardness tests (10-kg. load) were made on the Charpy specimens after breakage. The specimens were not mounted in bakelite before hardness testing, as the titanium-manganese alloy is subject to age hardening at 300°-400° F., temperatures that are employed in curing bakelite.

Results of these tests are given in table 4 Ingot 1 shows remarkable uniformity regardless of the method of forging used (hammer or press) and regardless of rolling temperature. Ingot 2 is much less uniform, as one would expect from the variation in chemical composition (table 3). The lower hardness of the sponge making up this ingot is reflected in the lower strength of the bars from the hammer-forged and rolled sections. The slightly higher manganese content, as compared to that of ingot 1, is not enough to make up for the strength imparted to ingot 1 by virtue of the higher interstitial content. Bars from the press-forged and rolled sections (ingot 2), however, are the strongest listed; here the particularly high manganese content (about 9 percent) has overcome the initial low hardness of the sponge. As far as impact strength is concerned, however, replacement of interstitial content by an increase in manganese has done little to improve this property. The value of 32 ft.-lb. impact for P2BL represents a step in this direction, but the other bars in the series do not follow. Moreover, the ductility of this series is considerably lower than that of the bars from ingot 1. It is suspected that unknown segregations may have been present in this ingot.

That impact strength varies inversely (in a rough fashion) with ultimate strength is seen in figure 9. The nonuniformity of ingot 2 is again manifest here, as there is more spread than in ingot 1.

Rolling temperature had little effect on mechanical properties. In ingot 1, the higher rolling temperature resulted in higher impact strength; the reverse was true with ingot 2. On the whole, however, these changes were slight, and there is little to choose between the two temperatures.

TABLE 4. - Mechanical properties of rolled sections furnace cooled from the rolling temperature

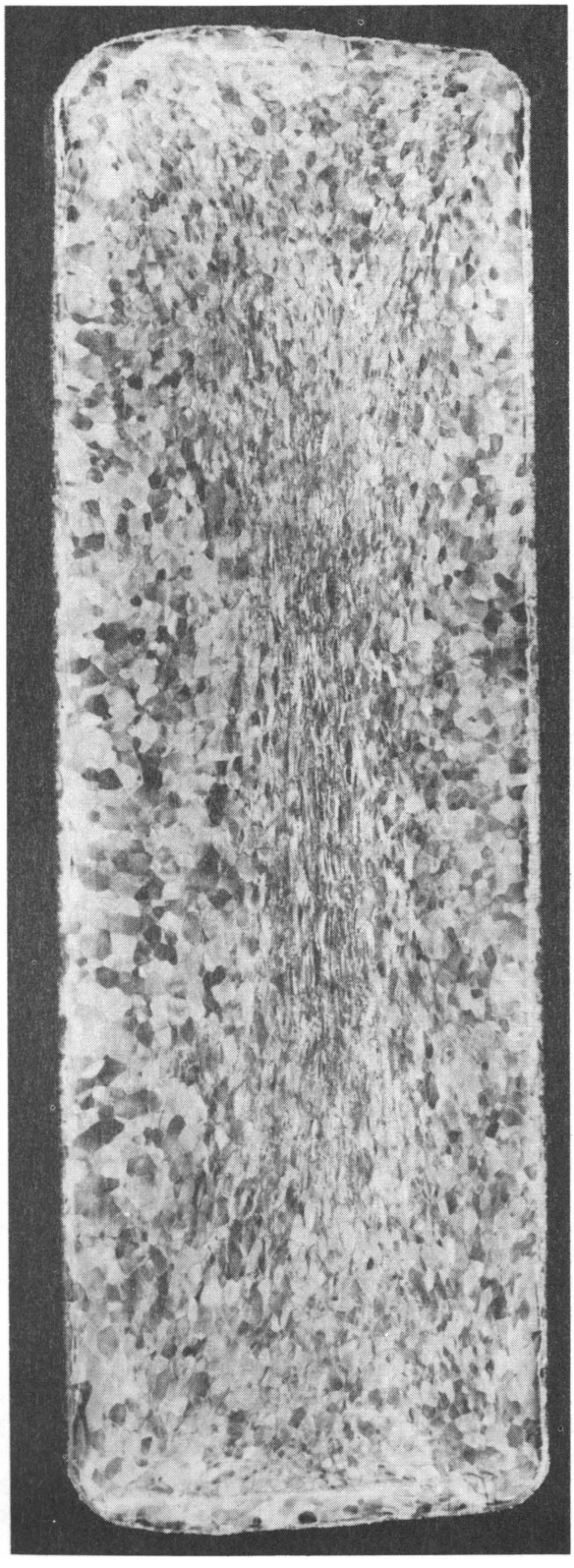
(Average of two tests)

Section[1] No.	Rolling temp., °C.	Yield strength, p.s.i. (0.2-percent offset)	Ultimate strength, p.s.i.	Elongation in 2 inches, percent	Charpy impact energy, ft.-lb.	Vickers hardness No. (10-kg. load)
		Ingot 1 (Ti-7 percent Mn)				
H1AL.....	850	118,400	133,500	19.0	22.0	360
H1AT.....	850	121,900	134,300	15.5	25.0	357
H1BL.....	750	121,100	131,500	18.5	19.0	370
H1BT.....	750	129,300	139,400	20.0	13.5	368
P1AL.....	850	109,400	131,600	17.0	22.0	320
P1AT.....	850	114,300	132,400	17.0	21.5	318
P1BL.....	750	123,200	131,200	16.5	16.5	340
P1BT.....	750	121,200	136,100	16.5	19.0	330
		Ingot 2 (Ti-7 percent Mn)				
H2AT.....	850	95,100	115,300	18.3	27.5	277
H2BL.....	750	104,500	122,800	18.0	29.5	297
H2BT.....	750	121,800	127,200	16.5	14.5	293
P2AL.....	850	128,100	142,200	9.5	13.0	317
P2BL.....	750	126,800	133,100	13.5	32.0	329
P2BT.....	750	128,800	139,400	12.0	20.0	327
		Ingot 3 (unalloyed)				
H3AL.....	950	41,000	55,600	34.0	65.5	176
H3AT.....	950	51,100	61,100	29.3	79.5	172
H3BL.....	850	42,100	60,300	32.8	103.5	179
H3BT.....	850	53,200	61,900	30.5	113.0	168
P3AL.....	950	50,800	69,200	25.0	40.0	197
P3AT.....	950	64,100	77,900	25.0	47.0	183
P3BL.....	850	45,700	65,700	30.8	55.5	190
P3BT.....	850	55,900	67,700	30.0	147.5	188

1/ "L" or "T" refers to longitudinal and transverse sections, as related to rolling direction.

Unalloyed ingot 3 is low in strength and very ductile, as would be expected. Somewhat higher strength is observable in the press-forged half of the ingot than in the hammer-forged half; this is felt to be due to differences in chemistry, probably oxygen content, rather than to differences in forging method. Very high impact values are evident in some cases, notably H3BL, H3BT, and P3BT - all products of the lower rolling temperature (850° C.). This higher impact strength is probably due to the smaller grain size obtained with the lower rolling temperature. The maximum impact value (147.5 ft.-lb.) is somewhat of an anomaly, as the section with this value (P3BT) has higher strength than either H3BL or H3BT.

These high values are not truly indicative of the total energy required to break the specimen because some bending occurs when material of high ductility is tested and part of the absorbed energy is thus used in deforming the specimen. It is observed from table 4 also that the high impact values occur in the bars cut transverse to the rolling direction, whereas one would expect that bars cut in the direction of rolling would show the better impact strength. It may be that directional properties induced during forging (which elongated the bar in the reverse direction from rolling) were retained through the rolling process.



1 X

Figure 5. - Etched cross section of press-forged slab, ingot 1. Kellers etchant.

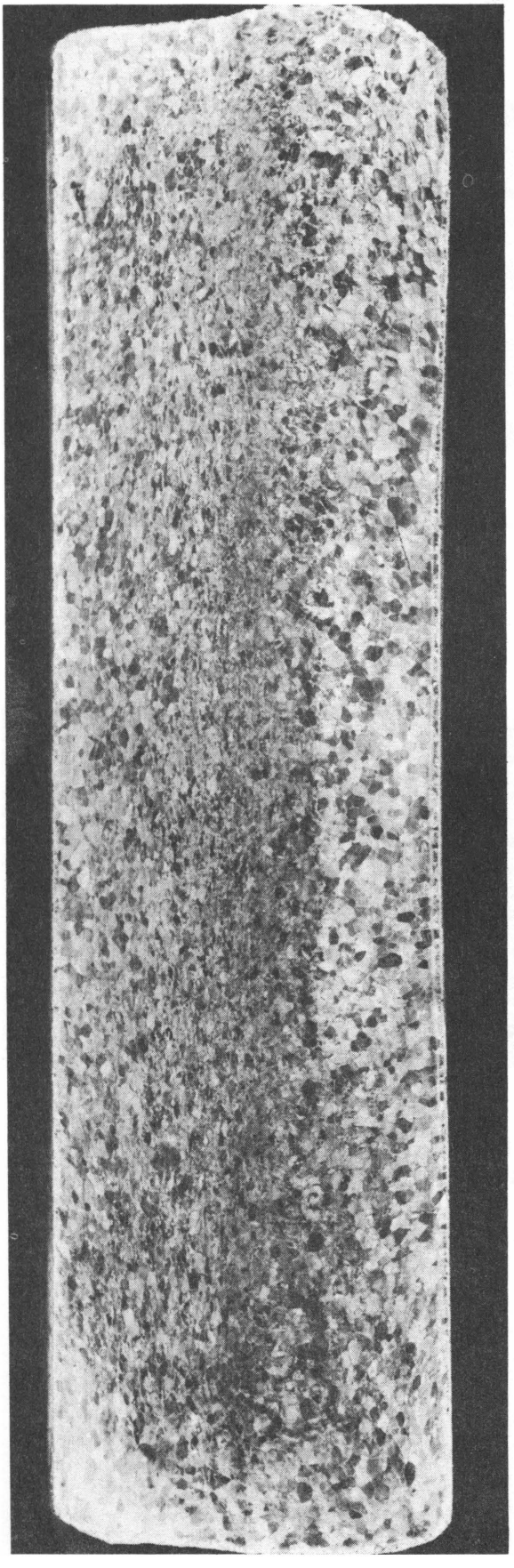


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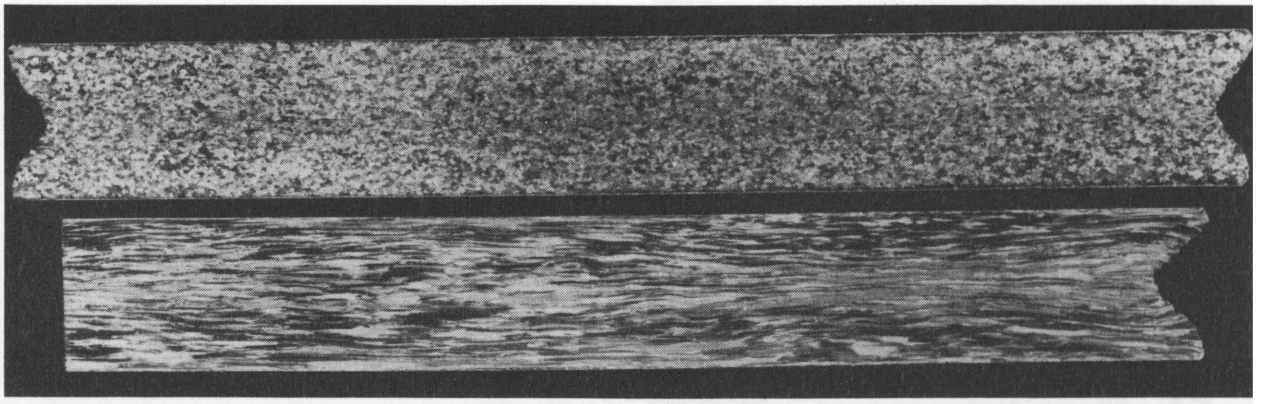
Figure 6. - Etched cross section of hammer-forged slab, ingot 1. Kellers etchant.



1 X

Figure 7. - Macrostructures of rolled sections from press-forged billet, ingot 1. Kellers etchant. A. Upper 850° C. B. Lower 750° C.

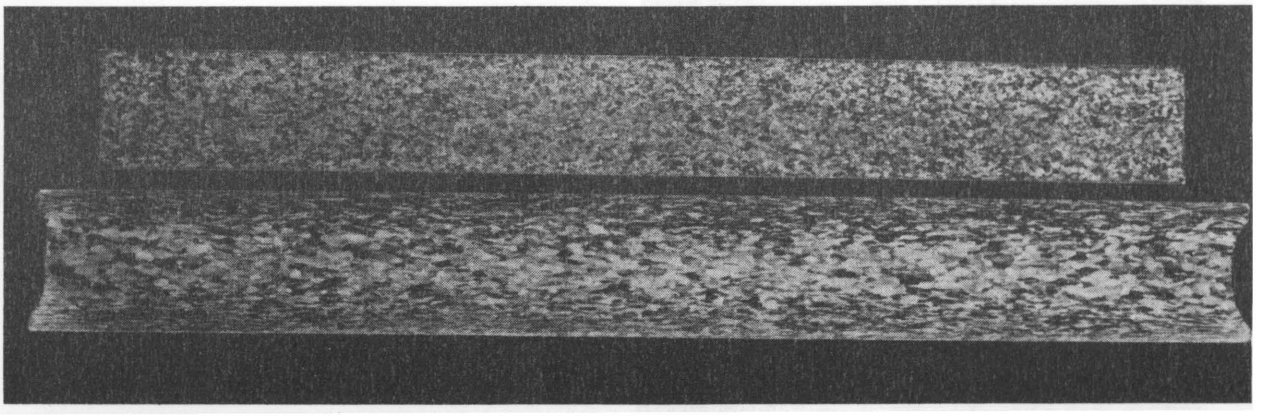


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Figure 8. - Macrostructures of rolled sections from hammer-forged billet, ingot 1. Kellers etchant. A. Upper 850° C. B. Lower 750° C.

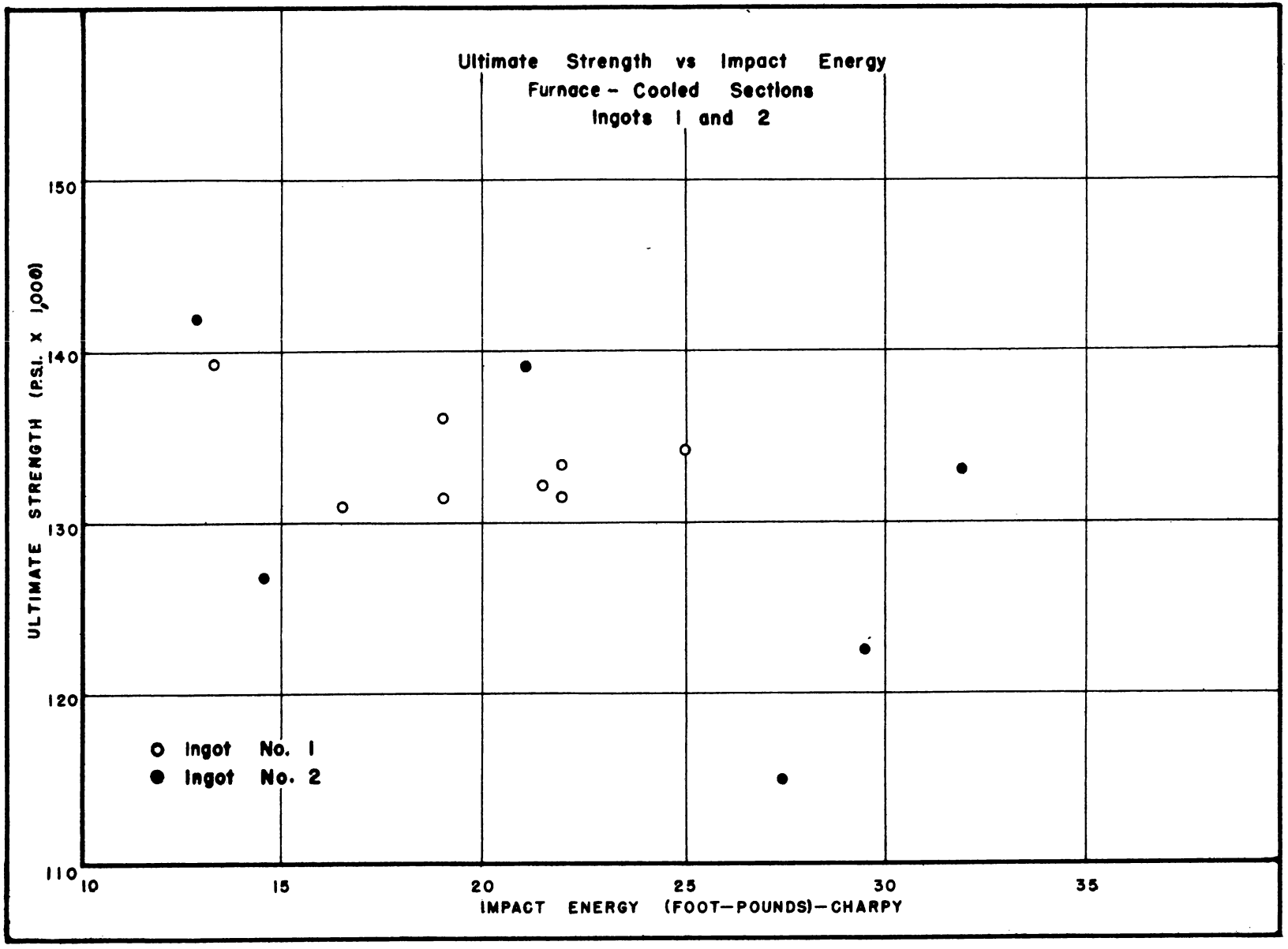


Figure 9. - Relationship between ultimate strength and impact energy, alloy material.

Mechanical properties of the section from ingot 2, which had been press-forged and then rolled at 650° C. (1,200° F.), are as follows:

Section P2D

Direction	Yield strength, p.s.i. (0.2-percent offset)	Ultimate strength, p.s.i.	Elongation in 1 inch, percent	Charpy impact energy, ft.-lb.
Longitudinal	118,200	131,000	17.5	10
Transverse	134,000	135,700	20.3	9.5

The tensile bars are subsize standard ASTM (0.250-inch diameter measured section).

These properties compare favorably strengthwise with those of the sections rolled at higher temperatures; as a matter of fact, the elongation here is somewhat higher. The impact strength, however, is much lower, with no apparent reason.

Table 5 gives the mechanical test results of the bars water-quenched from the rolling temperature. These also are subsize standard bars, 0.250-inch diameter measured section. The specimens from ingot 1, which had been quenched from above the transus, were so hard and brittle that they broke in the test-bar shoulder with low elongation and without a true strength recording. The bars (from both ingots), which were quenched from below the transus were also very hard and strong with low impact strength, but their ductility was fair.

TABLE 5. - Mechanical properties of rolled pieces water-quenched from rolling temperature

(Ingots 1 and 2)1/

From billet No.	Rolling temp., °C.	Yield strength, p.s.i. (0.2-percent offset)	Ultimate strength, p.s.i.	Elongation in 1 inch percent	Charpy impact energy, ft.-lb.	Vickers hardness No. (10-kg. load
H1.....	845	No curve obtained	166,800	(2)	3	443
H1.....	835	do.	119,300	(2)	4	439
H1.....	815	do.	161,800	(2)	6	446
H1.....	760	167,800	176,000	7.0	4	416
H1.....	745	161,700	167,600	7.5	5	399
H1.....	730	151,000	159,800	11.5	9	391
P2.....	760	148,100	161,100	11.0	8	390
P2.....	745	152,500	157,600	10.5	9	396
P2.....	730	150,000	154,600	8.0	7	399

Note: Refer to table 3 for chemical analysis.

1/ Each value is an average of 2 tests. All specimens are longitudinal with respect to the rolling direction.

2/ Broke outside gage length.

The low impact values of all specimens may have been due in part to the fact that the sections had been rolled perpendicular to the forging direction (that is, the slab thickness became the rolling plane). Impact energies might have been higher if simple cross rolling had been resorted to, as in the case of the furnace-cooled sections.

There was little to choose between the properties of H1 and P2, except that the latter were slightly softer and more ductile, as would be expected.

Some of the rolled sections were chosen for a study of the effect of temperature on impact properties. For the manganese alloys, sections P1B (ingot 1 press-forged, then rolled at 750° C.) and P2B (ingot 2 press-forged, then rolled at 750° C.) were selected, and the Charpy bars taken from them were cut in the transverse direction. For ingot 3 (unalloyed), sections P3A (press-forged, then rolled at 950° C.) and H3B (hammer-forged, then rolled at 850° C.) were selected, the bars for the former taken in the longitudinal direction and those for the latter taken in the transverse direction.

Temperatures of impact testing ranged from -195° to 400° C. Heating and cooling mediums used to obtain these temperatures were liquid nitrogen, a dry ice-alcohol mixture, temperature-control baths, and electric furnaces.

All specimens were held at temperature for 15 minutes. The specimens were transferred from the bath to the impact-testing machine with a special pair of tongs that automatically centered the specimen in the impact testing machine. In this way no more than 5 seconds was lost between removal from the bath and striking with the hammer. The tongs were also immersed in the bath with the specimens during the heating and cooling periods.

Table 6 lists the results of this test, and figures 10 and 11 show the transition curves for the alloy ingots and for the commercially pure ingot, respectively. The third curve in figure 10 for H2T is discussed later. The two sets of data on the alloy ingots agree very well; the transition curves (fig. 10) are virtually superimposed on each other. These are typical curves for this alloy. It can be seen that the lower interstitial content of ingot 2 did not increase the impact strength of this material above that of ingot 1 at high temperatures, as would ordinarily be expected. The unalloyed material (fig. 11) retains high impact strength even at low temperatures. The difference in the two curves is probably attributable to the difference in rolling temperature. The falling off of values at higher temperatures, particularly noticeable in the case of Section H3BT, is unusual as there are no microstructural or hardness changes.

TABLE 6. - Impact energy at various temperatures

Testing temperature, °C.	Impact energy, ft.-lb.1/			
	Ti-Mn alloy		Unalloyed titanium	
	Ingot 1, press-forged, rolled at 750° C. (P1BT)	Ingot 2, press-forged, rolled at 750° C. (P2BT)	Ingot 3, hammer-forged, rolled at 850° C. (H3BT)	Ingot 3, press-forged, rolled at 950° C. (P3AL)
-195.8.....	3	-	-	23
- 78.5.....	-	5	52	26
0	-	13	94	27
Room temp..	19	20	113	37
80	-	23	150	65
125	34	32	143	-
160	-	-	-	81
200	44	46	126	91
300	60	53	115	-
400	70	-	115	83

1/ Average of 3 values.

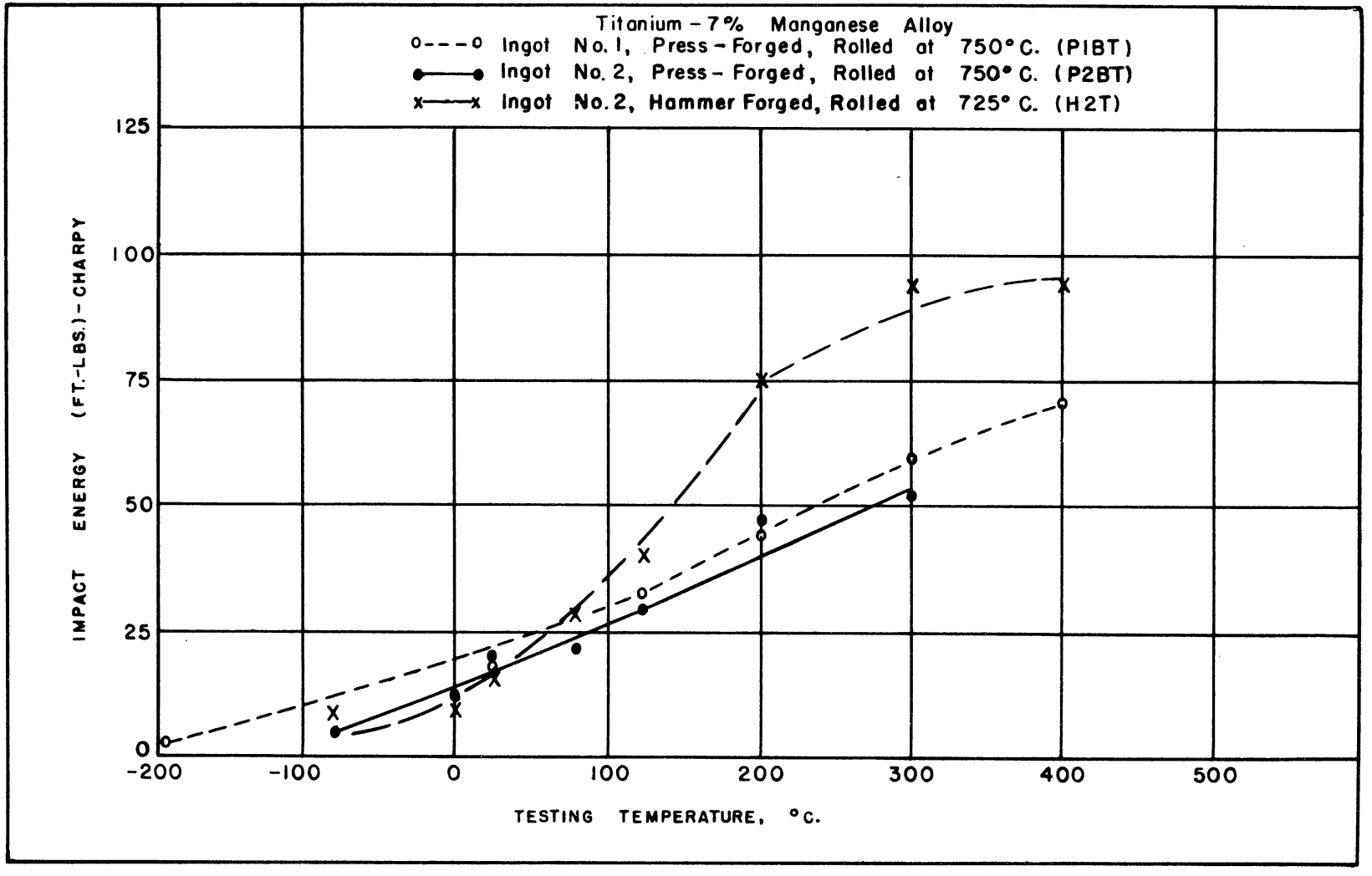


Figure 10. - Impact-temperature curves, alloy material.

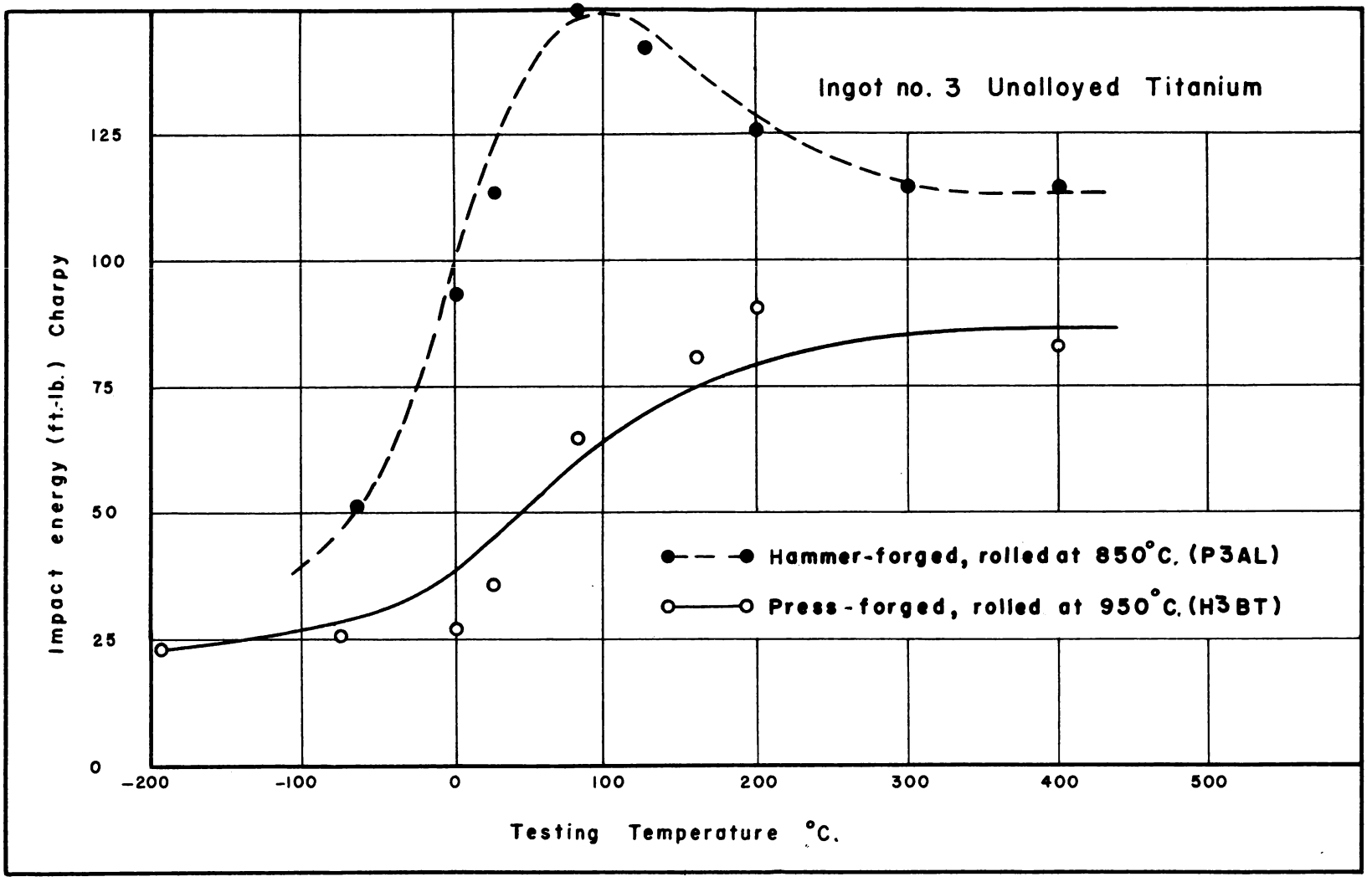


Figure 11. - Impact-temperature curves, unalloyed material.

Microstructure and X-ray Diffraction Studies

In preparing samples for metallographic study, precautions were taken to remove disturbed metal produced by cutting and to prevent overheating during cutting and grinding. Metallographic specimens were cut with a power hacksaw cooled by liquid, and rough ground (removal about 0.010 inch) on a wet-belt sander (80 grit) to remove disturbed metal from the cutoff operation. After the specimens were mounted in bakelite, they were fine-ground on 220- and 320-grit silicon carbide papers, then on 400-and 500-grit emery papers. They were then polished electrolytically under the following conditions:

Solution: Methyl alcohol, 59 percent by volume.
Butyl cellosolve (ethylene glycol monobutyl ether), 35 percent by volume.
70 percent perchloric acid, 6 percent by volume.

Cathode: Stainless steel.

Anode current density: 0.5 amp. per sq. cm. of exposed surface area.

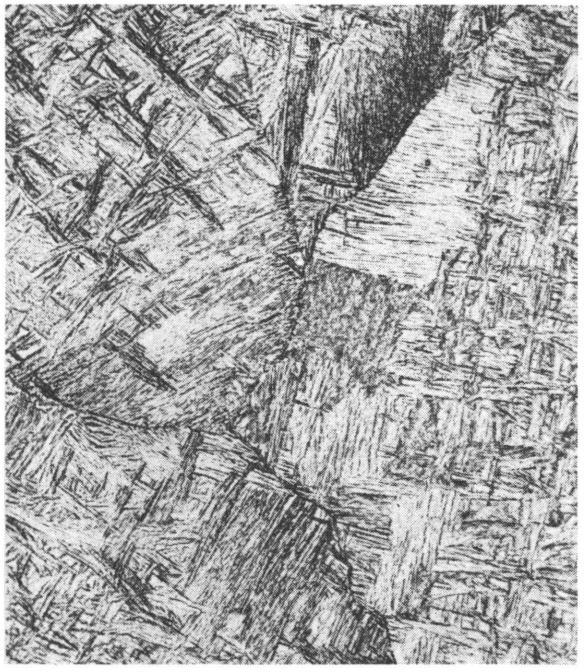
Polishing time: 20-25 seconds.

The metallographic structure was developed by etching in an aqueous solution containing 1.5 percent hydrofluoric acid and 3 percent nitric acid.

Figures 12 through 19 show the microstructures at 250 diameters of the rolled sections from the 2 alloy ingots as furnace-cooled from the rolling temperature. Following is a key to these figures:

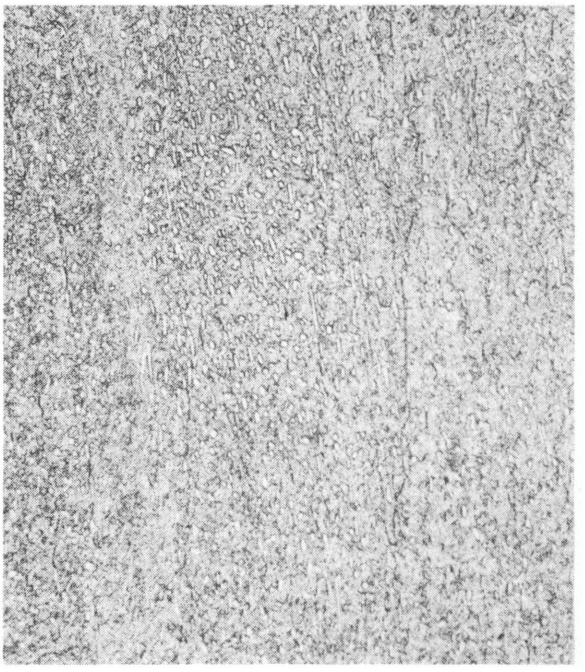
Fig.	Section No. (see table 1)	Rolling temp., °C.
12.....	P1A	850
13.....	P1B	750
14.....	H1A	850
15.....	H1B	750
16.....	P2A	850
17.....	P2B	750
18.....	H2A	850
19.....	H2B	750

All sections rolled at 850° C., which is above the transformation temperature, have the same type of structure. This displays the familiar "basket-weave" or Widmanstatten pattern, in which alpha platelets have precipitated along cleavage planes in a beta matrix. Some differences are observable, however, in sections rolled at 750° C. Those sections from ingot 1 (medium sponge hardness), the structures of which are shown in figures 13 and 15, show globular alpha precipitation in a beta matrix; this is carried farther in figure 15 than in figure 13, where one notes also some fine unresolved alpha in the matrix. This is the expected structure for material rolled below the transus. In the case of the sections from ingot 2, however, such globular alpha precipitation is only slightly evident in figure 19 (H2B) and not present at all in figure 17 (P2B), which contains instead a Widmanstatten structure. It is deduced from these figures that a rolling temperature of 750° C. is above the transus in the case of section P2B and at or slightly above the transus in the case of section H2B. This is plausible, as the higher manganese content and lower interstitial content of ingot 2 should make for a lower transition temperature than would be the case for ingot 1.



250 X

Figure 12. - Microstructure of press-forged material rolled at 850° C.–ingot 1. Kellers etchant.

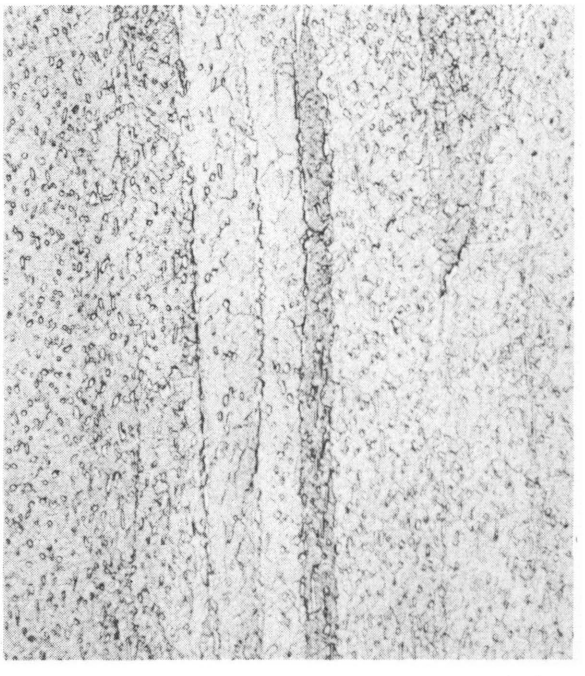


250 X

Figure 13. - Microstructure of press-forged material rolled at 750° C.–ingot 1. Kellers etchant.

250 X

Figure 14. - Microstructure of hammer-forged material rolled at 850° C.–ingot 1. Kellers etchant.

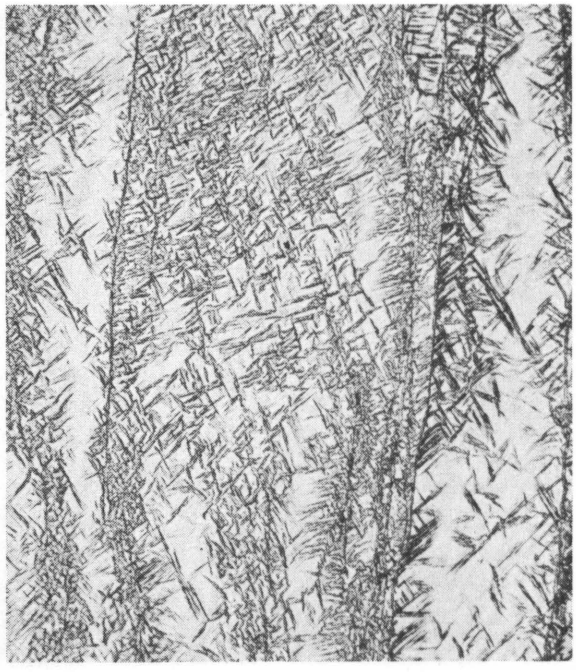


250 X

Figure 15. - Microstructure of hammer-forged material rolled at 750° C.–ingot 1. Kellers etchant.

250 X

Figure 16. - Microstructure of press-forged material rolled at 850° C.–ingot 2. Electropolish.

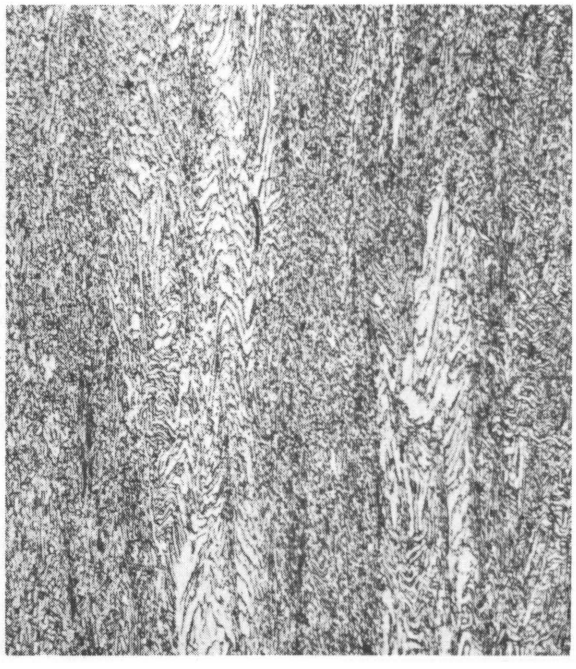


250 X

Figure 17. - Microstructure of press-forged material rolled at 750° C.–ingot 2. Kellers etchant.

250 X

Figure 18. - Microstructure of hammer-forged material rolled at 850° C.–ingot 2. Electropolish.



250 X

Figure 19. - Microstructure of hammer-forged material rolled at 750° C.–ingot 2. Keller etchant.

Because of these findings, it was thought best to determine the transformation temperatures of samples from both ingots. To obtain this information, 5 samples measuring 1 by 3/4 by 1/8 inch were cut from each of four sections: P1B, H1B, P2A, and H2A. The specimens were heated to temperatures ranging from 690° to 770° C. in 20° steps and then water-quenched to retain whatever beta was formed. The specimens were held for 2 hours at temperature in a tube furnace through which a positive flow of helium was maintained. After heat treatment, 0.010 inch was removed from the specimens by wet grinding to rid them of surface contamination. The specimens were then hand-polished to a micro finish and etched.

X-ray diffraction data on these specimens were obtained with a wide-angle diffractometer employing Cu K-alpha radiation at 40 kilovolts and 20 milliamperes. The intensity of the alpha-titanium 101 reflection was measured for each specimen. Three independent runs were made in each case, and averages were taken. The data are listed in table 7 and plotted in figure 20. The curves of figure 20 were extrapolated to zero intensity to locate the transus temperature. As the relationship is purported to be linear and as values at lower intensities are more precise, straight lines were drawn, and points representing lower intensities were given more weight. Little difference would exist in the final result if curves were drawn instead of straight lines.

TABLE 7. - Determination of alpha intensities on water-quenched specimens

Sample No.	Quenching temperature, °C.	Relative intensity of 101 reflection alpha titanium
P1B....	770	7
	750	20
	730	32
	710	49
	690	68
H1B....	770	10
	750	26
	730	40
	710	57
	690	66
P2A....	770	-
	750	-
	730	7
	710	19
	690	32
H2A....	770	-
	750	7
	730	42
	710	47
	690	61

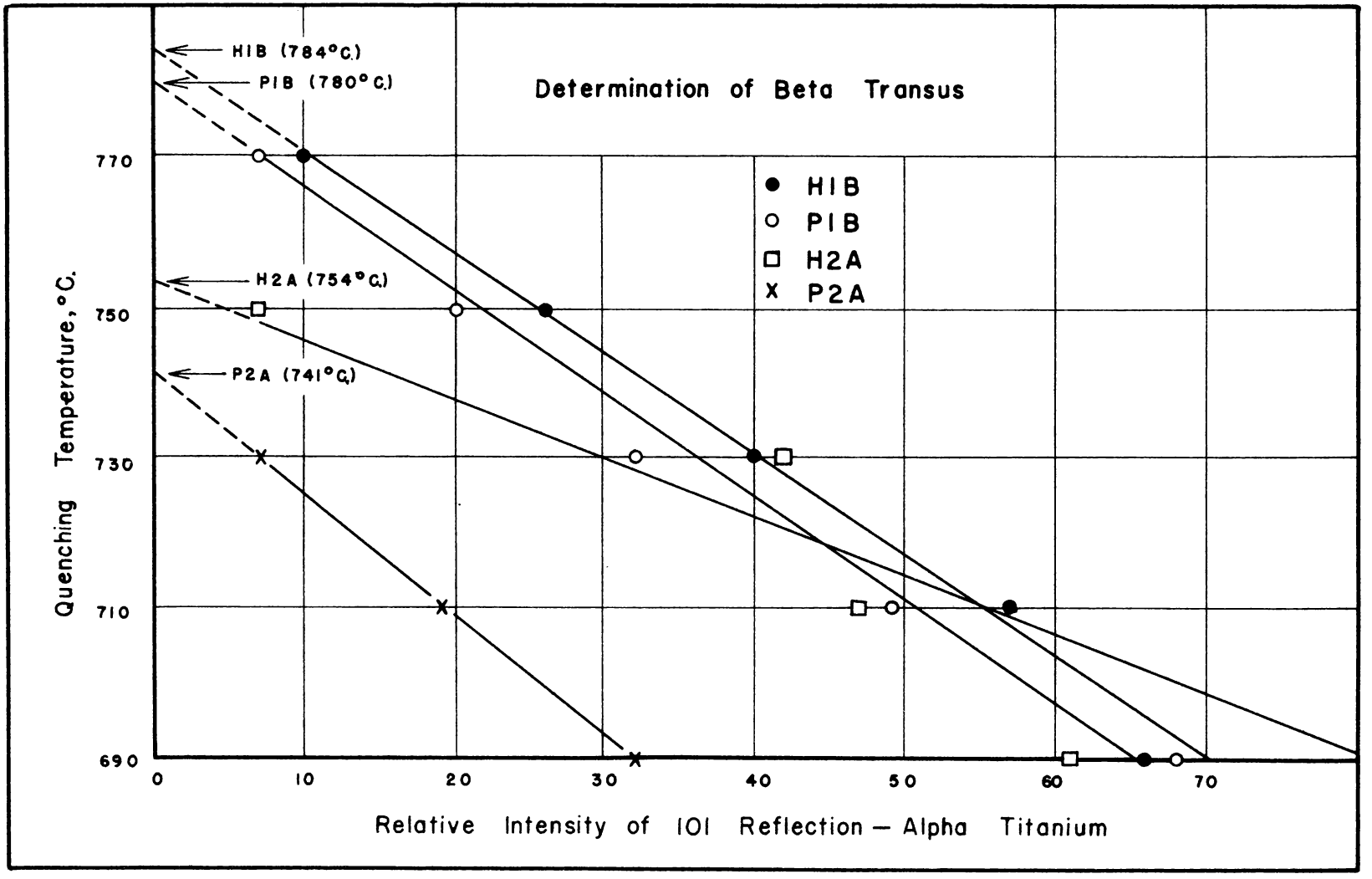


Figure 20. - Determination of beta transus, ingots 1 and 2.

The manganese content of the specimens was determined also, by means of a fluorescent X-ray spectrograph.7/ The polished and etched samples were excited at 30 kilvolts, 45 milliamperes, and the resultant fluorescent radiation was refracted by a quartz-crystal analyzer. The Mn K-alpha line intensities were recorded, and the percentage manganese was computed from standards.

The transition temperatures and manganese contents of the specimens are as follows:

Section No.	Transition temperature, °C.	Manganese, percent
P1B.....	780(1,436° F.)	8.26
H1B.....	784(1,443° F.)	7.82
P2A.....	741(1,366° F.)	9.32
H2A.....	754(1,389° F.)	7.86

As had been suspected, the transition temperatures shown for the specimens from ingot 2 were in one instance somewhat below the lower rolling temperature and in the other at about the same level. The structures observed in figures 17 and 19, then, are understandable in the light of these data.

From the above, it became evident that no sections from ingot 2 had been rolled below the transition temperature. It was decided to remedy this situation to see if any improvement in mechanical properties would result. Accordingly a section from billet H2, which was all that was left of ingot 2, was rolled at 725° C., about 25° below the transus. The section was heated to 725° C. (1,337° F.) and given a 1/2-hour soak at this temperature before rolling was begun. The section was cross-rolled in relation to the forging direction. From the original thickness of 2-1/2 inches down to 1-1/2 inches, the section was reduced 0.050 inch per pass, with 2 passes between reheats. From a thickness of 1-1/8 inches down to the final thickness of 3/4 inch, only 1 pass (0.050-inch reduction) was taken between reheats. After rolling, the plate was again heated to 725° C., and then furnace-cooled at a rate of about 3° per minute.

Four standard 0.505 inch-diameter tensile bars and enough Charpy impact specimens to establish a temperature-impact transition curve were machined from this rolled section. Both longitudinal and transverse tensile and impact bars were prepared for room temperature testing. Tensile results and impact values at room temperature are as follows:

Direction	Yield strength, p.s.i. (0.2 percent offset)	Ultimate strength, p.s.i.	Elongation in 2 inches, percent	Charpy impact energy, ft.-lb.
Longitudinal	103,700	116,500	17.5	16
Transverse	114,700	121,800	17.5	15

Results are averages of 2 tests.

There is little to choose between these properties and those of billet H2 rolled at 750° C. (see table 4); moreover, the impact value is somewhat less in the longitudinal direction. It appears that, insofar as room-temperature properties are concerned, nothing is to be gained from rolling at a temperature well below the transus.

7/ American Society for Testing Materials, Symposium on Fluorescent X-ray Spectrographic Analysis: Special Tech. Pub. 157, June 29, 1954, 68 pp.

Impact specimens for the transition curve were all in the transverse direction. Results have been plotted in figure 10 and are as follows:

Temperature of Testing, °C.	Charpy impact energy, ft.-lb. (ave. of 3 tests)
-78.5	9
0	11
Room temperature	16
80	28
125	43
200	75
300	92
400	91

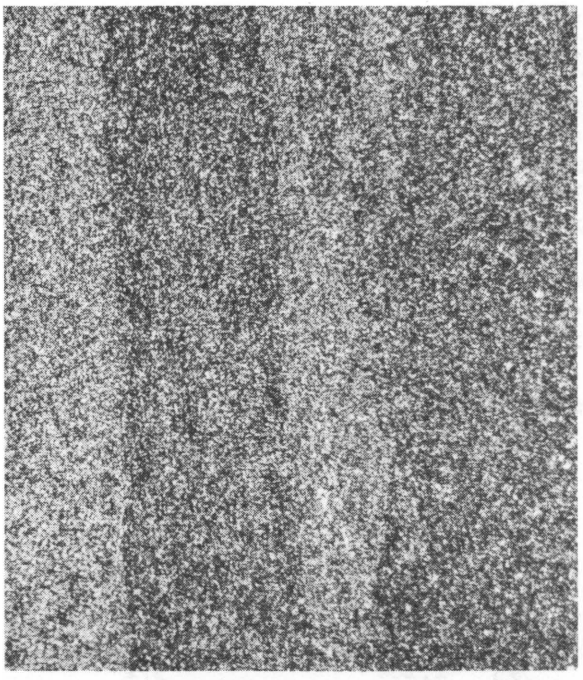
Up to 80° C. this material is no better in impact than that rolled at 750° C. However, at 80° C. and above, the energy absorbed in impact increases at a much greater rate, until values of over 90 are recorded at 300° and 400° C. as opposed to a maximum of 53 (at 300° C.) for the material rolled at 750° C. (see table 6). This greater toughness at high temperatures may indicate greater structural stability, owing to rolling at subtransus temperatures. It is interesting to note that this material also has higher impact strengths in the range 80°-400° C. than does the section from ingot 1 rolled at 750° C. The lower interstitial content of ingot 2 may be responsible for this difference.

The microstructure of billet H2 rolled at 725° C. is what would be expected; it resembles figures 13 and 15.

Figure 21 shows the microstructure of the section from billet P2, which had been warm-rolled at 650° C. (1,200° F.). It consists of fine alpha particles in a beta matrix; the temperature reached was not high enough for agglomeration of the alpha.

Age-Hardening Studies

Commercially pure titanium is the so-called all-alpha type of titanium; that is, on cooling it transforms from the high-temperature beta form to the low-temperature alpha form. Changes in cooling rates from the beta field do not affect this transformation; hence the properties and microstructure of commercially pure titanium remain virtually the same regardless of heat treatment. In an alloy such as the 7 percent manganese-titanium alloy, however, profound changes can occur through heat treatment. This alloy is one of a family called the alpha-beta type. This means that at room temperature, owing to the fact that manganese stabilizes the beta phase, the common microstructure consists of a beta matrix containing alpha particles. This is the condition on slow cooling. When the alloy is quenched rapidly from the beta field, however, the transformation is suppressed, and the room-temperature microstructure consists of beta alone. This is an unstable condition, and heating to 400° F. or above will cause precipitation of the alpha by a nucleation and growth process. The rate of precipitation increases as the temperature is raised above 400° F. At low temperatures and short times the alpha particles formed are submicroscopic in size and distributed in a fine array, and hardnesses are higher than the as-quenched hardness. At higher temperatures and longer times the alpha particles become larger and more numerous, with consequent softening of the material. When this occurs the material is said to be overaged. These phenomena occur in most age-hardening processes.



250 X

Figure 21. - Microstructure of press-forged material rolled at 650° C.–ingot 2. Kellers etchant.

It was thought worthwhile to study age hardening as applied to the 7 percent-manganese alloy, particularly to determine what temperatures and times were needed to cause complete stabilization, that is, overaging, of the alloy. For this study, bars 1/2 inch square by 4 inches long were cut from bar H1B (medium-hardness sponge, hammer-forged, rolled at 750° C.). These were solution-treated for 2 hours at 950° C. (1,742° F.) in a 2-inch-diameter tube furnace and then water-quenched. Contamination at temperature was avoided by maintaining a positive helium flow of 4 cu. ft. per hour through the furnace tube. The solution treated bars were then cut transversely into 1/8-inch slices, which were used for the age-hardening studies. The bars were cut with a water-cooled milling cutter so that localized heating would be avoided. Temperatures of aging ranged from 205° to 595° C. (400° to 1,100° F.), in steps of 100°; time at temperature was 2 hours. The hardness of each specimen after treatment was as follows:

Aging temperature		Vickers hardness No.
°C.	°F.	
204	400	391
260	500	417
315	600	522
371	700	560
427	800	530
482	900	449
538	1,000	403
593	1,100	371

The hardness of the as-quenched specimen (no reheat) was 375 Vickers.

It can be seen from the above table that a 700° F. treatment yielded the highest hardness. To confirm this, 2 more specimens were aged, 1 at 650° F. and the other at 750° F.; resulting hardnesses were 539 and 545 Vickers, respectively, both lower than that resulting from the 700° F. treatment. The curve of hardness against aging temperature is shown in figure 22.

The most effective aging temperature having been established, the next step was to determine the effect of aging time at temperatures at and near this temperature. For this study, temperatures of 600°, 650°, 700°, 750°, and 800° F. were chosen, and samples were held at these temperatures for 1/4 hour to 200 hours. For aging periods under 4 hours, samples were heated in a helium atmosphere; for longer aging periods, each sample was sealed with titanium chips (to act as a getter) in an evacuated pyrex tube. After treatment, the samples were tested for hardness. The results are plotted in figure 23, which shows the variation in hardness as time at temperature is increased. The following table lists some of the salient data to be gained from figure 23:

Aging temperature		Max. hardness	Time to obtain max. hardness, hr.	Time at which overaging begins, hr.
°C.	°F.	VPN		
315	600	> 553	> 200	> 200
343	650	554	24	24-72
371	700	560	2	24
399	750	551	<1/4	4
427	800	556	<1/4	4

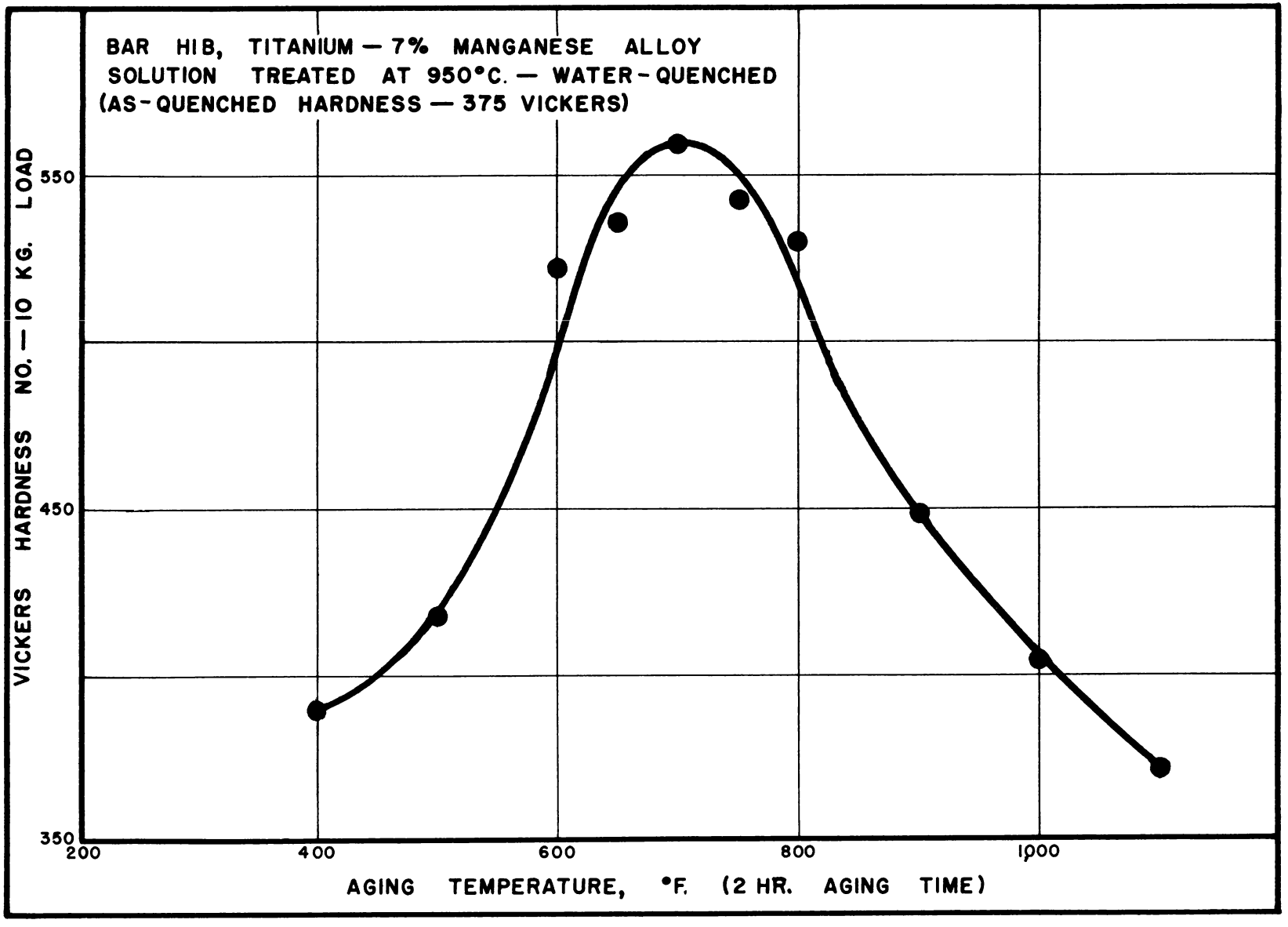


Figure 22. - Hardness-temperature curve for aging times of 2 hours—ingot 1.

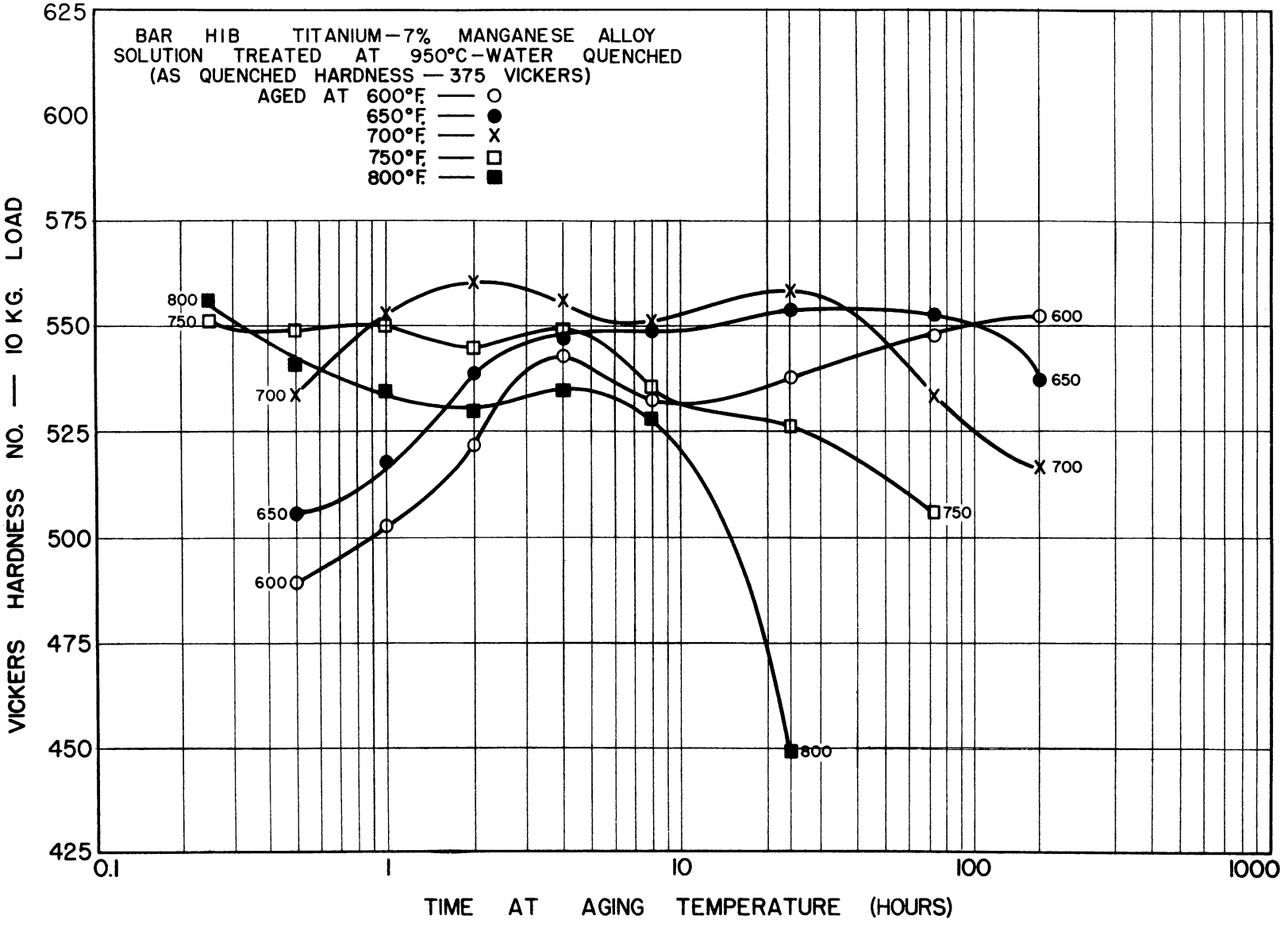


Figure 23. - Curves showing hardness changes as a result of variations in aging temperatures and times—ingot 1.

It can be observed that the time to maximum hardness and that to the onset of overaging (the period, after maximum hardness has been reached, during which a definite softening trend occurs) are of long duration at the lower temperatures and of short duration at the higher temperatures. There is little variation in maximum hardness, which was over 550 VPN regardless of temperature. It can also be seen that each curve in figure 23 shows two hardness humps, with softening in between; for the lower temperatures, the second hump is higher than the first, whereas the reverse is true at the higher temperatures. It has been suggested that this phenomenon is caused by the appearance of transition phase as beta transforms to alpha during aging.8/

Overaging is significant in that it represents a stabilized condition, as opposed to the unstable as-quenched and partly aged states. For some of the commonly used titanium alloys, it is standard practice to solution treat and overage, thus rendering the alloy stable with balanced mechanical properties.

Precipitation of alpha in the aged samples was not observed microscopically until the samples were overaged. Figure 24, a photomicrograph of a specimen overaged by a treatment of 4 hours at 800° F., shows a fine peppery array of alpha in the retained beta matrix. Relief lighting was necessary to photograph the structure. Lack of this fine precipitation is evident in the photomicrograph (fig. 25), which shows a sample water-quenched from 950° C. with no subsequent aging treatment. This sample was also photographed with relief lighting.

CONCLUSIONS

1. Hammer forging of these ingots appears to result in more even working and greater homogeneity than press forging.

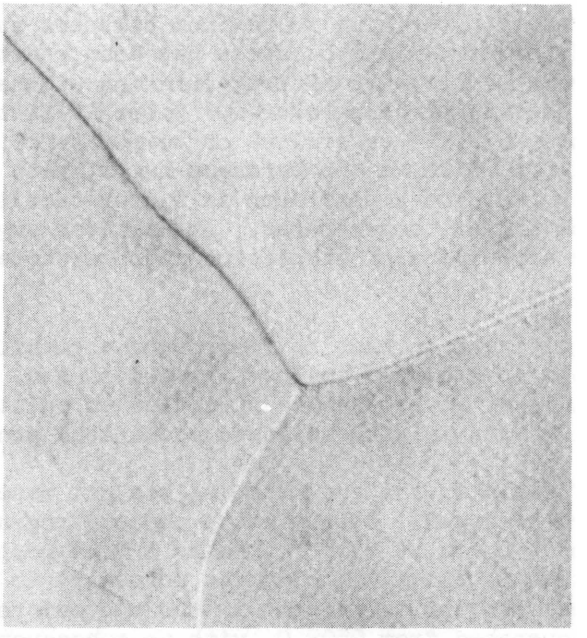
2. Due to higher strength and the necessity of a lower rolling temperature, rolling reductions per pass on the titanium-manganese alloy are about half those for unalloyed material. No difference was noted in this respect between the two alloy ingots.

3. Differences between ingot 1 and ingot 2 (medium sponge hardness vs. low sponge hardness) were masked by the chemical inhomogeneity in ingot 2. Therefore, no definite conclusions as to the beneficial effects of low interstitial content can be made from this report.

4. Variations in mechanical properties of the alloy material due to forging technique or to rolling temperature are slight. There is some evidence in favor of hammer rather than press forging and of subtransus rather than supertransus rolling. However, rolling at a lower than average temperature (650° C.) was not only more difficult but also did not yield better mechanical properties. There is an indication that low interstitial content combined with rolling just below the transus results in good impact properties at temperatures above ambient.

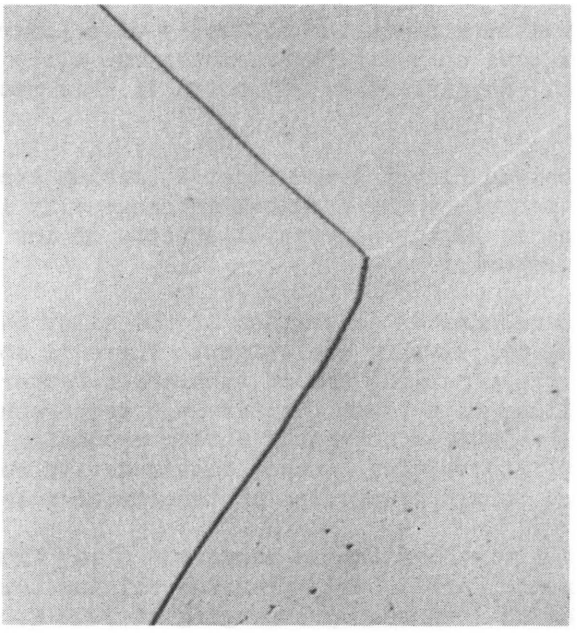
5. Water quenching of the titanium-manganese alloy from the rolling temperature causes embrittlement, particularly when the rolling temperature is above the transus. Softening can be restored by overaging at 700° F. or above; however, a good balance of mechanical properties and a stabilized condition obtain when the alloy is cooled slowly from the rolling temperature.

8/ Frost, P. D., Parris, W. M., Hirsch, L. L., Doig, J. R., and Schwartz, C M., Isothermal Transformation of Titanium-Manganese Alloys: Trans. ASM, vol. 46, 1954, pp. 1056-1074.



1500 X

Figure 24. - Microstructure of specimen from ingot 1 water-quenched from 950° C. and aged 24 hours at 427° C. Kellers etchant.



1500 X

Figure 25. - Microstructure of specimen from ingot 1 water-quenched from 950° C. Kellers etchant.

6. Transition-impact temperatures for the unalloyed material are lower than for the alloyed material, as would be expected.

7. The unalloyed material is soft and ductile and has remarkably high impact properties at room temperature and above. Anistropy apparently played a large part in determining the properties of this material.