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Metallographic and X-ray Diffraction Study of  
High Titanium-Iron Alloys

By  
Leonard E. Olds

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A thesis submitted to the Faculty and the Board  
of Trustees of the Colorado School of Mines in partial ful-  
fillment of the requirements for the degree of Master of  
Science in Metallurgical Engineering.

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## PART I

### INTRODUCTION

From the very beginning of the recent surge of interest in ductile titanium, the fact was realized that the greatest potential use for this so-called "Cinderella Metal" would be as a new base metal for alloys. Titanium holds one of the most favorable positions in the entire periodic table from the standpoint of alloying with other metals. Figure 1 shows that over 70 percent of all the common and some of the more rare metals have interatomic distances within 15 percent of that of titanium. According to Hume-Rothery's well-known alloying rule, we should expect therefore substitutional solid solution alloying of all these metals with titanium. In addition the smaller atomic-sized elements--carbon, hydrogen, oxygen, and nitrogen--could reasonably be expected to form interstitial alloys.

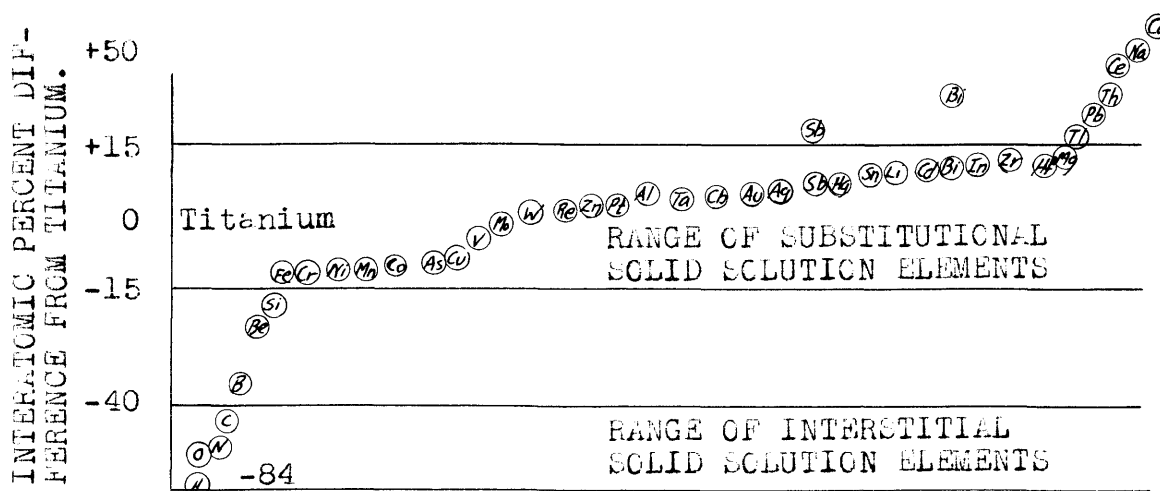


Figure 1. Chart taken from Gonser<sup>1/</sup> showing the possible alloying elements in relation to the atomic size of titanium.

PART IISUMMARY OF LITERATURE

Although titanium alloy research is rapidly increasing and considerable data have been published, the literature to date may be easily summarized. The early extensive work was that of Nielson<sup>2/</sup> on the interstitially alloying elements carbon and nitrogen. At the same time other investigators<sup>3/</sup> performed a limited amount of investigation on the following eighteen binary systems:

## Titanium with

Aluminum	Hydrogen	Nitrogen
Beryllium	Iron	Oxygen
Boron	Indium	Silicon
Carbon	Molybdenum	Tungsten
Chromium	Manganese	Vanadium
Cobalt	Nickel	Zirconium

The purpose of this investigation was to get a quick bird's-eye view of the most promising alloys. These systems plus additional investigation were summarized by Gonser one year later.<sup>4/</sup> His report covered the following systems:

## Titanium with

Aluminum	Columbium	Tantalum
Beryllium	Iron	Tin
Boron	Lead	Thorium
Carbon	Molybdenum	Tungsten
Chromium	Manganese	Vanadium
Cobalt	Nickel	Zirconium
Copper	Silicon	

The Bureau of Mines perhaps is doing more fundamental research upon individual binary systems than anyone else. It has already published an incomplete report on the

phase diagram of the titanium-nickel system.<sup>5/</sup> The most recent of all published investigations is that of Craighead and coworkers at the Battelle Memorial Institute.<sup>6/</sup> They have delved considerably deeper than Gonser into fourteen binary systems, sixteen ternary systems, and ten quaternary systems. These are:

Binary:

Titanium with

Beryllium	Iron	Silicon
Carbon	Lead	Silver
Boron	Molybdenum	Tin
Chromium	Manganese	Vanadium
Copper	Nickel	

Ternary:

Titanium and Carbon with

Copper	Iron	Silicon
Cobalt	Manganese	Vanadium
Chromium		

Titanium and Chromium with

Cobalt	Nickel	Tungsten
Molybdenum		

Titanium and Manganese with

Chromium	Silicon	Tungsten
Iron		

Titanium and Nickel with Chromium

Quaternary:

Titanium, Chromium, and Carbon with

Copper	Molybdenum	Nitrogen
Iron	Nickel	Vanadium
Manganese		

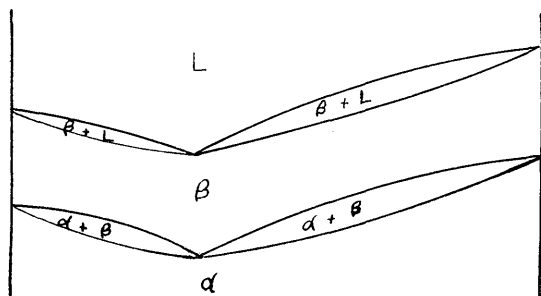
Titanium, Chromium, and Nitrogen with

Molybdenum	Nickel	Vanadium
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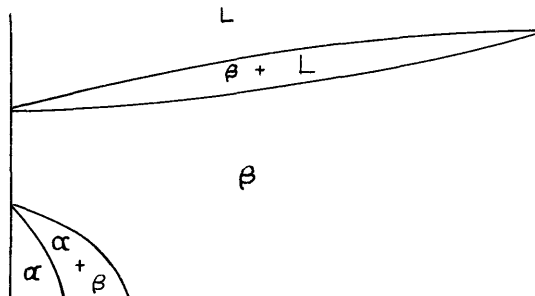
## PART III

ALLOYING CONSIDERATIONS

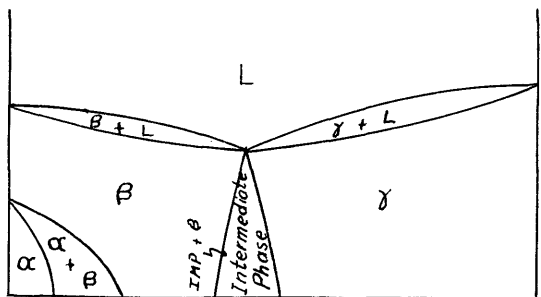
Pure titanium is a polymorphic metal. Alpha titanium has a hexagonal close-packed structure stable below 1625 F. Beta titanium has a body-centered cubic structure stable above this temperature. Generally speaking, alloying additions may affect the alpha and beta fields in one of the four following ways:<sup>4/</sup>



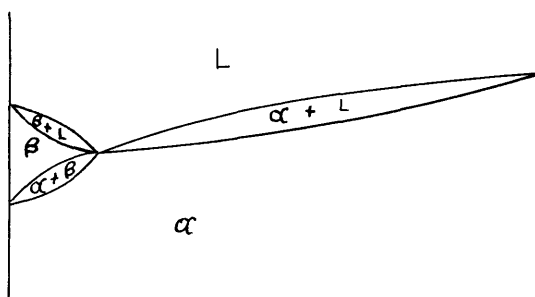
1. A continuous series of solid solutions form in both the alpha and beta fields.



2. The alpha field is restricted while forming a continuous series of beta solid solutions.



3. The alpha field is restricted and an intermediate compound forms.



4. The alpha field is extended while the beta field is restricted.

Figure 2. General effects of alloying on the alpha and beta titanium fields.

Only zirconium and hafnium, which have a favorable atom size, the same structure, and the same type of transition as titanium will alloy similar to 1. Molybdenum, tungsten, tantalum, and columbium, which have a body-centered cubic structure and a more favorable atom size than any other body-centered cubic metals, alloy similar to 2. Metals with less favorable combinations of crystal structure and atomic size such as iron, chromium, manganese, and nickel alloy similar to 3. Elements which alloy similar to 4 are usually those which alloy interstitially.

## PART IV

### THE TITANIUM-IRON SYSTEM

This paper deals with the metallographic and X-ray diffraction studies of four titanium-iron alloys. From the above considerations we might expect iron to form an eutectoid with high percentages of titanium and to have many similarities to the Bureau of Mines' Ti-Ni phase diagram.<sup>5/</sup> Craighead and coworkers substantiate this<sup>6/</sup> and report an approximate extent of alpha solubility at 1450 F of 0.3 percent. The results of their mechanical property tests show that iron gives an extensive increase in strength but with a considerable sacrifice in ductility. The most promising alloy was at 2.2 percent iron. It also exhibited age hardening characteristics. Figures 3, 4, and 5 are reproductions of their diagrams. The early work done by Larsen<sup>3/</sup> on titanium-iron showed that an addition of 10 percent iron reduced considerably the cold-working properties of the metal.

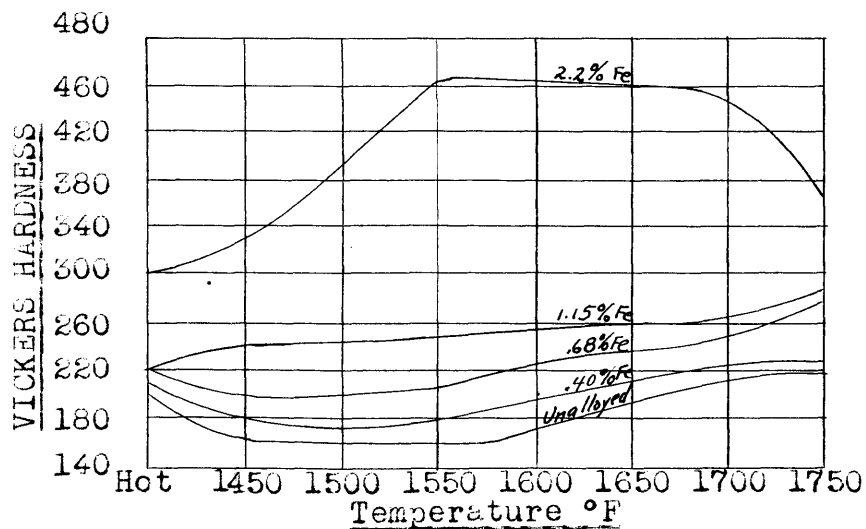


Figure 3. Effect of solution treatment on hardness.

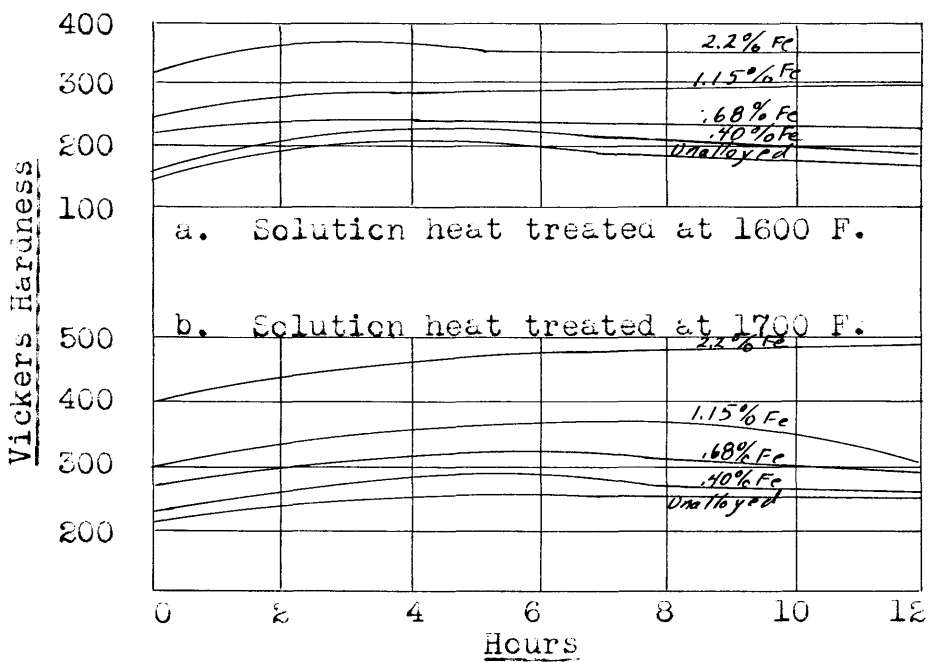


Figure 4. Effect of aging on hardness.

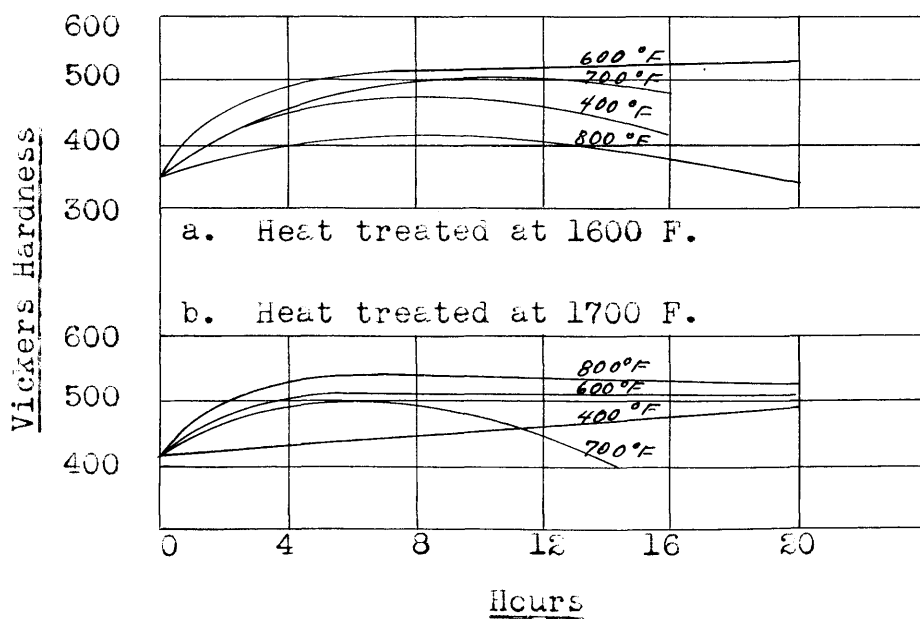


Figure 5. Aging curves for 2.2% iron.

PART VEXPERIMENTAL PROCEDURE

## 1. General:

The alloys investigated were pieces of broken tensile specimens containing 1 percent, 2.5 percent, 5 percent, and 10 percent iron additions to titanium. The alloys and a 5/8 inch rod of commercial titanium were supplied through the courtesy of the Allegheny Ludlum Steel Corporation. Sponge metal made by magnesium reduction and furnished either by the National Lead or by the Du Pont Companies was used as the source of titanium. The nominal analysis of such sponge metal and the ingots produced from it is shown in Table I.

	Sponge	Ingot
Titanium	99.5+%	98.8+%
Iron	.05 - .25%	.05 - .25%
Magnesium	.01 - .05%	less than .01%
Chlorine	.05 - .10%	less than .01%
Hydrogen	.006- .009%	.006- .009%
Nitrogen	.01 - .02%	.01 - .05%
Oxygen	.01 - .10%	.10 - .20%
Carbon	.01 - .05%	.2 - 1.0 %

Table I. Nominal analysis of sponge and ingot titanium.<sup>10/</sup>

The alloys were produced in one-half pound melts, arc-melted in a small 2 1/2-inch diameter furnace.<sup>12/</sup> The ingots were forged to one-half inch square bar at 1700 F. The alloys as received had been annealed after forging as follows:

	1%	iron	annealed	at	1700	F
	2.5%	"	"	"	1300	F
	5%	"	"	"	1300	F
	10%	"	"	"	1500	F

The titanium specimen was cut from a 5/8-inch centerless ground bar. This bar was produced by hot forging a seven-inch-diameter ingot in the temperature range of 1700-1500 F. The ingot was not cold worked at any time.

Micrographic examination of the polished alloys indicated that all of the alloys except the 1 percent iron had not reached their equilibrium state in the beta field by the above annealing. It was decided to heat treat the alloys as indicated:

#### Annealed

Alloy	Temp.	Time at Temp.	Method of Cooling
1% Fe	2000 F	3 hrs	Furnace cooled to room temperature.
2.5% Fe	1700 F	3 hrs	Furnace cooled to 400 F--Still air
5% Fe	1700 F	3 hrs	cooled to room temperature.
10% Fe	1700 F	3 hrs	" "

#### Quenched

Ti	1700 F	3 hrs	Water quenched to room temperature.
1% Fe	1700 F	3 hrs	"
2.5% Fe	1700 F	3 hrs	"
5% Fe	1700 F	3 hrs	"
10% Fe	1700 F	3 hrs	"

The titanium specimen was quenched for comparison with the quenched alloys.

## 2. Grinding and Polishing:

Pure titanium requires particular care in grinding and polishing to prevent the formation of mechanical twins. All specimens were mounted in bakelite and carefully ground on the belt grinder for at least one hour to remove surface scale. The grinding was finished on emery papers 1/0 through 3/0. Two methods of intermediate polishing were tried. By the first method 3/0 scratches were removed by using 600 mesh carborundum on a canvas cloth. Carborundum on a lead-foil lap on glass was used for the second method. The carborundum suspension was made by levigating with water twice and decanting after the suspension had settled for five minutes. The first decanting removed the +600 mesh material. The second eliminated the -600 mesh material. The levigated carborundum was introduced into the lead-lap by rubbing it in with a clean, flat metal disk. Polishing time averaged one minute. The lead-lap was found to be more satisfactory than the canvas cloth, although results often were erratic.

Final polishing of the titanium specimen was done in two steps: chromium oxide on silk for the first step and chromium oxide on Gamal cloth for the second. It was very difficult to remove the disturbed metal from the titanium sample. The tedious method of etch and polish gave fair results after many hours on the wheel.

Final polishing of the alloys was done by ordinary metallographic technique.

The etch used was:

- 1 part by volume 48% hydrofluoric acid
- 1 part by volume concentrated nitric acid
- 2 parts by volume glycerine.

### 3. X-ray Diffraction Procedure:

The X-ray diffraction work consists of a series of powder patterns of the alloys as received and back-reflection pictures of the alloys as received and as heat-treated. Preliminary work indicated the necessity of mounting a nickel filter between the specimen and film to prevent extraneous radiation from obscuring the pattern. This filter was attached to a very thin sheet of tracing paper by means of Kodak dry mounting tissue. The back-reflection filter required a double layer of nickel foil to prevent pin-hole light blotches.

The cylindrical camera was found to give a better powder pattern than the Debye. An attempt was made to check the radius of the camera by double-punching the film, but the back-reflection lines were too faint for reliable results. Hence the five-centimeter radius was used. All powder samples were -200 mesh filings, one part flour, mounted in Ducc cement, and rotated 360 degrees.

All back-reflection samples were polished and etched to remove disturbed metal. Three methods were experimented with in an attempt to get an accurate specimen-to-film distance.

1. Putting aluminum paint on surface of specimen.
2. Placing a thin nickel foil on surface of specimen.
3. Comparing a series of three pictures at the same distance, using the titanium sample for a standard.

The aluminum paint failed to give a pattern sufficiently well defined for measurement. The nickel foil had been cold rolled and gave lines that were too broad for accurate measurement. The third method was used since the titanium lines were sharp and well defined. The parametrical values used for titanium were those of Gonser:<sup>7/</sup>

$$\begin{aligned} a &= 2.946 \text{ kx units} \\ c &= 4.686 \text{ kx units} \end{aligned}$$

The mass absorption coefficient chart indicates that a molybdenum tube should be the best of the four tubes available. However, the short wave length of the molybdenum tube results in no lines appearing in the back-reflection region. For this reason a copper tube was used for the initial work and for all the back-reflection exposures. Because the copper tube failed to bring out lines of the second phase, exposures were also made using molybdenum and iron tubes. The molybdenum tube gave a well defined pattern but the iron tube failed to produce a pattern.

Tube	Mo	Cu	Fe	Cr	Density
Ti	23.7	204	377	603	4.53
1% Fe	23.8	205	374	598	4.53
2.5% Fe	24	207	369	591	4.59
5% Fe	24.4	210	362	578	4.64
10% Fe	25.5	221	347	554	4.78
Ti <sub>2</sub> Fe	29	247	267	426	5.74
Fe	38.3	324	73	115	7.85

Table II. Table of Mass Absorption Coefficients.

Measurements on the back-reflection pictures were carried to an accuracy of 0.001 centimeter by the means of a meter-bar, a bar widely used by the Geographic Survey for laying off map grids. Sine theta is independent of units. Its value depends only on the ratio of one distance to another. Therefore, any scale may be used in place of centimeters for measuring the X-ray pattern. Hence it is easy to obtain measurements with the above accuracy simply by carefully constructing a uniform scale on wood or stiff paper. An illustrative measurement is shown in Figure 6. The reading is 3.333 units.

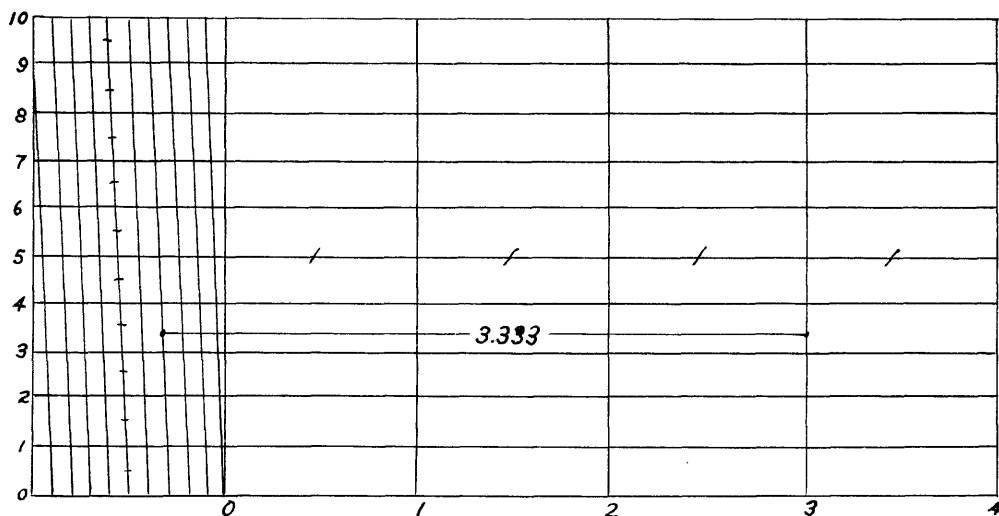


Figure 6. Basic concept of a meter-bar.

#### 4. Thermal Analysis:

Three heating and cooling curves were plotted for the 2.5 percent alloy in an attempt to locate the transformation temperatures. This work was performed rapidly using rough equipment. No indication of a transformation was observed.

## 5. Density Measurements:

Density measurements were made on the titanium and alloys using a beam balance measuring to four places. Measurements were corrected for buoyancy of the specimens and weights in air. The values are recorded in Table II.

PART VIRESULTS OF EXPERIMENTS

## 1. Microstructure of commercially pure titanium:

Figure 7 is a photomicrograph of a hot forged titanium bar. It was forged in the temperature range of from 1700 to 1500 F. The microstructure shows equiaxed grains of alpha titanium. The round islands are impurities.

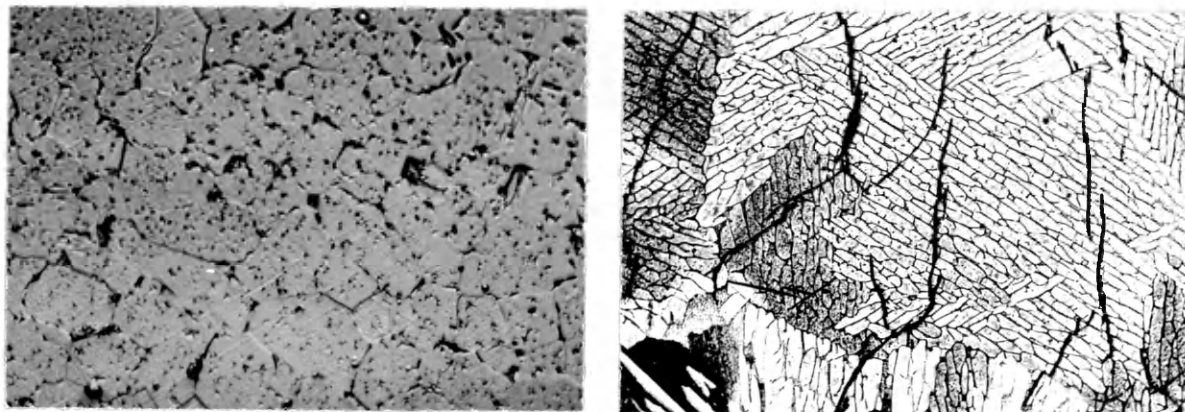


Figure 7. Hot forged titanium. 500x, 20 sec. etch. Figure 8. Quenched titanium. 50x, 20 sec. etch.

Figure 8 is a photomicrograph of a quenched titanium sample. The sample was heated for 3 hours at 1700 F then quenched to room temperature in water. The microstructure shows transformed grains of beta titanium. The small alpha grains have formed in a Widmanstaetten pattern. The heavy black lines are quench cracks.

## 2. Microstructure of Alloys as Received:

1 percent iron (Fig. 9-10): The micrographs are complex and difficult to interpret due to the fact that equilibrium conditions were not obtained in their primary heat-treatment. The 1 percent iron sample of Figure 9 would indicate a large proportion of a second phase. Later work showed that there is only a small amount of the second phase in this alloy. (See Fig. 20) The large transformed grains of Figure 9 indicate that the alloy had been annealed in the beta titanium field. Since the X-ray powder pattern gave only a pattern for alpha titanium (Fig. 17b) and the 1 percent annealed alloy showed only a slight amount of eutectoid, the large amount of slate-colored material in Figures 9 and 10 could only be alpha titanium with different etching characteristics. This slate-colored material resembles somewhat the alpha titanium of the pure titanium sample (Fig. 7) which transformed from the beta into equiaxed grains. The alpha titanium of the quenched titanium sample (Fig. 8) transformed from beta in a Widmanstaetten pattern with white alpha titanium plates. Since the annealing for Figures 9 and 10 was only for one-half an hour, it is possible that not all of the alpha titanium had time to change to the beta. If the cooling thereafter were fairly rapid, the beta would have transformed to alpha, giving a Widmanstaetten precipitate of white grains. This interpretation is valid only if equiaxed grains of alpha titanium would form in the 1 percent alloy.



Figure 9. 1% iron  
75x, 10 sec. etch.  
Annealed 1/2 hour  
at 1700 F.

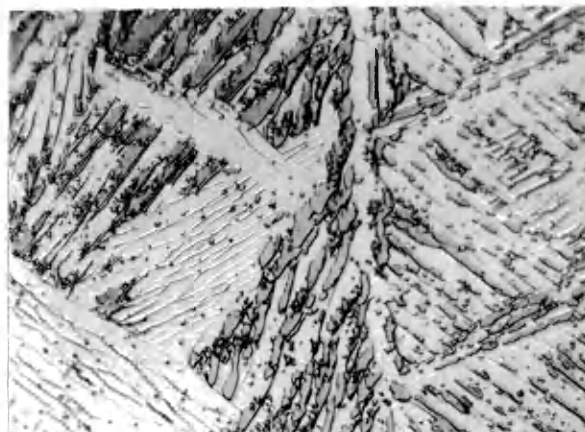


Figure 10. 1% iron  
800x, 10 sec. etch.



Figure 11. 2.5% iron  
75x, 10 sec. etch.  
Annealed 1/2 hour  
at 1300 F.



Figure 12. 2.5% iron  
270x, 10 sec. etch.

2.5 percent and 5 percent iron (Fig. 11-14):

These alloys were annealed at 1300 F. The structures are interpreted in the light of additional heat treatment as a eutectoid type of transformation in which the specimen was held for a short time just above the eutectoid line. The structures are hard to interpret because of the probable presence of untransformed alpha and eutectoid plus transformed and reprecipitated alpha and eutectoid.

10 percent iron (Fig. 15-16): The 10 percent alloy differs considerably from the others. It had been annealed also for one-half hour but at 1500 F. This structure shows large equiaxed grains throughout which are distributed small spheroidized particles. Figure 15 shows these spheroids in forging bands. This banding demonstrates the fact that the alloys did not reach equilibrium in the beta field in one-half hour at temperature. The equiaxed grains are alpha titanium which has lost its Widmanstaetten characteristics probably because of the effect of higher alloy content. Although these grains do not appear to be as dark as the gray areas in Figure 9, they are definitely not white. The spheroids are the second phase which spheroidized at the annealing temperature.

### 3. X-ray Diffraction Powder Patterns:

The powder patterns for titanium and the four alloys are shown in Figure 17. The first appearance of a second phase line is in the 5 percent alloy. (Fig. 17d) The 10 percent alloy showed a completely different pattern



Figure 13. 5% iron  
75x, 10 sec. etch.  
Annealed 1/2 hour  
at 1300 F.



Figure 14. 5% iron  
270x, 10 sec. etch.

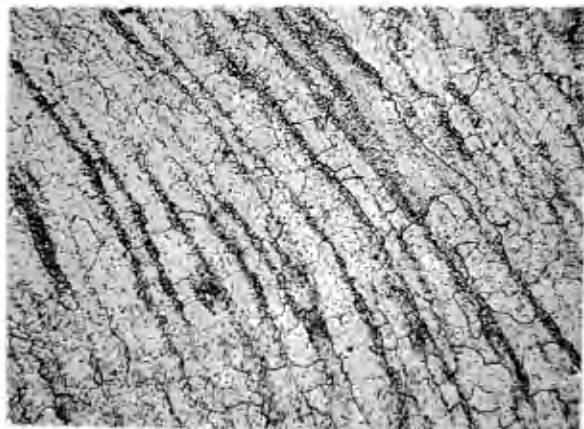


Figure 15. 10% iron  
50x, 10 sec. etch.  
Annealed 1/2 hour  
at 1500 F.

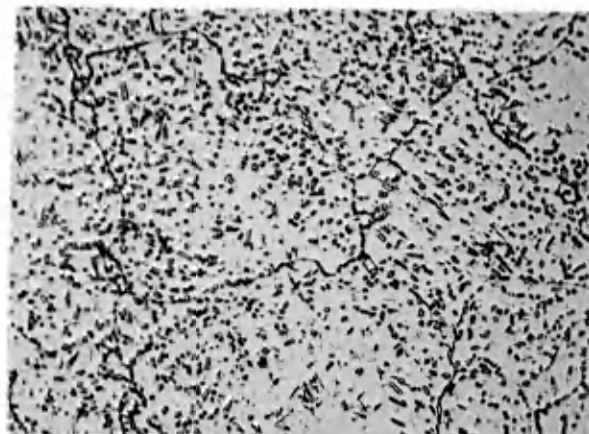


Figure 16. 10% iron  
250x, 10 sec. etch.

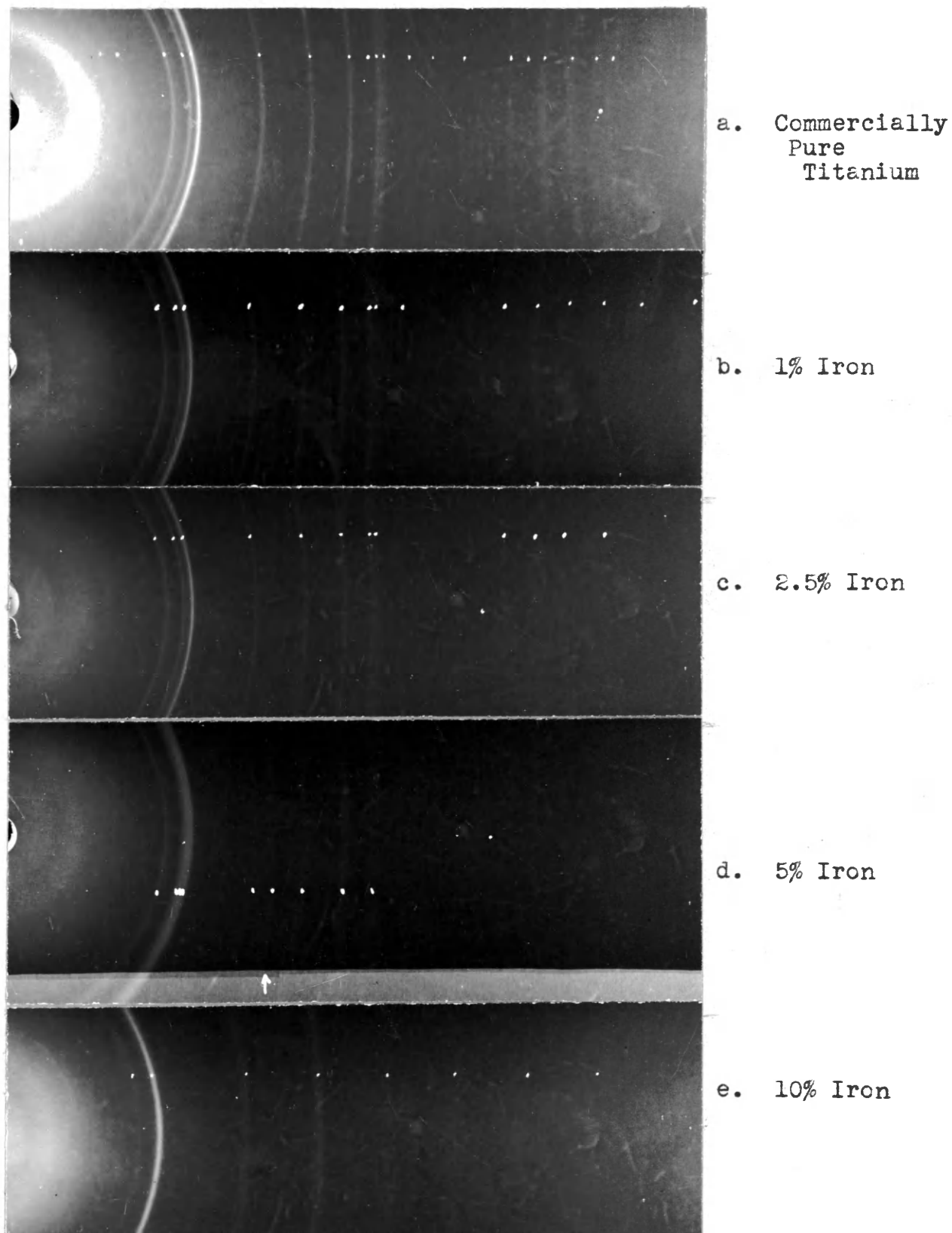


Figure 17. Powder Patterns for Iron-Titanium Alloys. Copper Target.

with only the 100, 103, and 105 lines of the alpha phase appearing. Duwez and Taylor believe that the first intermediate phase on the titanium side of the iron-titanium system is  $Ti_2Fe$ . According to their investigations<sup>8/</sup>  $Ti_2Fe$  has a face-centered cubic structure, a parameter of 11.305 kx, and 96 atoms to the unit cell. Using this parametrical value to calculate theoretical interplanar spacings, the d values of the 10 percent alloy powder pattern were checked within experimental error. (See pages 44-45)

The failure of second-phase lines to appear in the 1 percent alloy was quite disconcerting since the 1 percent iron micrograph exhibits relatively large amounts of the second phase. Even a 24-hour exposure on the 2.5 percent alloy showed no new lines appearing. (Figure 18)

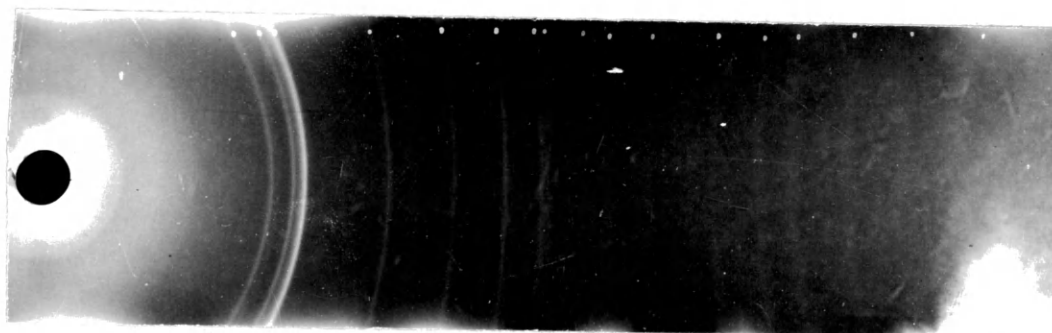


Figure 18: 2.5% iron. 24 hour exposure.  
Copper target.

Titanium-iron alloys have very high absorption coefficients which make them difficult to analyze by X-ray diffraction. Four X-ray tubes were available for use: molybdenum, copper, iron, and chromium. Of these, copper,

iron, and chromium have very high absorption coefficients. However, chromium and iron have lower coefficients for  $Ti_2Fe$  than for titanium. Molybdenum is frowned upon for use with metals because of its low wave length. However, it has the most favorable absorption coefficients. Exposures were made using the molybdenum tube on the 2.5 percent and the 5 percent alloys. (Fig. 19) Half of the lines in the 2.5 percent pattern may be assigned  $Ti_2Fe$  hkl values. (See pages 44-45)

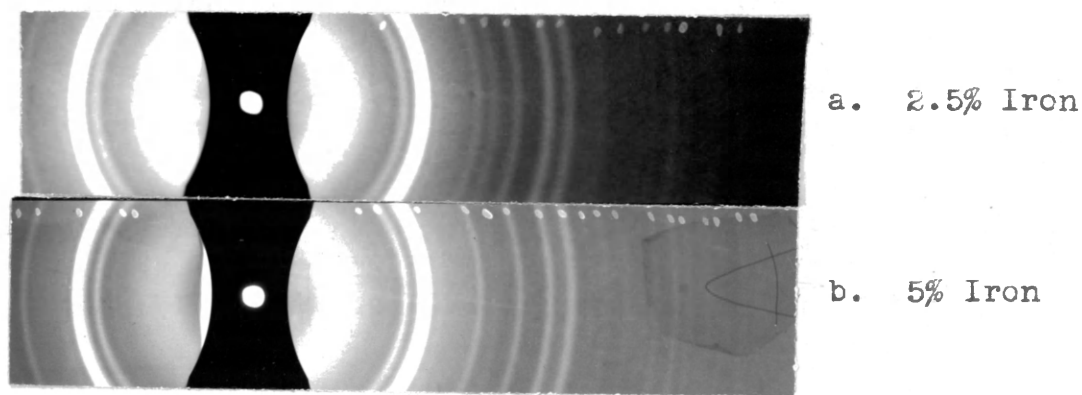


Figure 19. Powder Patterns using Molybdenum Tube.

#### 4. Microstructure of Heat Treated Alloys:

##### a. Annealed:

1 percent iron (Fig. 20-23): The second annealing of the alloys confirmed beyond doubt the fact that there is a eutectoidal transformation in the high titanium-iron system. Figure 20 exhibits the grain growth that has taken place in the 1 percent alloy as a result of a three-hour anneal at 2000 F. The microstructure shows a large beta grain size with thick plates of alpha. A fairly coarse eutectoid of

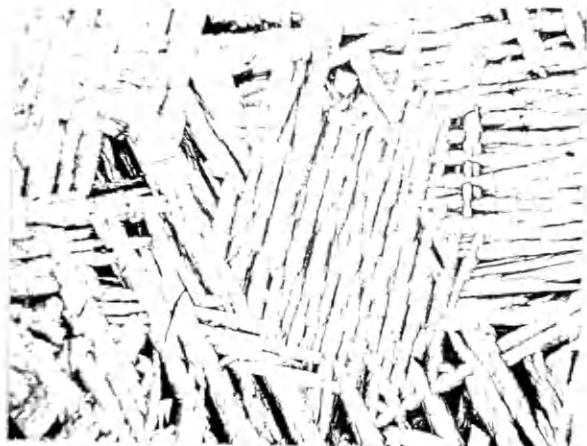


Figure 20. 1% iron  
20x, 20 sec. etch.  
Annealed 3 hours  
at 2000 F.



Figure 21. 1% iron  
280x, 20 sec. etch.

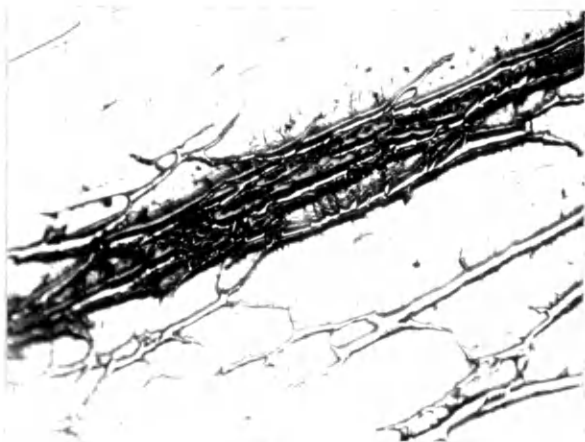


Figure 22. 1% iron  
180x, 20 sec. etch.

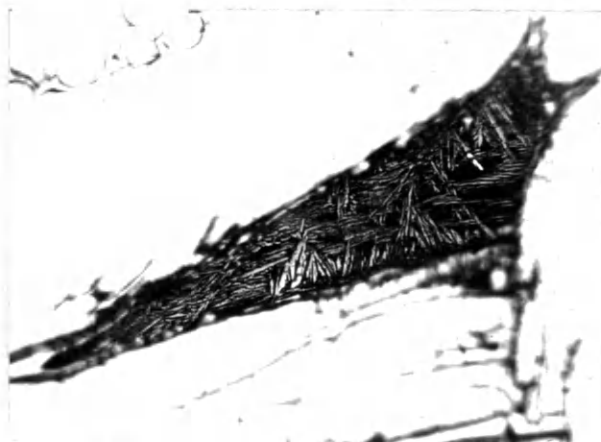


Figure 23. 1% iron  
180x, 20 sec. etch.

alpha and  $Ti_2Fe$  solidified last between the alpha plates and in other remaining beta areas. The eutectoid is coarse because slow cooling allowed considerable diffusion. The  $Ti_2Fe$  was heavily overetched in all but the 10 percent iron photomicrographs. Figure 23 shows that even the eutectoid in some of the larger areas tends to precipitate in a Widmanstaetten fashion.

2.5 percent iron (Fig. 24-27): This alloy shows a decrease in the amount of alpha and an increase in the amount of the eutectoid. Figure 27 is interesting in that it shows the growth of the eutectoid into the remaining beta areas by end growth of the plates.

5 percent iron (Fig. 28-31): This also shows a further decrease in the alpha and further increase in eutectoid.

10 percent iron (Fig. 32-38): At first the 10 percent alloy was thought to still be on the titanium side of the eutectoid. Longer etching finally brought out the darker etching characteristics of the  $Ti_2Fe$  phase. The microstructure generally shows  $Ti_2Fe$  grains in the grain boundaries surrounding eutectoidal areas. The eutectoid is fine in some areas and divorced in others.

Figures 38 and 39 indicate the hardness of the  $Ti_2Fe$  phase. These pictures are taken of the same area: Figure 38 with the microscope focused on the  $Ti_2Fe$  plate in the center of the picture and Figure 39 with the microscope racked down to focus on the impurities in the alpha phase. This shows the intermetallic compound to be harder,



Figure 24. 2.5% iron  
20x, 15 sec. etch.  
Annealed 3 hours  
at 1700 F.

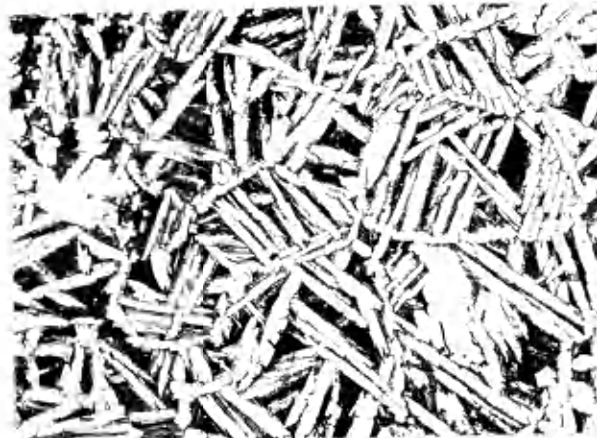


Figure 25. 2.5% iron  
50x, 15 sec. etch.



Figure 26. 2.5% iron  
180x, 15 sec. etch.



Figure 27. 2.5% iron  
600x, 15 sec. etch.

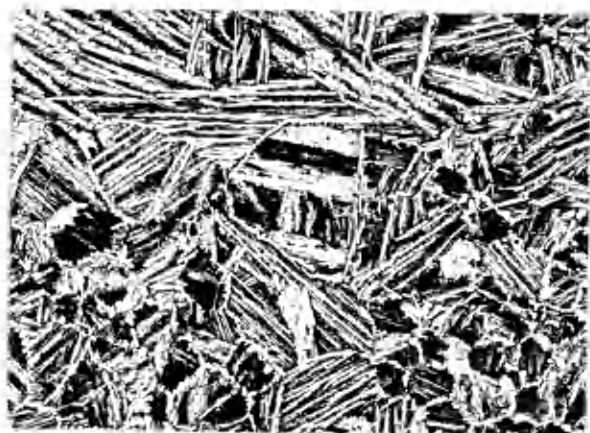


Figure 28. 5% iron  
60x, 15 sec. etch.  
Annealed for 3 hours  
at 1700 F.



Figure 29. 5% iron  
130x, 15 sec. etch.

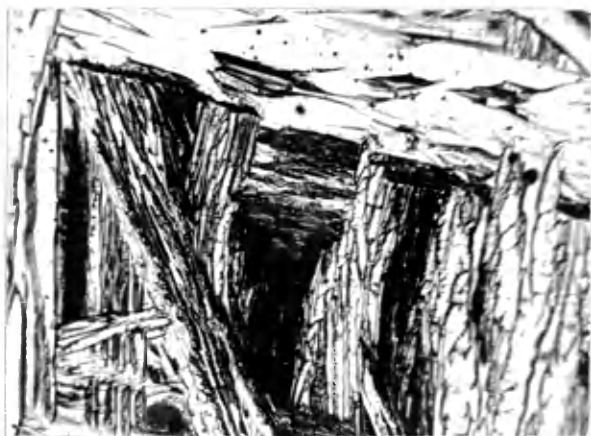


Figure 30. 5% iron  
300 x, 15 sec. etch.



Figure 31. 5% iron  
850x, 15 sec. etch.



Figure 32. 10% iron  
60x, 20 sec. etch.  
Annealed 3 hours  
at 1700 F.



Figure 33. 10% iron  
60x, 40 sec. etch.



Figure 34. 10% iron  
80x, 20 sec. etch.



Figure 35. 10% iron  
100x, 40 sec. etch.

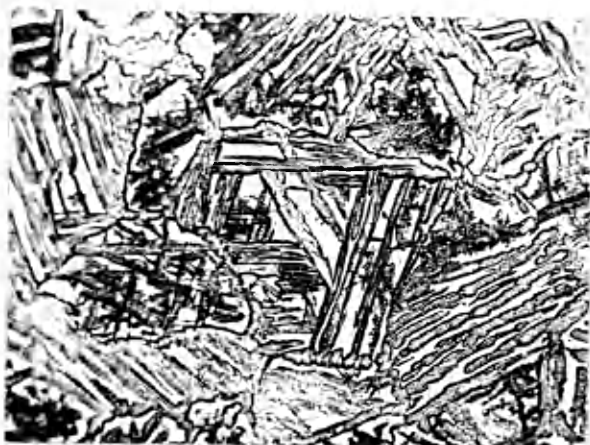


Figure 36. 10% iron  
300x, 20 sec. etch.  
Annealed 1700 F.



Figure 37. 10% iron  
130x, 20 sec. etch.



Figure 38. 10% iron  
240x, 40 sec. etch.



Figure 39. 10% iron  
240x, 40 sec. etch.

with the implication that it is also brittle. This brittleness would explain the ragged appearance of many of the  $Ti_2Fe$  plates. In polishing it is likely that instead of being worn away, some  $Ti_2Fe$  areas broke away.

b. Quenched:

1 percent iron (Fig. 40-42): Again the beautiful Widmanstaetten characteristics of titanium and its alloys is prominent. The alpha plates are more thin and jagged. At first the white areas in the center of the transformed beta grains were thought to be retained beta. However deeper etching revealed their fine eutectoidal nature.

2.5 percent iron (Fig. 43-45): A structure similar to that of the 1 percent alloy has been developed here. The beta grain size is smaller, and the alpha plates are larger. Figures 43 and 44 contrast light and dark etching.

5 percent iron (Fig. 46-48): The result of greater alloy content has affected the formation of the eutectoid. In the transformation of the beta, alpha is still precipitated at the grain boundaries and on Widmanstaetten planes. The eutectoid although still very fine has coarsened.

10 percent iron (Fig. 49-50): In view of the X-ray diffraction work, the quenched 10 percent iron microstructure is interpreted as being a two-phase structure with only a slight amount of eutectoid present.  $Ti_2Fe$  grains surround alpha areas. Lack of a Widmanstaetten structure through these alpha areas is disturbing. Some of the areas do have a Widmanstaetten structure. It is absent from other

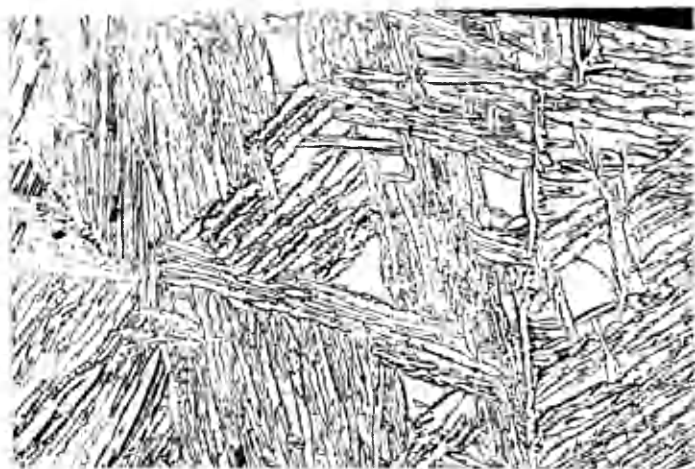


Figure 40. 1% iron  
50x, 5 sec. etch.  
Quenched from  
1700 F.



Figure 41. 1% iron  
50x, 30 sec. etch.



Figure 42. 1% iron  
200x, 30 sec. etch.

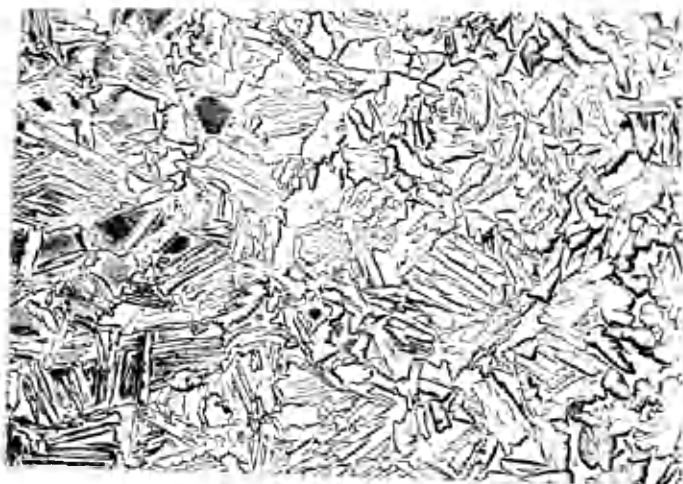


Figure 43. 2.5% iron  
50x, 10 sec. etch.  
Quenched from  
1700 F.

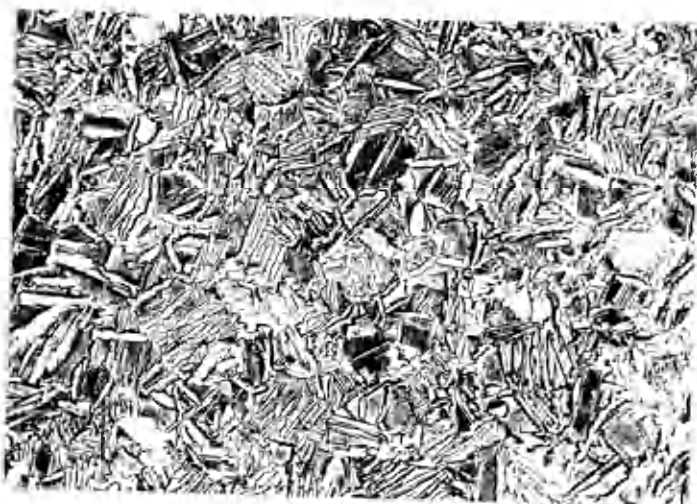


Figure 44. 2.5% iron  
50x, 30 sec. etch.



Figure 45. 2.5% iron  
300x, 30 sec. etch.



Figure 46. 5% iron  
50x, 45 sec. etch.  
Quenched from  
1700 F.



Figure 47. 5% iron  
180x, 45 sec. etch.



Figure 48. 5% iron  
200x, 45 sec. etch.



Figure 49. 10% iron  
50x, 30 sec. etch.  
Quenched from  
1700 F.



Figure 50. 10% iron  
100x, 30 sec. etch.

areas probably because of the effect of the high alloy content.

#### 5. X-ray Diffraction Back-Reflection Patterns:

The back-reflection work was done in order to provide an accurate measurement of lattice distortions. The following back-reflection d-values for alpha titanium, beta titanium, and  $Ti_2Fe$  were computed from their respective parameters: (All measurements are in kx units.)

Parameters:	<u>kx units</u>	<u>Source</u>
alpha Ti	a = 2.946, c = 4.686	Gonser <sup>7/</sup>
beta Ti	a = 3.32	Barrett <sup>8/</sup>
$Ti_2Fe$	a = 11.305	Duwez <sup>8/</sup>

#### Computed d-values:

	d-value	hkl	d-value	hkl	d-value	hkl
alpha Ti	0.8205	213	0.7994	320	0.7810	006
beta Ti	0.8300	400			0.7825	411
$Ti_2Fe$	0.8267	599	0.8159	888	0.7783	799

The following averaged experimental d-values were obtained:

#### 1 percent, 2.5 percent, and 5 percent alloys:

line	as received	annealed	quenched	index assigned	theoretical d-value
1	0.8208	0.8214	0.8220	213 alpha Ti	0.8205
2	0.7997	0.8003	0.8006	302 alpha Ti	0.7994
3	0.7813	0.7820	0.7830	006 alpha Ti	0.7783

#### 10 percent alloy:

1	no pattern	0.8236	0.8234	213 alpha Ti	0.8205
2	"	0.8015	0.8019	302 alpha Ti	0.7994

This shows that the greatest distortion of the parameters is in the quenched samples. The least is in the as received series. No  $Ti_2Fe$  lines appeared because of the higher absorption coefficient for this phase.

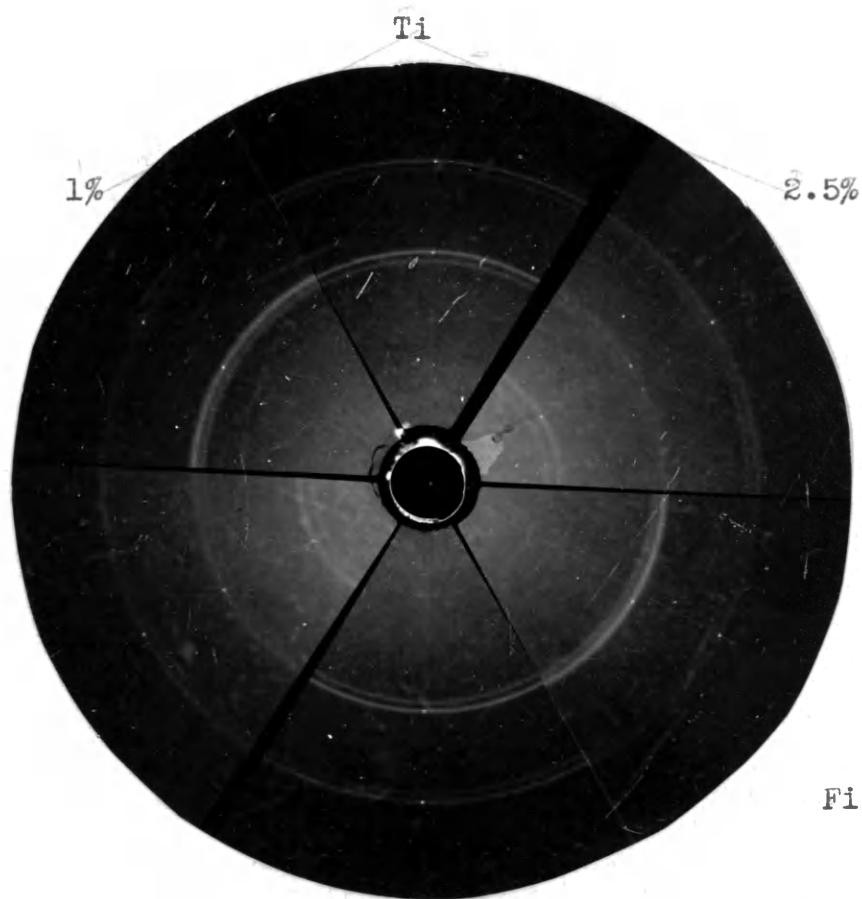


Figure 51. As-received alloys. Copper radiation.

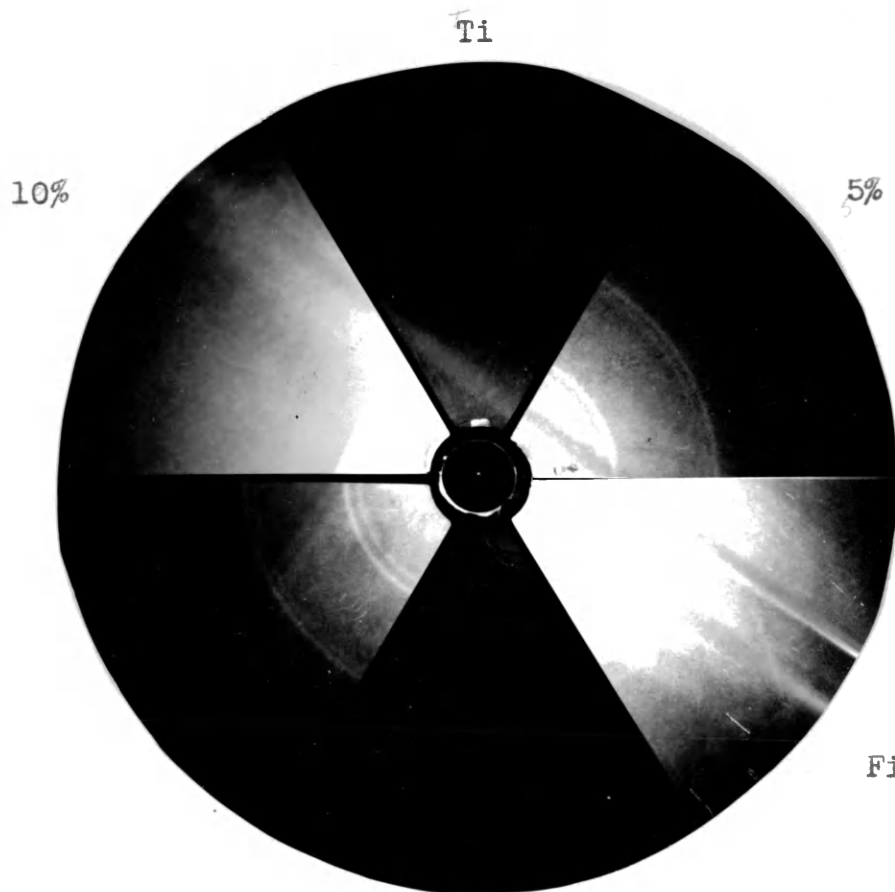


Figure 52. As-received alloys. Copper radiation.

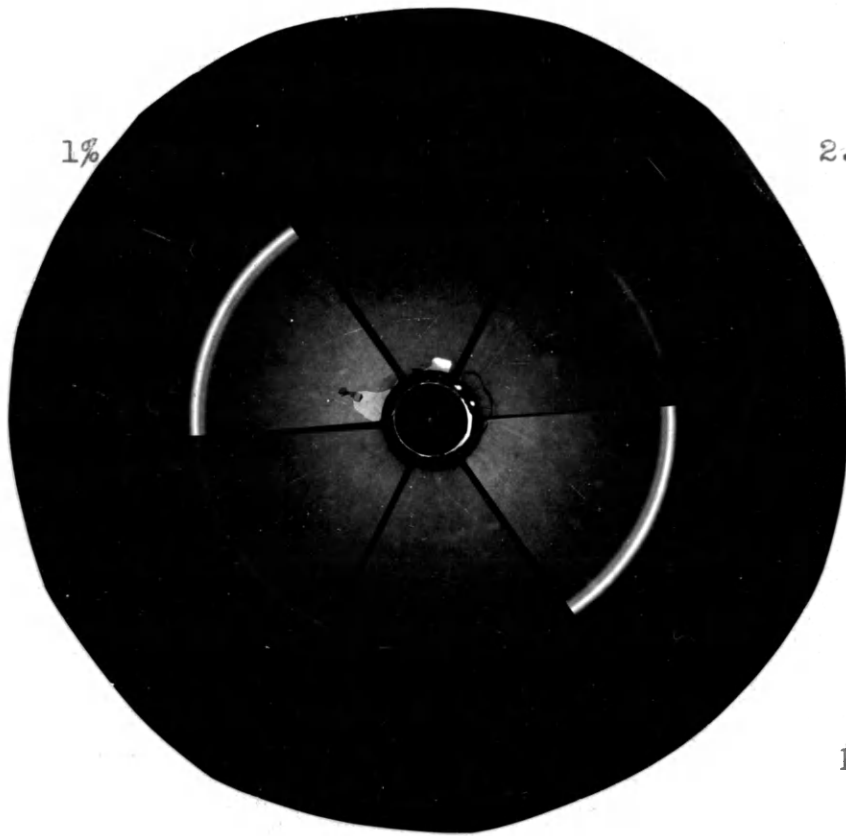


Figure 53. Annealed alloys. Copper radiation.

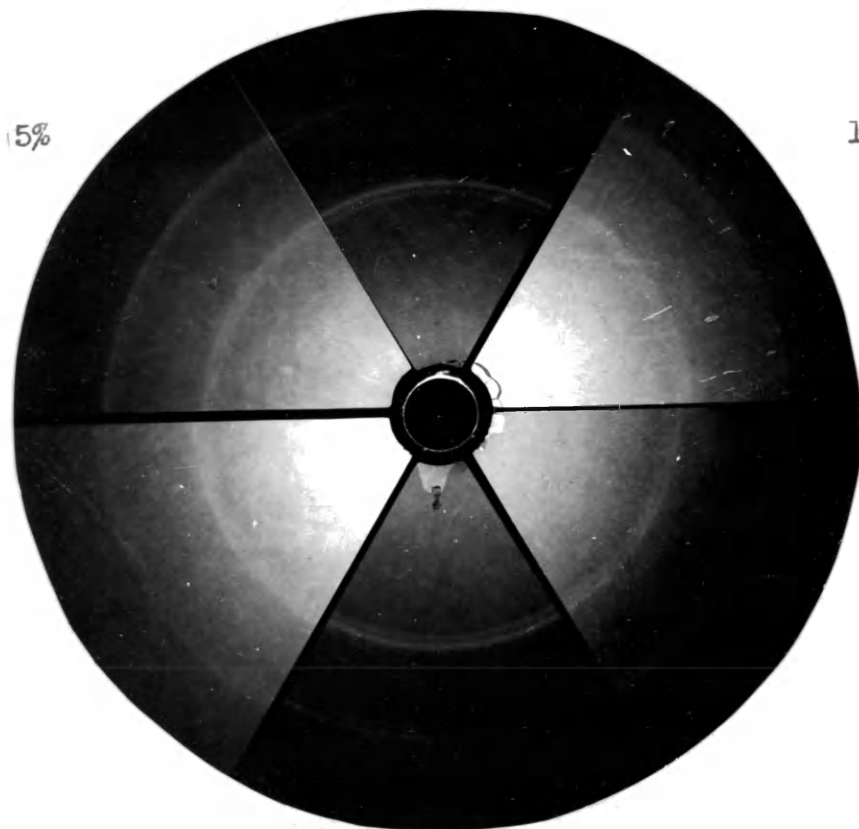


Figure 54. Annealed alloys. Copper radiation.

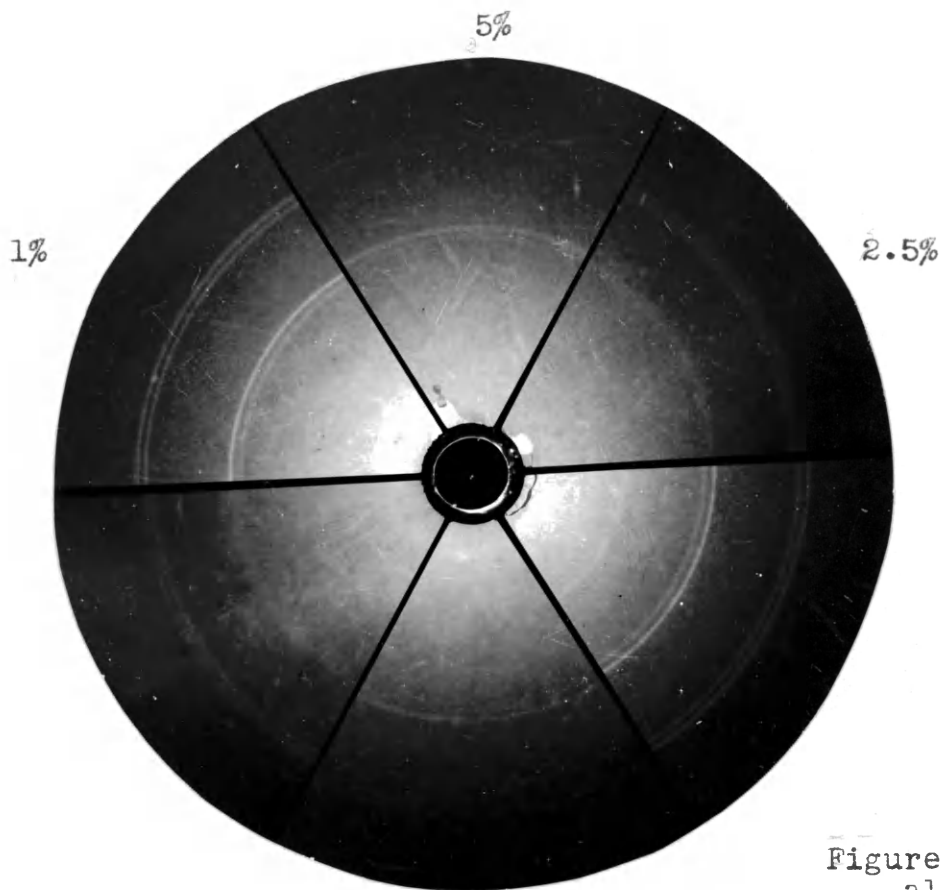


Figure 55. Quenched alloys. Copper radiation.

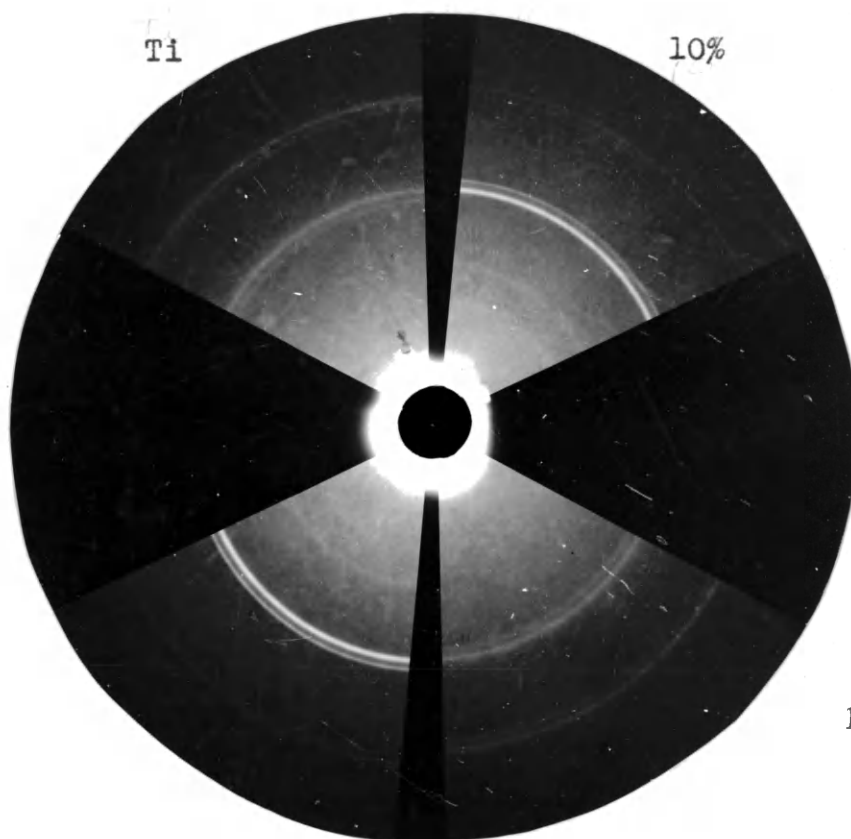


Figure 56. Quenched alloys. Copper radiation.

A 24-hour exposure of the 1 percent annealed sample brings into view the line from the alpha 006 plane. (Fig. 57)

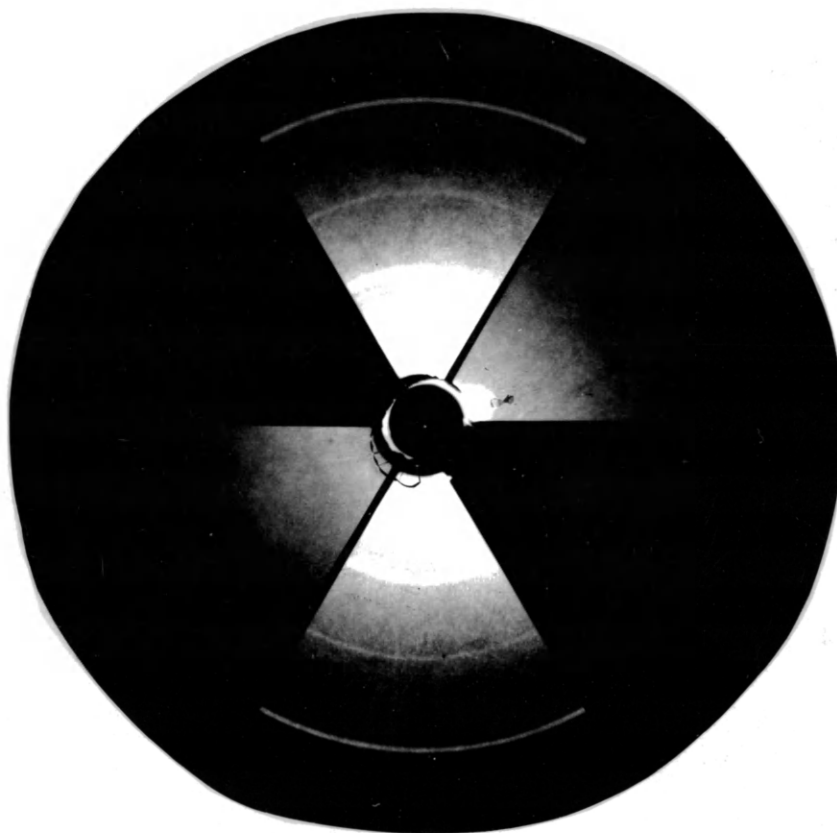


Figure 57. 24-hour exposure of 1% iron. Copper target.

## 6. Mechanical Properties:

## a. Tensile:

The Allegheny Ludlum Steel Corporation performed the tensile tests on the alloys.<sup>11/</sup> The titanium value is taken from the Office of Naval Research Symposium on Titanium.<sup>9/</sup>

Tensile Strength of Titanium-Iron Alloys

		Ductility
Titanium	72,000 psi	
1% iron	81,500	25%
2.5% iron	111,000	23%
5% iron	138,000	21%
10% iron	190,800	6%

## b. Hardness:

Hardness tests were made in the Colorado School of Mines laboratory using the Rockwell C scale.

	As-received	Annealed	Quenched
Titanium	10 Rc	--	20
1% iron	15	29	36
2.5% iron	27	31	36
5% iron	34	37	45
10% iron	34	44	41

On the basis of these data the 5 percent alloy shows the most promise from the standpoint of strength, ductility, and hardness. However, Craighead and co-workers<sup>6/</sup> reported more favorable aging characteristics in a 2.2 percent iron alloy. They also report a much higher hardness after giving this alloy a solution heat treatment at 1550 F.

PART VIICONCLUSIONS

From the experimental work of this thesis it is evident that a simple eutectoid is the only transformation occurring below 2000 F in the range between zero and 36 percent iron. Figure 58 is the approximate equilibrium diagram for this range.

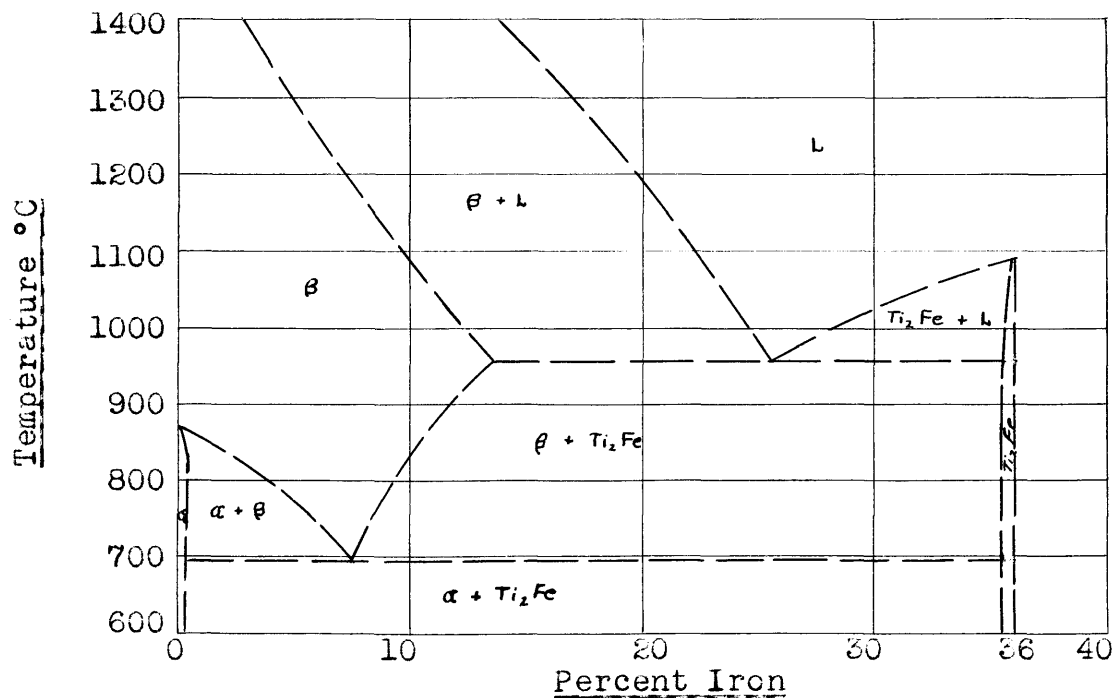


Figure 58. Approximate Titanium-Iron Phase Diagram in the Range Between Zero and 36% Iron.

The eutectoid has a composition lying between 5 and 10 percent iron. Using a value of 7.5 percent for this composition, the eutectoid is composed of approximately 25 percent  $Ti_2Fe$  and 75 percent alpha titanium. The  $Ti_2Fe$  is hard, brittle, and probably very strong. Alpha titanium is soft and ductile.

Although the work performed in this paper of necessity was limited, enough has been done to indicate that the Titanium-Iron system is quite promising and well worth more study.

PART VIIISUGGESTIONS OF POSSIBLE HEAT TREATMENT TO IMPROVE  
MECHANICAL PROPERTIES

Undoubtedly many desirable heat treatments may be obtained in this alloy series by taking advantage of the eutectoidal transformations. Apparently the transformations of beta titanium do not include a martensitic type of reaction. Hence it probably will not be possible to obtain the spectacular effects of heat-treated steels. However, the eutectoid undoubtedly will be changed in degree of fineness by varying the rate of cooling. Time might well be profitably spent in studying isothermal transformations.

Probably the best heat treatment for the 1, 2.5, and 5 percent alloys from the standpoint of improving mechanical properties would be as follows: Hold the alloy in the furnace at a temperature about 100 degrees higher than the alpha-beta transformation line (about 1700 F for 1 percent and 1500 F for 5 percent.) Heat it for a sufficient length of time to allow the object to become entirely beta. Remove it and cool it at an accelerated rate to yield a fine eutectoid. Final tempering may not be necessary since no brittle martensitic product is formed.

There is little hope for improving the properties of the 10 percent alloy since even with the  $Ti_2Fe$  spheroidized the ductility was only 6 percent.

Perhaps the most promising alloy is one with a eutectoidal composition (probably around 7.5 percent.) Its density would be about 4.7. Such an alloy would be light, strong, and ductile.

COMPARISON OF EXPERIMENTAL d VALUES WITH CALCULATED d VALUES:

a. Powder patterns:

Tl	<u>d values (experimental)</u>				<u>d values (calculated)</u>				
	<u>copper tube</u>		<u>moly. tube</u>		alpha Tl	hkl	Ti <sub>2</sub> Fe hkl	beta Tl	hkl
	1%	2.5%	5%	10%					
2.560	2.556	2.556	2.556	2.554	2.551	100	2.528	420	
2.356	2.348	2.344	2.344		2.343	002	2.307	422	2.348 110
2.250	2.247	2.244	<u>2.243</u>	<u>2.310*</u>	2.241	101	2.175	511	
							1.998	440	
					<u>1.997</u>		1.911	531	
							1.884	620	
							1.788	600	
1.735	1.731	1.726	1.728		1.726	102	1.724	533	1.660 200
							1.675	622	
							1.631	444	
							1.583	711	
							1.568	640	
							1.511	642	
1.481	1.478	1.472	1.475		1.473	110	1.472	731	
							1.392	464	
							1.388	373	
1.336	1.334	1.331	1.335	1.332	1.332	103	1.305	280	1.321 211
1.278	1.277				1.276	200	1.297	282	
1.252	1.251	1.249	1.247		1.247	112	1.241	175	
1.234	1.234	1.229			1.231	201	1.234	284	
1.174	1.173				1.171	004	1.185	193	1.174 220
							1.154	484	
1.123	1.122				1.120	202	1.136	575	
							1.109	286	
							1.093	195	
1.066	1.066				1.065	104	1.088	666	1.050 310

\*All underlined entries are assigned the corresponding Ti<sub>2</sub>Fe hkl index.

Ti	1%	2.5%	5%	10%	2.5%	5%	alpha Ti hkl	Ti <sub>2</sub> Fe hkl	beta Ti hkl
.989	.989	.987	<u>1.022</u>	<u>1.023</u>	<u>1.023</u>	<u>1.026</u>	.988 203 .964 210 .945 211 .917 114 .892 212 .880 105 .863 204 .851 300 .821 213 .799 302 .781 006 .755 205 .747 106	1.048 486 1.019 577 .988 595 .932 777 .908 597 .886 199 .883 688 .865 399 .845 797 .827 599 .816 888 .778 799	.954 222 .887 321 .830 400 .781 411 .742 420
.946	.946	.945	<u>.934</u>	<u>.936</u>	<u>.940</u>				
.916	.917	.916							
.895	.894								
.882	.882		.880	.879	.879				
.821	.821	.820							
.801				.804					
				.750	.751				

b. Back reflection patterns:

Annealed samples:

	<u>1%, 2.5%, &amp; 5%</u>	10%
	.8505 300	
	.8205 213	
	.7994 302	
	.7810 006	
	.7553 205	
		.845 797
		.8267 599
		.8159 888
		.7805 411
		.742 420
	.8214	.8236
	.8003	.8015
	.7820	

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