CANADA

A STUDY OF THE CONSTITUTION OF THE TITANIUM-RICH CORNER OF THE TITANIUM-ALUMINUM-MOLYBDENUM SYSTEM

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PHYSICAL METALLURGY DIVISION

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A STUDY OF THE CONSTITUTION OF THE TITANIUM-RICH CORNER OF THE TITANIUM-ALUMINUM-MOLYBDENUM SYSTEM

by

A. J. Williams*

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ABSTRACT

The constitution of the titanium-rich corner of the titanium-aluminum-molybdenum system, based on a four-hour annealing time, has been investigated. Vacuum heat-treatment and metallography have been used to determine the β-transus on nine constant titanium sections of this system in the composition range of 0-25 wt % molybdenum and 0-15 wt % aluminum. Using these sections, together with published data on the titanium-aluminum and titanium-molybdenum binary diagrams, isothermal sections at 850, 900, 950 and 990°C (1562, 1652, 1742 and 1814°F) have been developed and tie-lines have been determined for these sections. Four discontinuities in the β-transus surface have been discovered. These discontinuities appear to indicate the presence of four three-phase fields which are contiguous with two-phase fields in the titanium-aluminum binary diagram discovered by several other workers.

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ÉTUDE DE LA CONSTITUTION DES ALLIAGES RICHES EN TITANE DU SYSTÈME TITANE-ALUMINIUM-MOLYBDÈNE

A. J. Williams*

RÉSUMÉ

L'auteur a fait des recherches sur la constitution des alliages riches en titane du système titane-aluminium-molybdène après un recuit de quatre heures. L'étude métallographique après traitement thermique sous vide a déterminé les limites du domaine de la phase β sur neuf coupes à teneur invariable en titane pour des compositions pondérales allant de zéro à 25% de molybdène et de zéro à 15% d'aluminium. En employant ces coupes ainsi que les données publiées provenant des diagrammes binaires titane-aluminium et titane-molybdène, on a pu tracer les isothermes pour des températures de 850, 900, 950, et 990°C (1562, 1652, 1742, et 1814°F), et des connodes ont été déterminées sur ces isothermes. On a trouvé dans la surface du domaine de la phase β, quatre discontinuités qui semblent indiquer la présence de quatre domaines à trois phases contigus à des domaines à deux phases découverts par plusieurs autres chercheurs dans le diagramme binaire titane-aluminium.

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Ce rapport est une étude dont l'auteur a donné lecture au troisième congrès annuel des métallurgistes, Institut canadien des Mines et de la Métallurgie, le 10 septembre 1964, à Montréal, Québec.

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INTRODUCTION

In the course of the examination of a proposed system for the design of heat-treatable titanium alloys, published in 1958[1], the writer found it necessary to develop a constitution diagram of the titanium-rich corner of the titanium-aluminum-molybdenum system. Discussion by Ence and Farrar[2] of the diagram developed at that time made it evident that further work was being done on the titanium-aluminum system and showed that there were at that time insufficient data in the literature upon which to base a valid interpretation of the experimental results. The results of further investigations of the titanium-aluminum and titanium-aluminum-vanadium systems[3,4,5,6,7,8], although still the subject of some controversy, have provided a firmer basis for a fresh approach to the redetermination of the titanium-aluminum-molybdenum constitution diagram.

Briefly, the proposed design system involved the choice of the optimum β composition along the trace of the β-transus surface at the proposed solution temperature for the alloy, and the addition to this of the proportion of α necessary to achieve the desired properties after ageing of the β. The elements of constitution most necessary for such choices to be made are (a) an accurately determined β-transus surface and (b) ternary tie-lines in the two-phase region; both of these must be in the temperature range proposed for solution treatments.

It was the purpose of the present work, therefore, to define the contours of the β surface between 75% and 100% titanium*, and to develop several isothermal sections, complete with ternary tie-lines, between 850°C and 1000°C (1562°F and 1832°F), the probable temperature range for solution treatment of these alloys. In order to relate these

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*All compositions are expressed as per cent by weight.

isothermals to practical solution treatment times, the constitution was determined on the basis of a four-hour anneal rather than true equilibrium.

EXPERIMENTAL

Alloy Preparation

All alloys except those used to determine the 75% titanium cross-section were made from high-purity titanium electrolytic crystals which had an average as-melted hardness of approximately 75 BHN (1000 kg load). This material was manufactured and supplied by the United States Bureau of Mines. The 75% titanium alloys were made from iodide titanium crystal bar. Alloy additions were 99.9+% pure aluminum and high-purity sintered molybdenum powder.

The alloys, which weighed 15 to 20 g, were melted in a cold-hearth tungsten-electrode arc furnace under 400 mm pressure of argon. Ingots were homogenized by remelting several times in the V-shaped bottom of the hearth after the method of Suiter[9].

All ingots which gained weight or lost more than 10 mg were discarded. According to Yao[4], titanium-aluminum alloys melted in the same apparatus and controlled in this way had compositional deviations which did not exceed 1.5% of the nominal weight compositions. Therefore, nominal compositions were used in all cases.

Most ingots were hot-swaged to 0.280 in. diameter rods and were stress-relieved at the swaging temperature. Swaging temperatures were in the range 850° to 1150°C (1562° to 2102°F), with the alloys of higher alloy content requiring the higher temperatures. To minimize oxidation, the ingots were heated in an argon atmosphere in a stainless steel muffle. The 75% titanium alloys were used in the as-cast condition.

After swaging, the alloy rods were machined to approximately 1/4 in. diameter to remove the contaminated layer and were cut into 1/4 in. lengths for heat treatment.

Annealing and quenching of the alloys were carried out in a vertical vacuum-quenching furnace, similar to that described by Bennett[10] The furnace was equipped with a saturable reactor control system which enabled the temperature to be controlled to within ±1°C over the range 700° to 1200°C (1292° to 2192°F).

Metallography

All alloys were prepared for metallographic examination by electropolishing, using an electrolyte of 6% perchloric acid in glacial acetic acid or one developed by Ence and Margolin[11] consisting of:

6% perchloric acid
59% methyl alcohol
35% butyl cellusolve

Specimens for the determination of the β-transus temperatures were etched in a solution of 2% HF, 4% HNO_3 in water. Tie-line alloys were etched in the "R-etch" described by Ence and Margolin[3]. The latter etch was found to minimize level differences between the phases, thus facilitating point counting.

Most specimens were examined on the light microscope. A few tie-line alloy structures, however, were found to be so fine that it was necessary to examine chromium-shadowed collodion replicas of them on the electron microscope at a magnification of 12000X. Point counts on these fitted well with the rest of the data.

Constant Titanium Sections

1. The Beta Transus

All experimental points on the β-transus surface were determined by the temperature bracket method[12]. The β-transus was determined for nine constant titanium sections: 95%, 93%, 91%, 88%, 86.5%, 85%, 84%, 82.5% and 75% titanium. The compositions and the β-transus temperatures of all alloys used for these determinations are shown in Table 1, and the temperatures are plotted as a function of composition in Figures 1, 2 and 3.

A typical β-transus temperature determination for an alloy on the 84% titanium section is shown in Figure 4.

2. The "Alpha" Boundary

The term "alpha" boundary will be used to refer to the boundary across which metallographic examination showed the β phase to disappear as the titanium-aluminum binary was approached.

The "alpha" boundary was determined in the 95%, 93%, 91% and 88% titanium sections. The compositions of alloys used for these determinations are shown in Table 2 and the results are plotted in Figure 1. A typical determination on the 93% titanium section is shown in Figure 5.

Isothermal Sections

Data derived from the constant titanium sections described above were used to develop the β-transus and "alpha" boundary on isothermal sections at 850°, 900°, 950° and 990°C (1562°, 1652°, 1742° and 1814°F). The data points are plotted in Figures 6, 7, 8 and 9. The actual drawing of the boundaries on these isothermals is discussed in a later section.

Ternary Tie-Lines

Using the data plotted on the isothermal sections as a guide, alloy compositions were chosen for the determination of the ternary tie-lines in these sections. The alloy compositions are shown in Table 3.

These alloys were vacuum-annealed at the temperature of the isothermal section in question for four hours and oil-quenched. Longitudinal sections were electropolished and etched, and photographs of four different areas of each sample were taken at a magnification high enough to facilitate point counting. During printing, a reduced millimeter grid was superimposed on each print. Counting was carried out by the previously described method[1], which resulted in the percentage of β being calculated on the basis of the examination of 14,560 grid intersections per alloy. Percentages of β are listed in Table 3.

DISCUSSION

The Binary Diagrams

In the titanium-aluminum constitution diagrams published in 1951 by Ogden et al[13] and in 1952 by Bumps et al[14], aluminum was shown as soluble in alpha titanium up to 24.5%. Many subsequent investigations (3, 4, 5, 6, 7, 15 and 16), however, have shown that this is not the case.

In 1956, Sagel, Schulz and Zwikker[16] published a titanium-aluminum diagram which showed an aluminum solubility of approximately 7% at 800°C (1472°F). A two-phase field, $\alpha + \alpha_2$, was shown between 7% and 11% aluminum. The field of α_2, another hexagonal phase, extending from 11% to 25% aluminum, was interrupted below 1000°C (1832°F) by an $\alpha_2 + \epsilon$ field between 16% and 23% aluminum. The composition of ϵ (probably a tetragonal phase) was 18.6% aluminum.

In 1959, the diagram of Sato, Huang and Kondo[6] showed an aluminum solubility similar to that of Sagel et al, but the $\alpha + \alpha_2$ field extended to 18% aluminum. The α_2 field was located between 18% and 24% aluminum, and no evidence of an ϵ phase was found.

The diagram of Ence and Margolin[3], in 1961, showed an aluminum solubility of about 8% at 800°C (1472°F). The constitution beyond 8% aluminum, however, differed considerably from that shown in previous diagrams. A γ_{Ti_3Al} field and its associated two-phase fields $\alpha + \gamma$ and $\gamma + \delta$ occurred between 8% and 14% aluminum, and a δ_{Ti_2Al} field extended from 14% to 26% aluminum. The diagram, to which an ϵ field has been tentatively added, is shown in Figure 10. Ence and Margolin did not show an ϵ phase in the range of 14% to 24% aluminum but noted that both the transformation markings which they had observed between 16% and 18% aluminum and the veins -- considerably harder than the surrounding δ -- which had appeared between 18% and 22% aluminum were "not inconsistent with the formation of an ordered structure, ϵ , suggested by Sagel et al as occurring at 18% aluminum".

At about the same time, Yao[4], using magnetic susceptibility measurements, offered further evidence for the existence of ϵ and indicated that it had a range of composition of 17% to 18% aluminum. He placed the $\epsilon + \delta$ fields below 1000°C (1832°F) and between 15% and 22% aluminum. It is the boundaries drawn from Yao's data that are shown as ϵ and $\epsilon + \delta$ in Figure 10.

In 1963, the diagram of Clark, Jepson and Lewis[7] showed an aluminum solubility ranging from about 8% at 600°C (1112°F) to 14% at 1100°C (2012°F). The α phase was separated from an ordered phase, α_2, by a two-phase region, the boundary of which extended from about 10% aluminum at 600°C (1112°F) to nearly 16% aluminum at 1100°C (2012°F). A solubility gap was shown in the α_2 field between 11% and 14% aluminum

below a temperature of about 900°C (1652°F). No evidence was found for the existence of an ϵ phase, although the authors remarked that "spurious" microstructures could easily be produced in this composition range.

Thus, although there is some measure of agreement on the extent of aluminum solubility in titanium, there is a wide measure of disagreement on the form of the diagram beyond 8% aluminum, as well as a considerable body of contradictory evidence. However, the existence of the γ phase appears to have been well documented with X-ray, metallographic and microhardness data by Ence and Margolin[3], and for this reason their diagram has been used as the basic binary in the interpretation of the present results. Added to this have been the ϵ and $\epsilon + \delta$ fields of Sagel et al[16] as modified by Yao[4], since this feature has been supported by X-ray and microstructural and hardness data by Sagel, magnetic susceptibility measurements by Yao[4], and microstructural and hardness data by Ence and Margolin[3]. This composite diagram is shown in Figure 10.

The titanium-molybdenum diagram used is that of Hansen et al[17] and is shown in Figure 11.

The Ti-Al-Mo Ternary Diagram

Isothermal sections constructed for 850°, 900°, 950° and 990°C (1562°, 1652°, 1742° and 1814°F) are shown in Figures 6, 7, 8 and 9. In brief, these sections were constructed as follows.

Points on the experimentally determined β-transus and "alpha" boundary at the above temperatures were first plotted on their appropriate isothermal sections and the β-transus was drawn on each.

The "alpha" boundary at this point was drawn from the high titanium end to the 88% titanium section. The β-transus, since it deviated so markedly from a straight line, was drawn as containing a series of cusps, four in number, each of which marked the corner of a three-phase field contiguous with a two-phase field in the titanium-aluminum binary. These three-phase fields were then roughly drawn in.

The tie-lines between 88% and 100% titanium were then plotted using the point-count data, and their direction tended to confirm the initial plot of the $\alpha + \beta + \gamma$ and $\beta + \gamma + \delta$ boundary directions. Since the position of the β-transus was known, it was possible to plot the high aluminum end of each of the remaining tie-lines as a locus. Knowing the approximate direction of the tie-line, it was then possible to plot an "alpha" boundary on each isothermal section and compare the four boundaries thus drawn. Small adjustments within the range of experimental uncertainty were then made, to make all boundaries and tie-lines in the four isothermal sections self-consistent.

The isothermal sections thus derived were used, along with the two binary diagrams, to produce the isometric view of the constitution diagram shown in Figure 12. The third side of the figure is bounded by the 75% titanium section.

The β-transus surface is cut by four valleys each of which is the locus of the tip of a three-phase triangle. The two lower titanium valleys originate in the two peritectoids shown by Ence and Margolin[3], which produce the α and γ phases. The other two originate at a point on the β surface, estimated to be between 1050°C and 1100°C (1922°F and 2012°F), where the β and δ phases react to form ε as shown by the dotted line at ~1080°C (1976°F). The temperature of formation of ε was placed between these limits because the trace of the β-transus on a 1050°C (1922°F) isothermal section drawn as previously described shows a sharp change in direction at approximately 82% titanium whereas a β-transus trace on a 1100°C (2012°F) isothermal section shows no corresponding change in direction. The extension of the four valleys below 850°C (1562°F) is merely tentative but their intersection with the 75% titanium section does suggest a means by which the β-transus trace, which in Figure 3 appears to be running parallel to the titanium-molybdenum binary, can meet it at approximately 640°C (1184°F).

The points of intersection between the isothermal sections and the constant titanium sections were then plotted on the latter and, using the isometric view as a guide, the appropriate boundaries were sketched as shown in Figures 1, 2 and 3.

The constitution diagram thus developed must be considered with two principal reservations in mind. First, no attempt was made to achieve equilibrium in the experimental work, and, secondly, no phase identification work was done. A further reservation is that the non-equilibrium data have been related to binary diagrams in the development of which equilibrium was closely approached. However, the examination of the shape of the β surface has been detailed enough to show conclusively the presence of three or more valleys which could, within reason, be related to the titanium-aluminum binary diagram as being the tips of a series of three-phase fields; the tie-lines have confirmed the directions of the sides of these three-phase fields; and the four isothermal diagrams developed have shown changes with temperature consistent enough to allow a reasonable isometric view of the whole titanium-rich corner to be drawn. It is felt, therefore, that the proposed constitution diagram, in spite of these reservations, can serve as a useful guide for further equilibrium studies and phase identification work.

If the titanium-aluminum diagram of Sato et al[6] were assumed to be correct, only one valley would be expected to appear on the β surface. The diagram of Clark et al[7] would lead to the appearance of only two valleys. The diagram of Sagel et al[16] would require the presence of three valleys but only one of these would occur at the high titanium end of the β surface, the other two being related to the $\epsilon + \delta$ fields. It is only the diagram of Ence and Margolin[3] that, with the addition of the ϵ and $\epsilon + \delta$ fields for which they have shown evidence, requires the presence of the four valleys in the β surface found in the present work. The present data tend to support, therefore, the findings of Ence and Margolin with respect to the titanium-aluminum diagram as a whole, and the findings of

Sagel et al and Yao with respect to the ε and $\varepsilon + \delta$ fields.

SUMMARY

1. The shape of the β-transus surface of the titanium-aluminum-molybdenum system between 75% and 100% titanium has been determined on the basis of a four-hour annealing time. This surface has been shown to contain four valleys, each of which indicates the presence of a three-phase field in the system.

2. From this, together with published data, isothermal sections of the constitution diagram of the system at 850°C, 900°C, 950°C and 990°C (1562°F, 1652°F, 1742°F and 1814°F) have been developed and tie-lines for each determined.

3. The data appear to support the findings of Ence and Margolin with respect to the titanium-aluminum diagram as a whole, and those of Sagel et al and Yao with respect to the ε and $\varepsilon + \delta$ fields.

4. The proposed constitution diagram, while it must be treated with reservations since it is not based on equilibrium data, can serve, nevertheless, as a useful guide for further equilibrium studies and phase identification work.

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AJW/(PES)/KW

TABLE 1

Beta Transus Alloys

Composition			β-transus Temp.	Composition			β-transus Temp.
Ti %	Al %	Mo %	± 3°C	Ti %	Al %	Mo %	± 3°C
95.0	4.5	0.5	983	86.5	12.0	1.5	1099
95.0	3.5	1.5	958	86.5	9.0	4.5	1015
95.0	2.75	2.25	935	86.5	6.0	7.5	942
95.0	2.0	3.0	916	86.5	3.0	10.5	857
95.0	1.0	4.0	881				
95.0	0.0	5.0	838	85.0	12.0	3.0	1077
				85.0	9.0	6.0	1004
93.0	6.0	1.0	1007	85.0	6.0	9.0	928
93.0	5.0	2.0	975	85.0	3.0	12.0	853
93.0	4.0	3.0	941				
93.0	3.5	3.5	920	84.0	12.0	4.0	1073
93.0	3.0	4.0	910	84.0	9.0	7.0	986
93.0	2.0	5.0	880	84.0	6.0	10.0	914
93.0	1.0	6.0	848	84.0	3.0	13.0	840
93.0	0.0	7.0	819				
				82.5	11.5	6.0	1044
91.0	8.5	0.5	1074	82.5	10.0	7.5	1004
91.0	8.0	1.0	1051	82.5	9.0	8.5	974
91.0	7.5	1.5	1036	82.5	8.0	9.5	936
91.0	6.75	2.25	1014	82.5	7.0	10.5	924
91.0	6.0	3.0	995	82.5	6.0	11.5	902
91.0	5.0	4.0	966	82.5	5.0	12.5	880
91.0	3.0	6.0	904	82.5	3.0	14.5	831
91.0	1.0	8.0	834				
91.0	0.0	9.0	805	75.0	11.0	14.0	1040
				75.0	9.0	16.0	1016
88.0	8.0	4.0	1008	75.0	7.0	18.0	973
88.0	7.0	5.0	985	75.0	5.0	20.0	860
88.0	6.0	6.0	959				
88.0	5.0	7.0	924				
88.0	4.0	8.0	899				
88.0	3.0	9.0	868				
88.0	1.0	11.0	812				

TABLE 2

"Alpha" Boundary Alloys

Composition			Temp. °C	No. of Phases	Composition			Temp. °C	No. of Phases
Ti %	Al %	Mo %			Ti %	Al %	Mo %		
95	5.0	0.0	900	1	91	8.5	0.5	900	1
95	4.5	0.5	900	2	91	8.25	0.75	900	1
					91	8.0	1.0	900	2
95	5.0	0.0	800	1	91	7.75	1.25	900	2
95	4.5	0.5	800	2	91	7.25	1.75	900	2
					91	8.5	0.5	800	1
93	6.75	0.25	1000	2	91	8.25	0.75	800	1
93	6.5	0.5	996	2	91	8.0	1.0	800	2
					91	7.75	1.25	800	2
93	6.5	0.5	950	2	91	7.25	1.75	800	2
93	6.5	0.5	900	1					
93	6.25	0.75	900	2	88	11.5	0.5	1000	1
93	6.0	1.0	900	2	88	11.0	1.0	1000	2
					88	10.5	1.5	1000	2
93	6.5	0.5	800	1	88	11.5	0.5	900	1
93	6.25	0.75	800	2	88	11.0	1.0	900	1
93	6.0	1.0	800	2	88	10.5	1.5	900	2
93	5.5	1.5	800	2	88	10.0	2.0	900	2
93	5.0	2.0	800	2	88	11.5	0.5	800	1
					88	11.0	1.0	800	1
91	8.75	0.25	996	2	88	10.5	1.5	800	2
91	8.5	0.5	996	2	88	10.0	2.0	800	2
91	8.0	1.0	996	2					

TABLE 3

Tie-Line Alloys

Composition			Temp. °C	Per Cent β	Composition			Temp. °C	Per Cent β
Ti %	Al %	Mo %			Ti %	Al %	Mo %		
81.5	12.0	6.5	990	66.4	80.0	10.0	10.0	900	67.4
83.5	11.5	5.0	990	60.1	83.5	11.5	5.0	900	36.3
85.5	10.0	4.5	990	65.9	85.5	10.0	4.5	900	37.9
88.5	8.5	3.0	990	62.1	88.5	8.5	3.0	900	26.0
91.25	7.0	1.75	990	61.7	90.5	7.0	2.5	900	29.4
					92.5	6.0	1.5	900	18.2
80.5	12.0	7.5	950	56.8	96.0	2.0	2.0	900	80.6
83.5	11.5	5.0	950	49.8					
85.5	10.0	4.5	950	48.4	79.0	10.0	11.0	850	58.5*
88.5	8.5	3.0	950	39.1	85.5	10.0	4.5	850	31.2*
91.25	7.0	1.75	950	35.4	88.5	8.5	3.0	850	21.1
92.5	6.0	1.5	950	38.7	90.5	7.0	2.5	850	24.3
					92.5	6.0	1.5	850	13.0
					96.0	2.0	2.0	850	38.9

*Derived from electron micrographs of chromium-shadowed collodion replicas, at a magnification of 12000X.

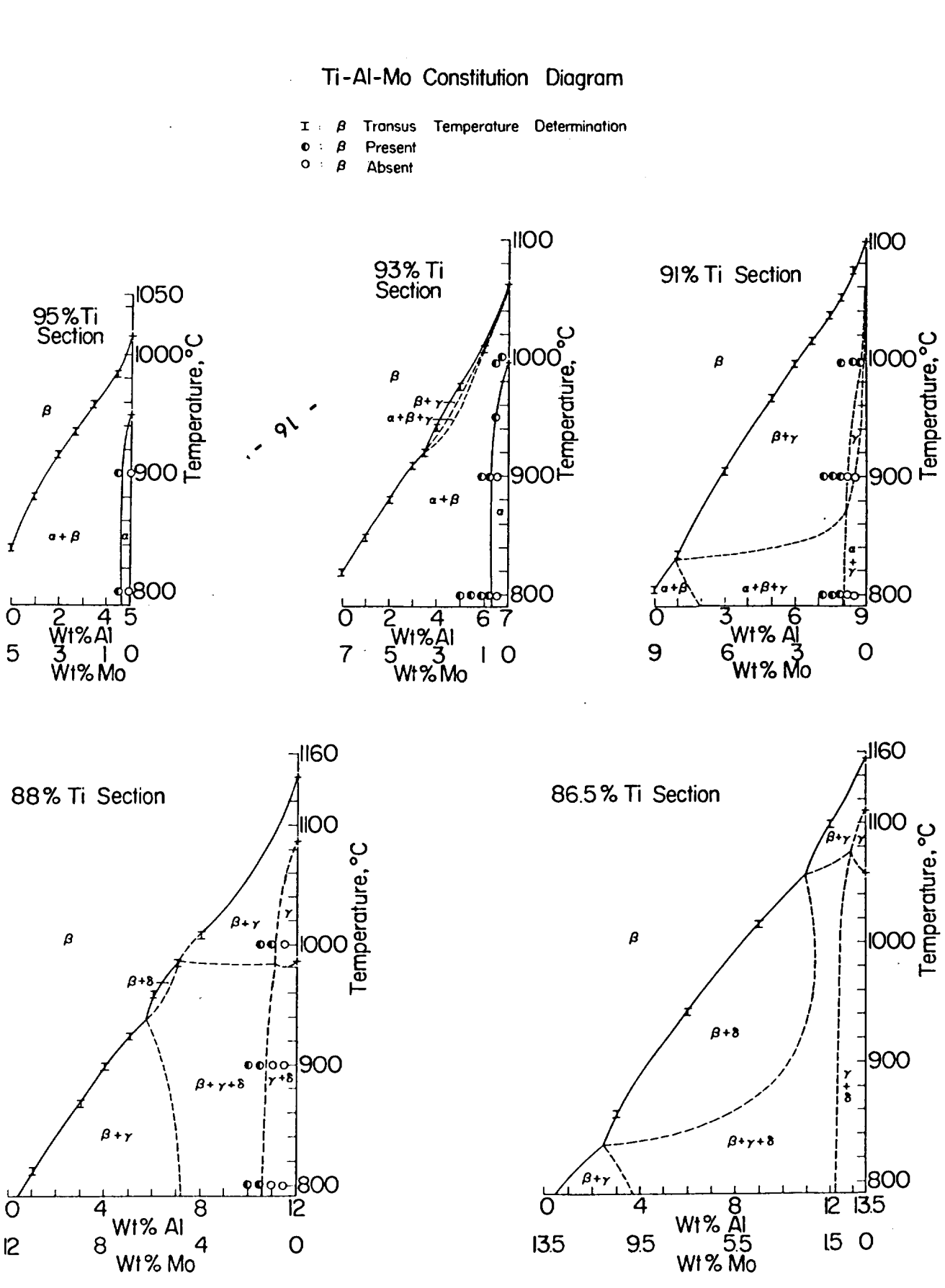
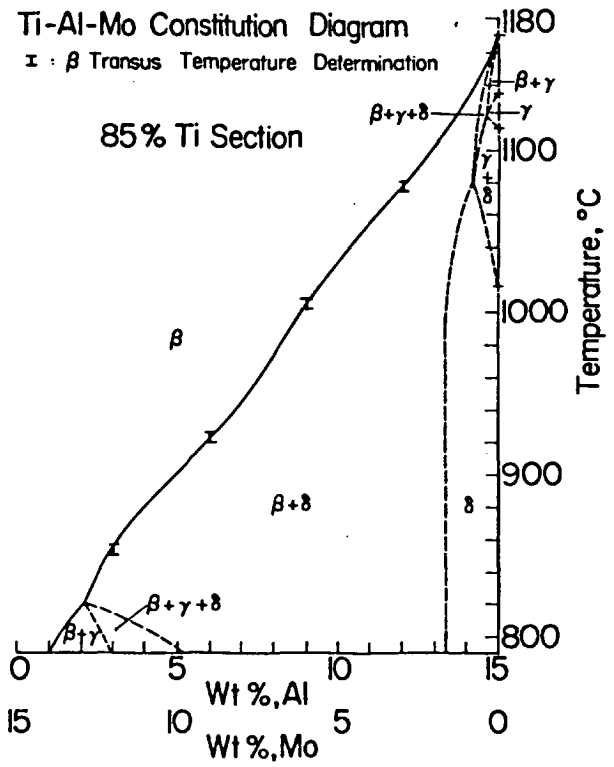


Figure 1. 95%, 93%, 91%, 88% and 86.5% Ti sections.



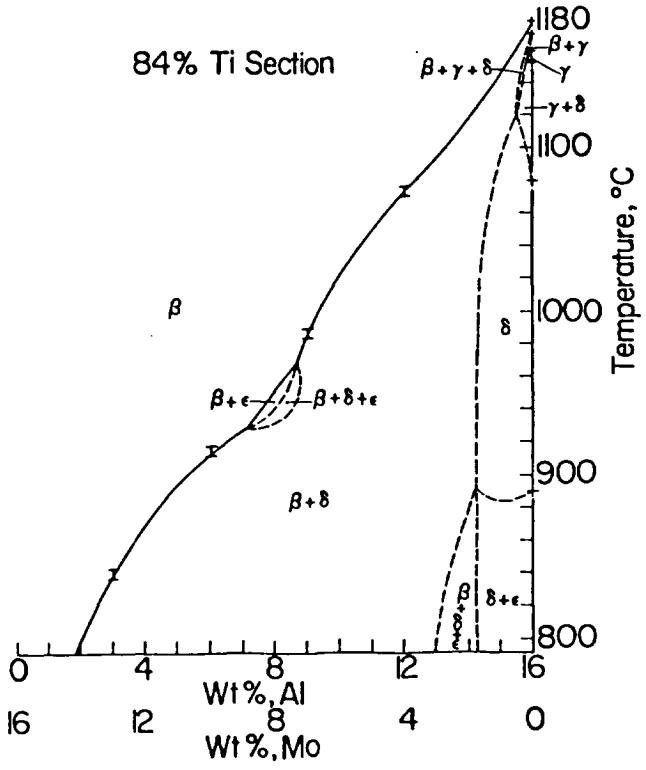


Figure 2. 85% and 84% Ti sections.

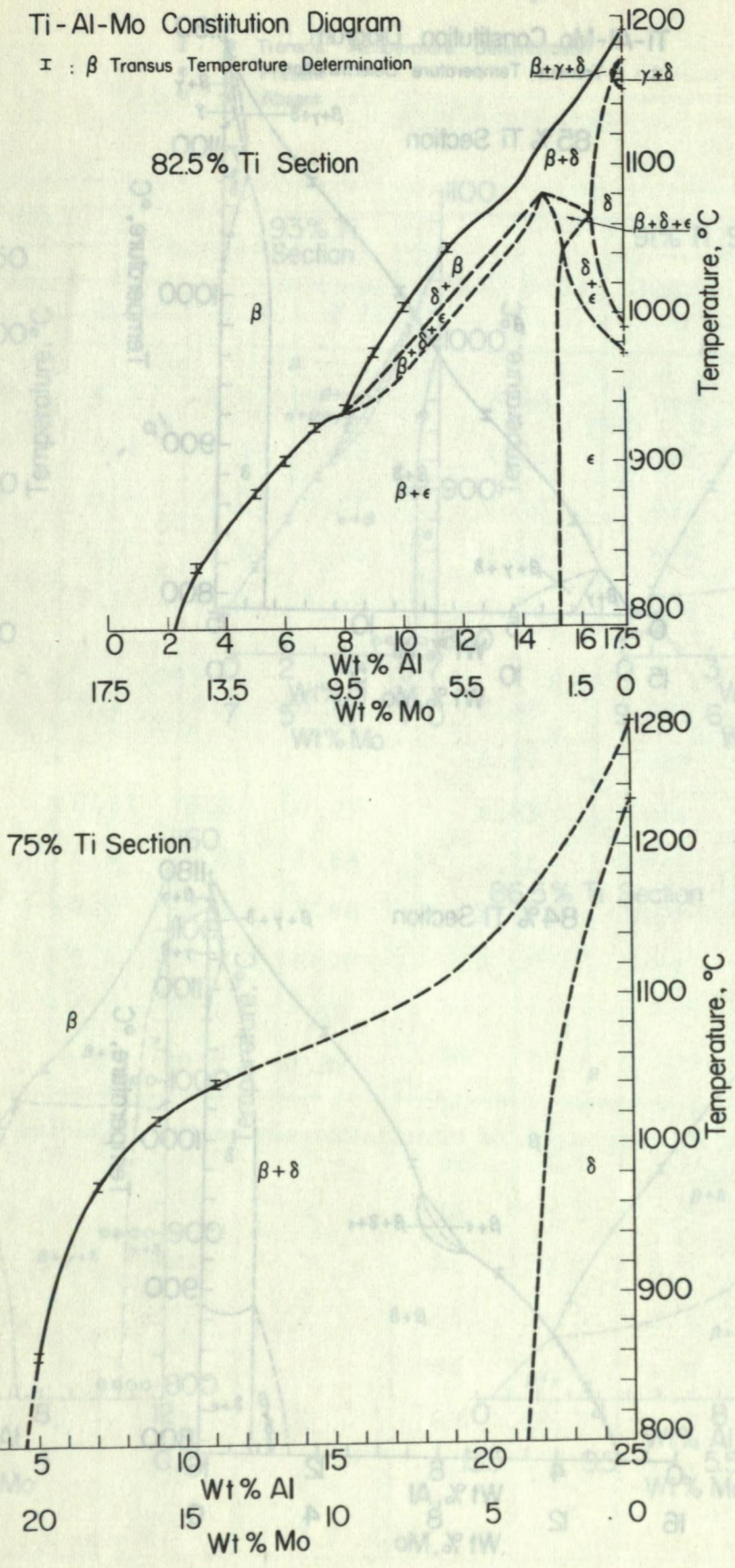
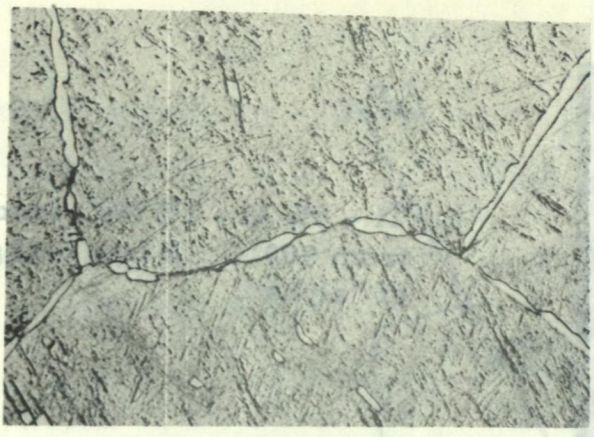
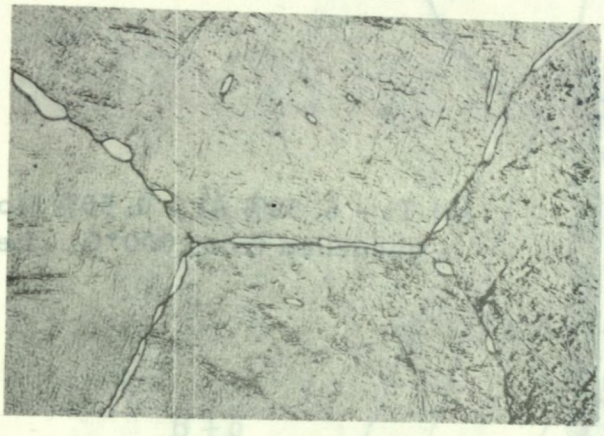


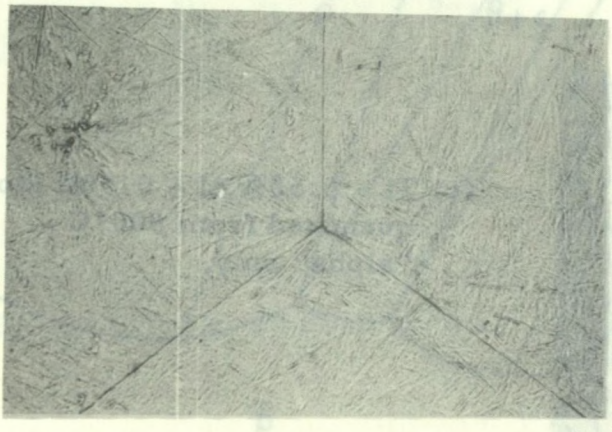
Figure 3. 82.5% and 75% Ti sections of the Ti-Al-Mo constitution diagram.



(a) Quenched from 1069°C - transformed β + second phase.

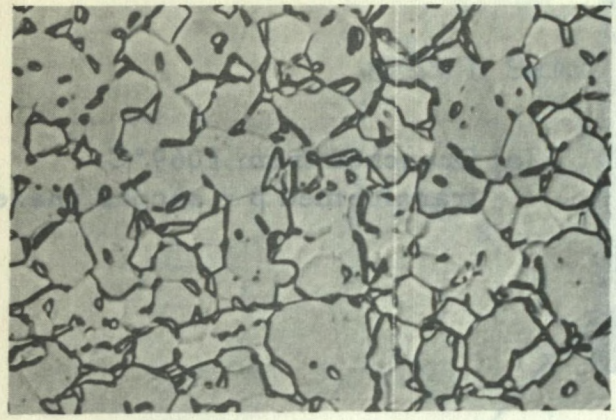


(b) Quenched from 1072°C - transformed β + less second phase than (a).

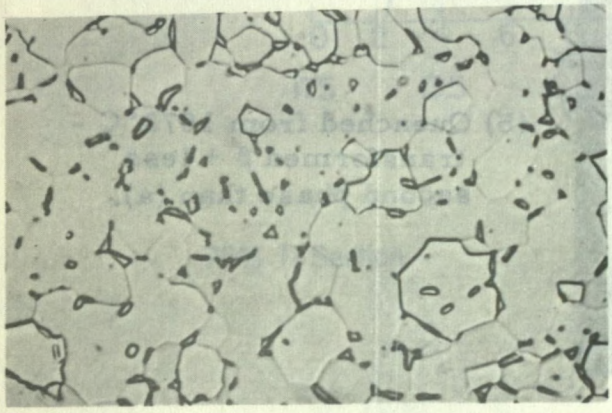


(c) Quenched from 1075°C - transformed β only.

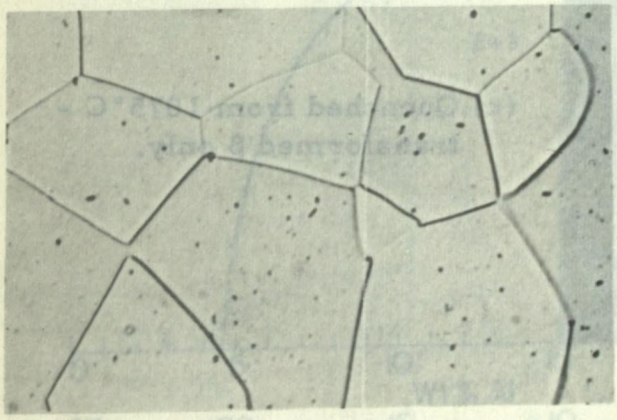
Figure 4. Beta transus determination on Ti-12%Al-4%Mo alloy. Beta transus at 1073° ±3°C. (X500)



(a) Ti - 6% Al - 1% Mo quenched from 900°C. β in a background of "alpha".



(b) Ti - 6.50% Al - 0.50% Mo quenched from 900°C. Less β.



(c) Ti - 6.50% Al - 0.50% Mo quenched from 900°C - "alpha" only.

Figure 5. "Alpha" boundary determination on the 93% titanium section at 900°C. "Alpha" boundary between (b) and (c). (X900)

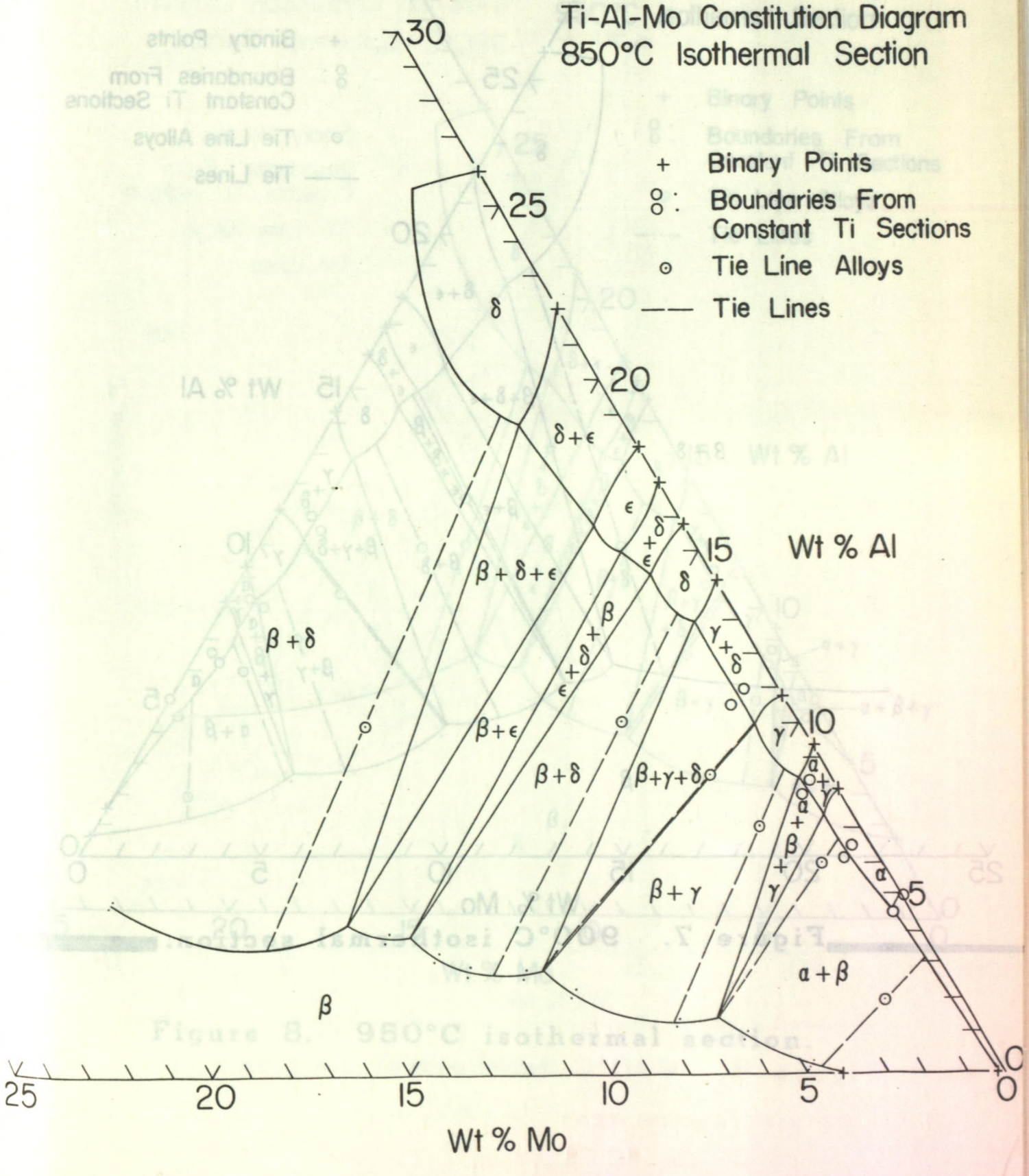


Figure 6. 850°C isothermal section.

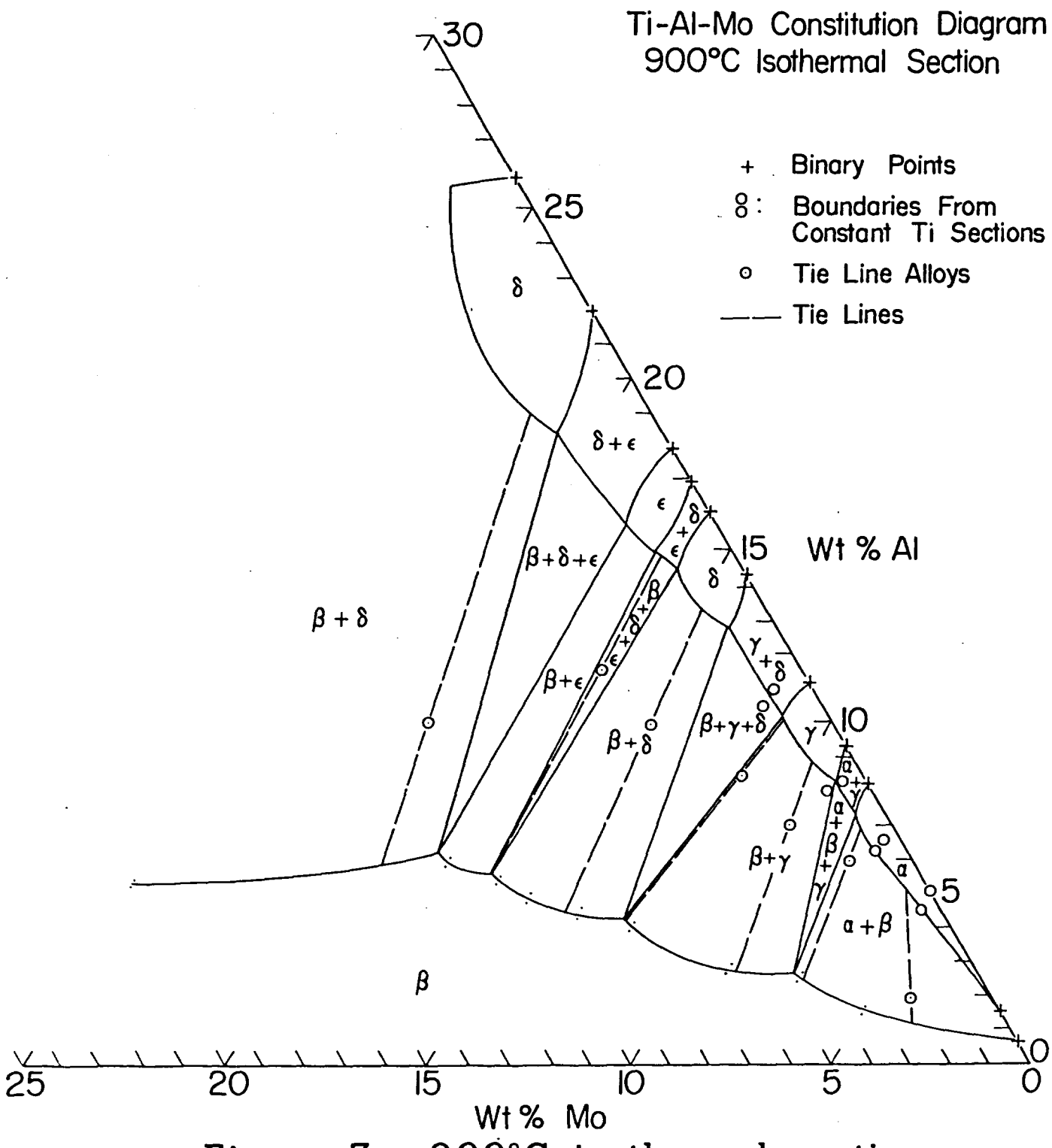


Figure 7. 900°C isothermal section.

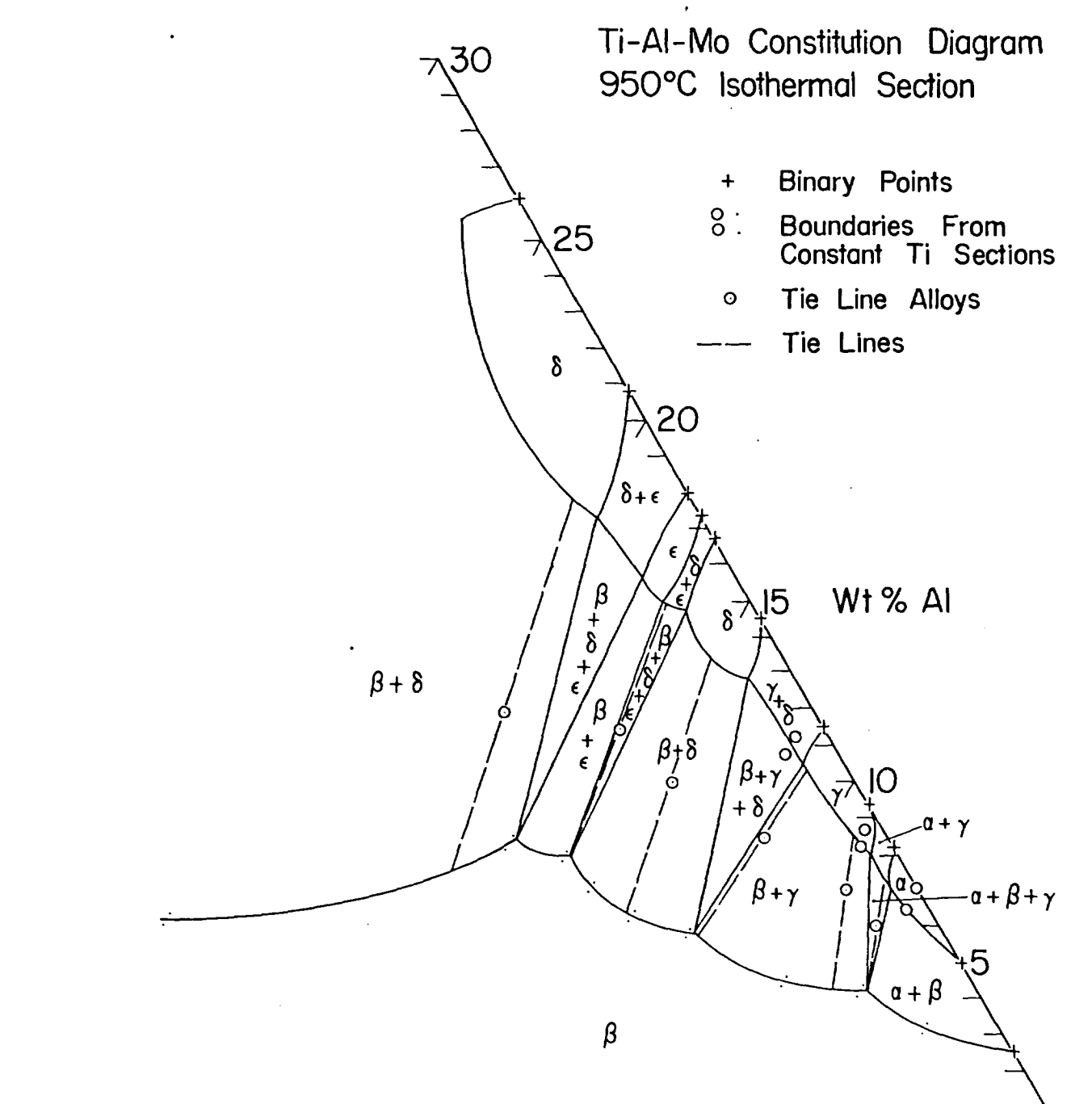


Figure 8. 950°C isothermal section.

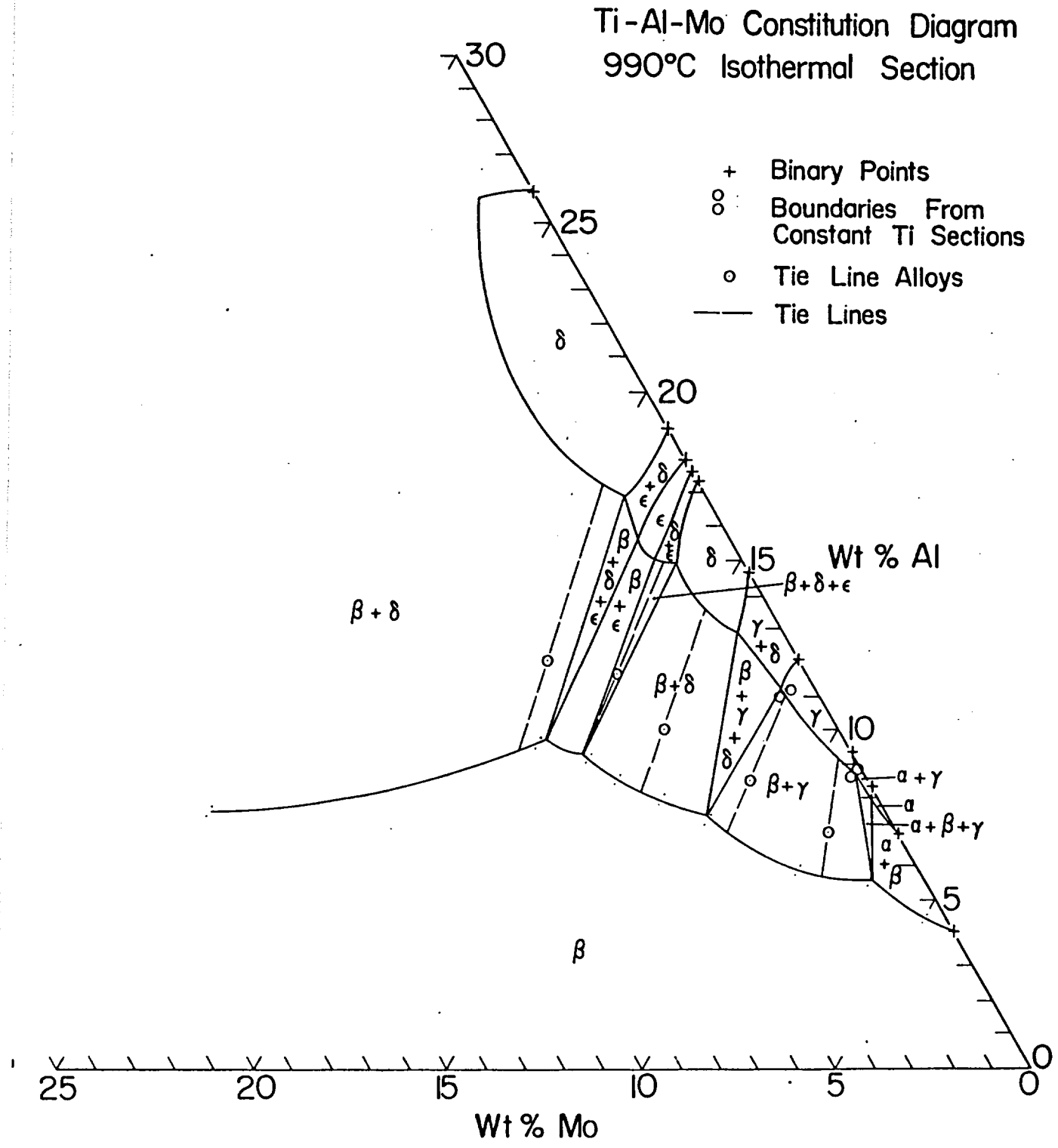


Figure 9. 990°C isothermal section.

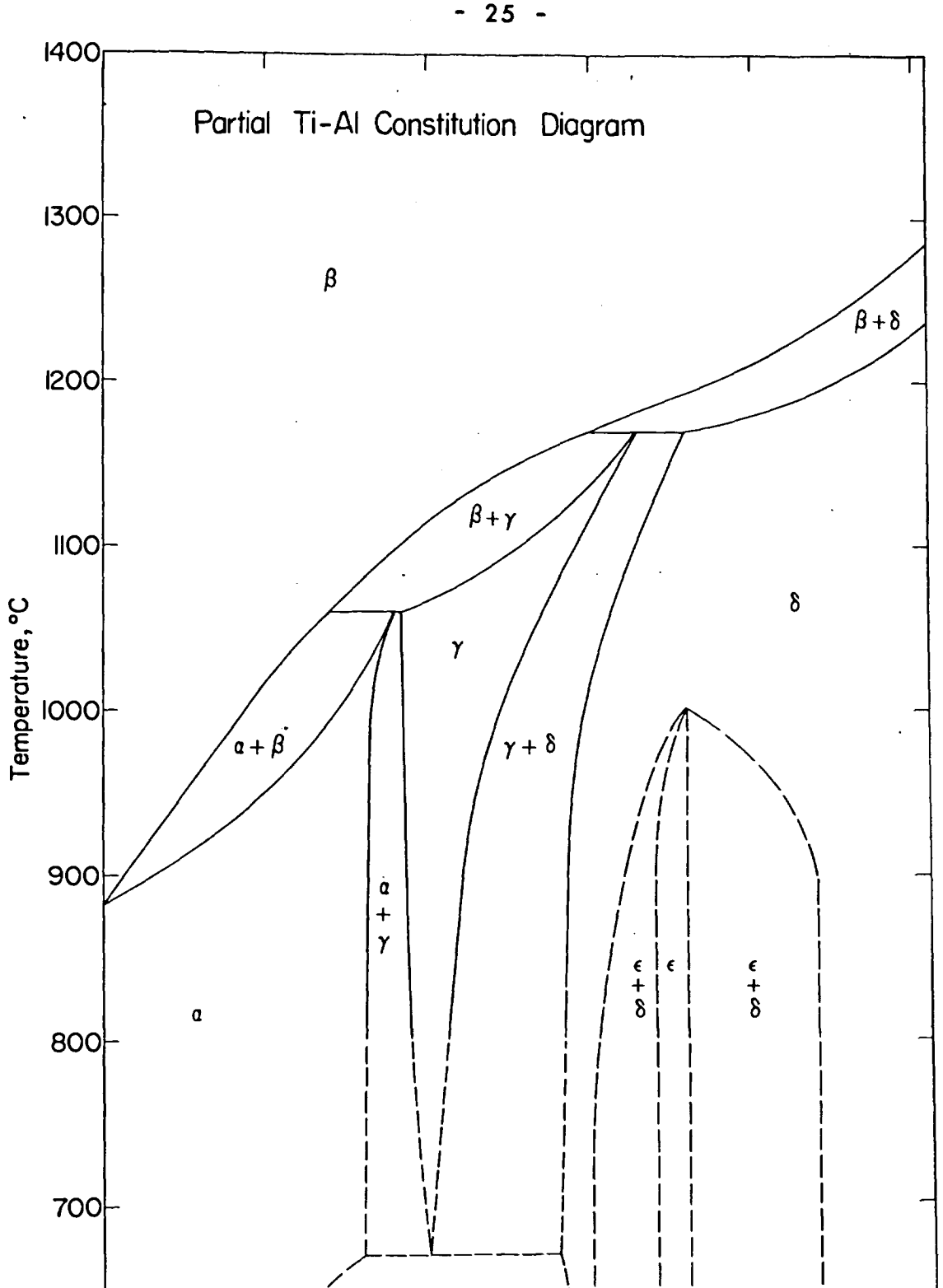


Figure 10. Partial Ti-Al constitution diagram.

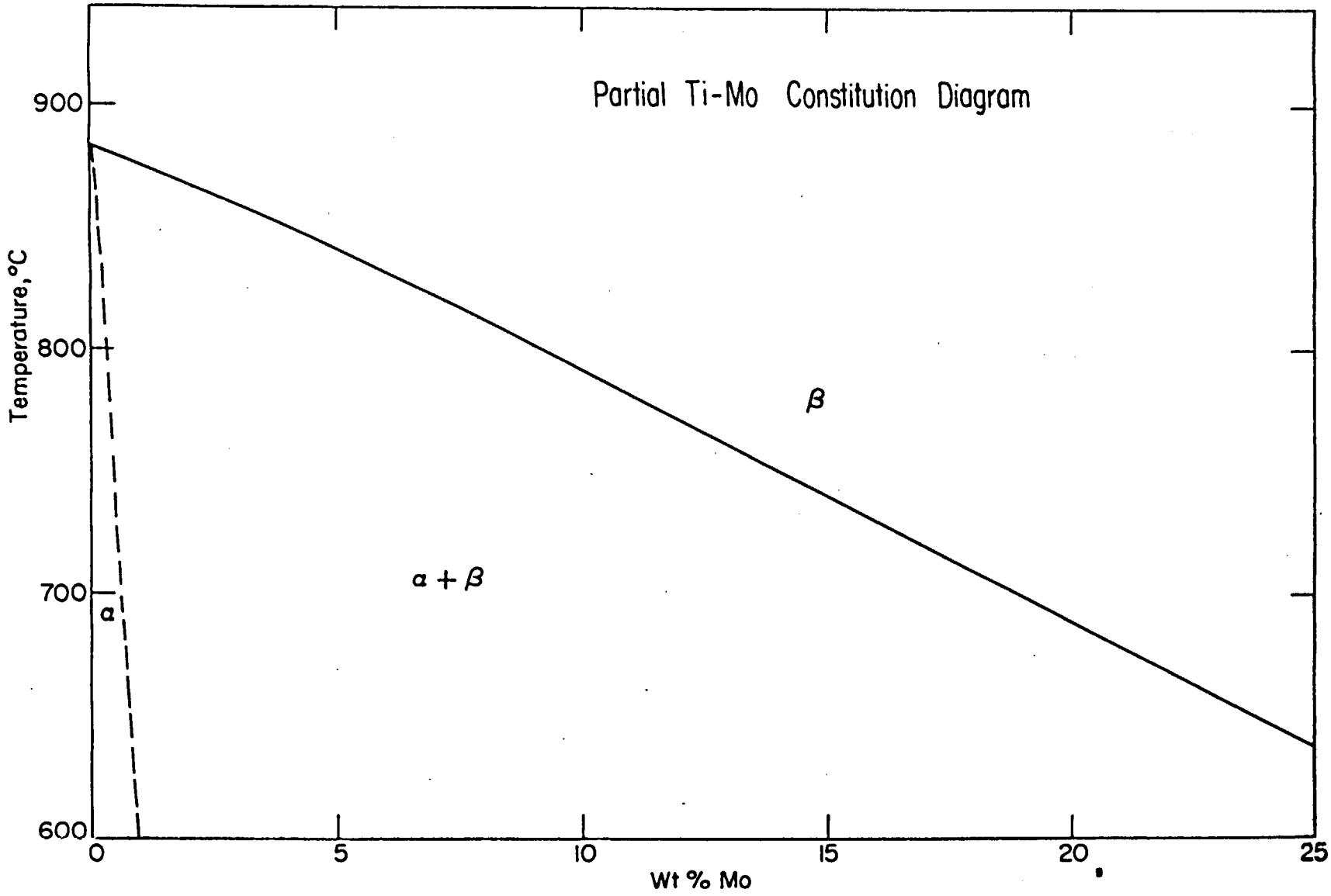


Figure 11. Partial Ti-Mo constitution diagram.

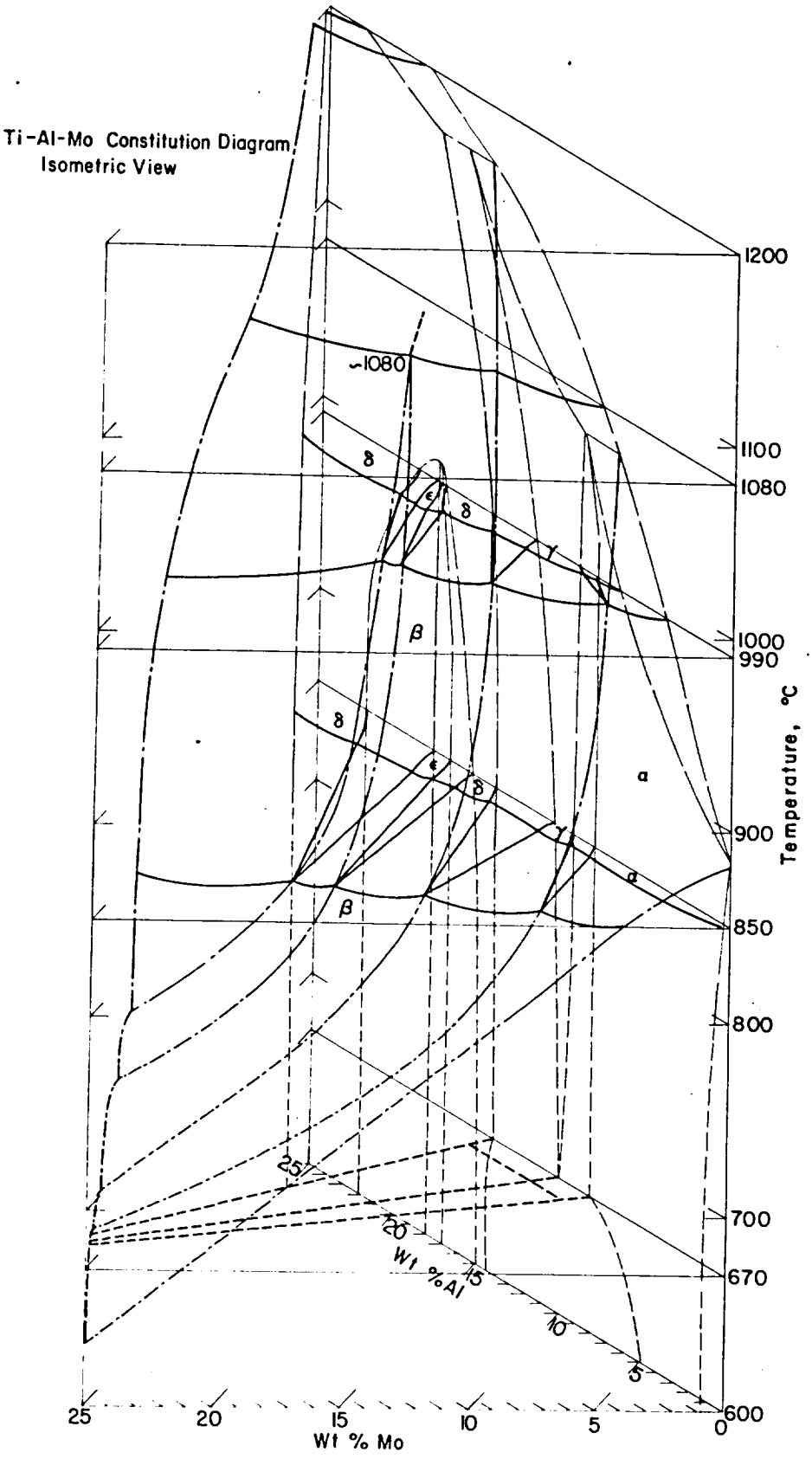


Figure 12. Isometric view of the Ti-Al-Mo constitution diagram.