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The Quench Hardening of Titanium-Manganese Alloys

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THE QUENCH HARDENING OF TITANIUM-MANGANESE ALLOYS

Interim Technical Report No. 1

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THE QUENCH HARDENING OF TITANIUM-MANGANESE ALLOYS

Interim Technical Report No. 1

Object: To evaluate the nature of the quench-hardening reaction in Ti-Mn alloys utilizing a critical review of the literature.

Summary: Evaluation of the quench-hardening reaction has been made on the basis of (a) consideration of alloy systems showing a similar crystallographic transformation (b.c.c. to c.p.h.), and (b) consideration of systems showing similarity in phase diagrams to the Ti-Mn diagram.

Conclusions: From a study of the evidence presented, the quenching transformation is believed to be of the martensitic type. The unstable α' formed on quenching, probably with a c.p.h. structure, may be responsible for the quench-hardening phenomenon in the Ti-Mn alloys.

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Introduction

Much work has been done in recent years to improve the strength of titanium-base alloys. Titanium has great potential value as an engineering material because it has one of the highest strength-to-weight ratios of materials known today and, in addition, displays superior corrosion resistance under various operating conditions. Moreover, the existence of an allotropic transformation and the concomitant possibilities for quench hardening have interested research workers engaged in alloy development.

Previous Work on the Ti-Mn System

According to recent work (1)*, the beta-to-alpha (b.c.c. to c.p.h.) transformation in iodide titanium occurs at $882.5^{\circ} \pm 1^{\circ}\text{C}$. It is known that the beta phase in pure titanium cannot be retained even by an extremely rapid quench to room temperature. The same phenomenon is also encountered in other pure metals, such as zirconium, thallium and iron. However, the introduction of alloying elements often makes the transformation of the high temperature phase so sluggish that a portion or all of this phase is retained on quenching or even slow cooling. This behavior is typical of titanium.

* References are listed consecutively at the back of the report

A number of elements lower the transformation temperature of beta titanium and tend to stabilize this phase. Among these elements are columbium (2), cobalt (3), chromium (4), copper (5), iron (6), manganese (7), molybdenum (2), nickel (8), silicon (9), tantalum (7), vanadium (10), tungsten (7) and zirconium (11).

Addition of the beta-stabilizing elements, manganese and chromium, has produced titanium alloys which quench harden. Figure 1, based on Battelle (12) data, shows the effect of homogenizing temperatures on hardness of titanium alloys containing up to 10% manganese. Greatest hardening is obtained on quenching from 1700°F, at which temperature all alloys used are in the beta field, with alloys containing 3-7% manganese developing maximum hardness. In addition, Table 1 (12) shows the effect of quenching temperatures on the final hardness of manganese and chromium alloys.

Table 1. Effect of Quenching Temperature on Hardness of Ti-Mn and Ti-Cr Alloys

Composition in %	Vickers Hardness Number	
	Homogenized at 950°C. for 10 min. in argon atmosphere	
	Quenched in cold water (0°C.)	Quenched in liquid nitrogen (-196°C.)
<u>Mn</u>		
1.0	268	218
3.5	377	383
5.0	373	514
10.0	351	351
<u>Cr</u>		
2.5	319	304
3.5	342	368
5.0	332	540
10.0	324	359
15.0	322	314

From Table 1, the effect of quenching in liquid nitrogen seems to be similar in both the Ti-Mn and Ti-Cr systems. Maximum hardness of almost identical value occurs in the 5% alloy of each system. It is of interest to note that the 5% alloys are the only ones to show an extensive increase in hardness between the cold water quench and liquid nitrogen quench data. Similar changes in hardness, both positive and negative, were observed in both systems for manganese or chromium content above and below five percent.

It is also interesting to note that age hardening occurs in the Ti-Mn alloys on annealing in the alpha-beta field and that highest hardness among the alloys listed is attained in the 5% Mn alloy. This is shown in Fig. 2, (12), which summarizes the isothermal transformation data obtained with Ti-Mn alloys homogenized at 950°C for 10 minutes in an argon atmosphere and quenched into a lead bath at $350 \pm 3^\circ\text{C}$.

The titanium-rich Ti-Mn equilibrium diagram determined at Battelle (7) is reproduced here as Fig. 3. The main features of this partial equilibrium diagram are that the β transus line decreases continuously with increasing Mn content, the amount of solubility of Mn in α phase is quite limited, and there is some evidence of the existence of a eutectoid reaction.

The Ti-Mn Quench-Hardening Reaction

The occurrence of quench hardening in the Ti-Mn system has been adequately substantiated in the present literature survey. It is the purpose of the present investigation, therefore, to study the conditions under which such hardening occurs and to determine the mechanism underlying that behavior.

The transformation in Ti-Mn alloys can be considered on the basis of (a) structural alteration (from b.c.c. to c.p.h.), and (b) the similarity existing between the Ti-Mn phase diagram and other alloy systems.

(a) Consideration of the b.c.c. to c.p.h. transformation:

Not many alloy systems show transformations like the beta-to-alpha transformation in titanium. The most extensively studied system was the Cu-Al system first investigated by Wassermann (13) in the composition range from 11.9 to 12.7% Al. Figure 4 shows the copper-rich portion of the Cu-Al equilibrium diagram containing the eutectoid composition. The high temperature phase, β , transforms into a structure of $\alpha + \delta$ on slow cooling, but when rapidly cooled, transforms to β' through a martensitic transformation. The β' phase, determined later (14), shows a distorted c.p.h. structure.

Wasserman suggested that the phase transformation around β eutectoid composition was as follows:

upon "rapid" cooling from the β field (Fig. 4),

β (disordered b.c.c.) $\rightarrow \beta_1$ (ordered b.c.c.) $\rightarrow \beta'$ (distorted c.p.h.)

and upon reheating, $\beta' \rightarrow \beta_1 \rightarrow \alpha + \delta \rightarrow \beta$;

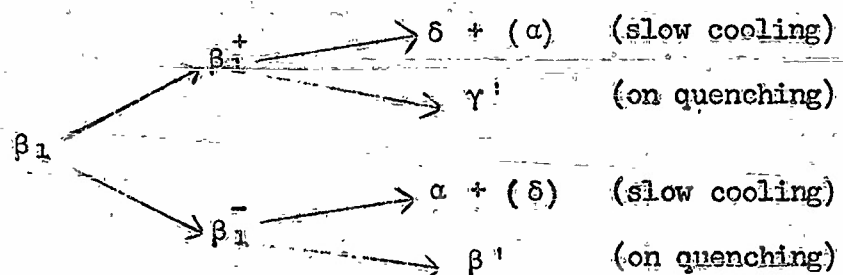
and that the $\beta_1 \rightleftharpoons \beta'$ transformation was completely reversible. Later, Kurdjumov and Stelletzku (15) discovered a new martensitic lattice having a crystal structure different from that of β' and which they designated γ' . According to them, β' resulted from rapid cooling of alloys containing between 12.9 and 13.1% Al, and γ' only from alloys above 13.1% Al. Microscopic work showed the metallographic structure of β' to be almost identical with γ' , the only difference being that γ' "needles" often contained apparent twin bands.

Klier and Grymko (16) recently made a microstructural study of this transformation and clarified the sequence of the decomposition of β_1 (or β). The isothermal transformation, at or below eutectoid temperature, in the 13.5% Al specimens investigated showed that two distinct reactions occurred. The first reaction resulted in the formation of an acicular precipitate but the structure was not stable, and on further holding at that temperature, was subject to a second reaction of agglomeration and growth. The resulting structure underwent no further change during continued isothermal holding.

After quenching from the isothermal anneal, prior to the start of the first reaction, the structure as quenched was γ' with possible traces of β' . For longer annealing times, through the course of the first reaction, the structure in the quenched specimen became a mixture of γ' and β' . This

meant that the γ' revealed was not original matrix γ' but rather, because of the formation of the large amount of β' , the γ' formed was of necessity rich in Al. The γ' was next replaced by δ and the structure was then δ and β' . For the fully transformed alloy, the structure was composed of α and δ .

Since both β' and γ' formed from β_1 (or β) of characteristic composition, Klier and Grymko pointed out that the structures from the first reaction before quenching consisted of β_1 of two compositions, namely, β_1^+ (Al-rich) and β_1^- (Al-depleted). When quenched, β_1^+ transformed to γ' and β_1^- to β' . On slow cooling, β_1^+ became enriched in Al to the point where it could precipitate as δ . The precipitation of δ depleted the β_1^- of Al, to the point where α could precipitate, the final structure then being made up of δ and α . The sequence was as follows:



As found by Isaitschen, Kaminsky, and Kurdjumow (14), γ' is a c.p.h. structure with an ordered arrangement of atoms. β' differs from γ' in that the [0001] direction is at an angle

of 2° to the (0001) plane normal, and the angle between the plane (10 $\bar{1}$ 0) and (01 $\bar{1}$ 0) differs about 1° from 120° . The writers also indicated that β' can be transformed to γ' by mechanical deformation, stating that ".....after deformation, some lines on the Debye pattern of β' phase merge together, giving broad lines; the new line system becomes analogous to the line system of the γ' phase".

The orientation relationship between β_1 and γ' as found by Greninger (17), may be defined crystallographically as follows:

$$\left. \begin{array}{l} (0001)_{\gamma'} \parallel (110)_{\beta_1} \\ [01\bar{1}0]_{\gamma'} \parallel [111]_{\beta_1} \end{array} \right\} A_1$$

$$\left. \begin{array}{l} (0001)_{\gamma'} : (011)_{\beta_1} = 4^\circ \\ [01\bar{1}0]_{\gamma'} \parallel [111]_{\beta_1} \end{array} \right\} A_2$$

A_1 and A_2 here are in twin relationship, with the (10 $\bar{1}$ 1) plane of A_1 as the twinning plane. Since β' is of a pseudo-hexagonal framework, as mentioned previously, its orientation relationship (17) can only be approximately represented as:

$$\begin{array}{l} (0001)_{\beta'} : (110)_{\beta_1} = 4^\circ \\ [10\bar{1}0]_{\beta'} \parallel [111]_{\beta_1} \end{array}$$

In addition, the habit plane as determined by Greninger (17) is (111) $_{\beta_1}$ for β' and (221) $_{\beta_1}$ for γ' in the transformations

$\beta_1 \rightarrow \beta'$ and $\beta_1 \rightarrow \beta''$ respectively.

The phase transformation in zirconium is another example of the transformation from b.c.c. to c.p.h. structure. From a study of the orientation relationship, Burgers (18) has proposed that the martensite transformation occurs in Zr by a heterogeneous shear on the system (112) [111]. The crystallographic principle underlying his proposal is that the configuration of atoms in the (112) plane of a b.c.c. structure is exactly the same as that in the (1010) plane of a c.p.h. structure based on the same atomic radius. Thus a close-packed hexagonal structure can be built up from a b.c.c. structure simply by displacing the (112) planes relative to each other. This mechanism leads to orientations that can be described by the relation:

$$\begin{aligned} (110)_{\text{b.c.c.}} &\parallel (0001)_{\text{c.p.h.}} \\ [111]_{\text{b.c.c.}} &\parallel [11\bar{2}0]_{\text{c.p.h.}} \end{aligned}$$

The habit plane resulting from this mechanism would then automatically be the (112) plane. However, the habit plane found by Bowles (19) in the b.c.c. to c.p.h. transformation in Li is a plane bearing Miller indices of the {111} type.

Another possible mechanism presented by Burgers (18) is that the structural transformation from b.c.c. to c.p.h. could have an intermediate f.c.c. transition lattice.

The structural cycle can be briefly illustrated as follows:

b.c.c. $\rightarrow$ f.c.c. (β -brass $\rightarrow$ α -brass)

$$(110)_{b.c.c.} \parallel (111)_{f.c.c.}$$

$$[111]_{b.c.c.} \parallel [110]_{f.c.c.}$$

f.c.c. $\rightarrow$ c.p.h. (β -Co $\rightarrow$ α -Co)

$$(111)_{f.c.c.} \parallel (0001)_{c.p.h.}$$

$$[110]_{f.c.c.} \parallel [11\bar{2}0]_{c.p.h.}$$

Thus, the net transformation will be:

b.c.c. $\Rightarrow$ c.p.h.

$$(110)_{b.c.c.} \parallel (0001)_{c.p.h.}$$

$$[111]_{b.c.c.} \parallel [11\bar{2}0]_{c.p.h.}$$

which is the same as was just given for the case of Zr.

Weerts (20) reported that the epsilon phase transformation in the Ag-Zn system also had a b.c.c. to h.c.p. structural alteration with the following relationships:

$$(111)_{b.c.c.} \parallel (0001)_{c.p.h.}$$

or

$$(110)_{b.c.c.} \parallel (10\bar{1}0)_{c.p.h.}$$

Bowles (19) reported that the habit plane of martensitic transformation in a b.c.c. matrix was located

mostly in the vicinity of some minor planes with indices corresponding to {hhl}. No satisfactory theoretical explanation has been offered for this observation.

(b) Consideration of the similarity between phase diagrams:

The quenching of iron-rich Fe-Ni alloys from the high temperature gamma field (f.c.c. structure), Fig. 5, as first reported by Owen and Sully (21), produced a distorted alpha structure (α'). They observed that, after quenching from 800°C a completely distorted alpha, α' , structure was present in alloys of Ni content from 2.96 to 23.32 atomic percent and co-existed with retained gamma in alloys from 27.12 to 30.60 atomic % Ni. The lines in the X-ray diffraction pattern of α' are different from those of α , in that they are much broader, the (310) doublet of α' with Co radiation usually being unresolved and diffuse. The material was obviously not in equilibrium, and the lattice was considerably distorted.

It was this distorted structure (α') which was unfamiliar to Osmond and Cartaud (22) and led them to speculate that a simple eutectoid reaction existed in the Fe-Ni equilibrium diagram. Also, this same distorted structure, α' was designated as martensite by Hanson and Freeman (23) owing to its resemblance to the martensite in carbon steel. Later, Owen (24), based on his observations on meteorites,

described α' as ".....a supersaturated alpha solid solution of Ni iron in which there will be internal stresses caused by the distortion of the crystal lattice owing to the excess of Ni,..... Such material is in an unstable condition, and if heated to moderate temperatures will separate into the stable components, kanacite and taenite, as found in meteorites".

The iron-rich portion of the Fe-Mn equilibrium diagram has phase boundary lines similar to those in the Ti-Mn system (Fig. 6). As shown by Troiano and McGuire (25), the gamma transformation in the Fe-Mn system occurs in almost the same manner as that in the beta-eutectoid Cu-Al system. On being quenched to room temperature, the gamma transformed to α' of the same composition and the amount of α' varied directly with the amount of gamma originally present. The α' was also characterized by diffuse lines displaced so as to indicate an abnormally large lattice parameter. The final X-ray pattern revealed the presence of two alpha solutions, namely, equilibrium alpha with sharp lines, and supersaturated alpha with diffuse lines. Gamma of approximately 3 to 15 atomic percent Mn gave an X-ray diffraction pattern corresponding to the lines of the supersaturated alpha phase.

Gamma of approximately 13 to 30 atomic percent Mn in the Fe-Mn system formed the unstable epsilon phase (c.p.h.)

during quenching. The quenching temperature and specimen size, apparently influenced the amount of epsilon phase formation. In general, the higher the quenching temperature and the larger the specimen, the greater was the amount of epsilon phase formed. However, under no conditions was epsilon observed below approximately 10 and above 26 atomic percent Mn.

The transformation temperatures of $\gamma \rightarrow \alpha'$ and $\gamma \rightarrow \epsilon$ are independent of the cooling rate. The presence of multiple twin-like structure in ϵ distinguishes its microstructure from the α phase.

Both ϵ and retained γ decompose during cold-rolling. In alloys of 10.62 to 13.00% Mn, the γ retained after quenching transformed almost entirely to supersaturated α' with a trace of ϵ phase. Alloys of 15.24 to 16.74% Mn showed approximately equal amounts of only γ and ϵ after quenching. At an intermediate stage of deformation, only ϵ with a small amount of α' was present, while after severe deformation, the Debye pattern indicated the structure to consist almost entirely of α' with only traces of ϵ . The large amount of additional α' could have been formed only from the decomposition of ϵ .

Duvez (26) also found the supersaturated alpha structure, α' , in the Ti-Mo system by means of X-ray diffraction patterns. He found it in Ti-Mo alloys of up to 12% Mo, after

homogenizing at 980°C for 24 hours and quenching from the beta field. Only partial transformation occurred from about 4 to 12% Mo, while above 12% Mo, the β structure was retained. Also, although the temperature of the allotropic transformation in pure Ti decreased progressively from 882°C to about 850°C with increasing rates of cooling from 4° to 10,000° per sec., the cooling rate had no effect on the temperature of transformation of β phase into α' (curve M in Fig. 7). He suggests the retention of β at room temperature might be explained on the basis of curve M, Fig. 7, extrapolated to the 12% Mo ordinate line below room temperature.

In alloys showing a transformed α structure, Duwez reported that the diffuseness of the lines corresponding to α' structure did not permit him to determine the lattice parameter of α' structure with any degree of certainty. Similar observations have been reported for the Fe-Ni (21) system. Obviously, no amount of annealing will improve the diffuse lines of α' introduced during quenching, since annealing would cause decomposition of the α' .

Hansen and his collaborators (2) were unable to differentiate between the transformed structure (α') and isothermal α structure on the basis of X-ray diffraction patterns. Nevertheless, the equilibrium β phase in the Ti-Mo system will transform during quenching partially or

wholly into α' at a given temperature in a two-phase region within definite composition limits. It would be reasonable to expect this metastable α' structure to play an important role in the quench-hardening mechanism.

The alloy systems Fe-Ni, Fe-Mn and Ti-Mo discussed above have the same type of equilibrium diagram at the base-metal end as for the Ti-rich Ti-Mn system. Their quenching products also appear to be related closely by the similarity of their equilibrium diagrams and this view can be even extended to the eutectoid-type equilibrium diagrams of Cu-Al (β -eutectoid) and Fe-C systems. Although the structural transformation in the Fe-Ni and Fe-Mn systems is from f.c.c. to b.c.c., the change in the Ti-Mn system is from b.c.c. to c.p.h. The Ti-Mo system, however, which exhibits the same structural change as does the Ti-Mn system, also shows the same quenching product appearing in the Cu-Al (β -eutectoid) system.

Summary

The nature of martensitic transformation is such that it does not require re-adjustment of the concentration of the alloying ingredients, i.e., it is a diffusionless type of reaction and the extent of the reaction usually is an inverse function of the reaction temperature. The martensitic transformation produces a metastable transition structure, the formation of which is accompanied by considerable hardening

of the metal. From the evidence presented, the quenching transformation in the Ti-Mn system would seem to be of the martensitic type. However, the form of the transition structure and the possible mechanism by which it is generated will have to be determined experimentally.

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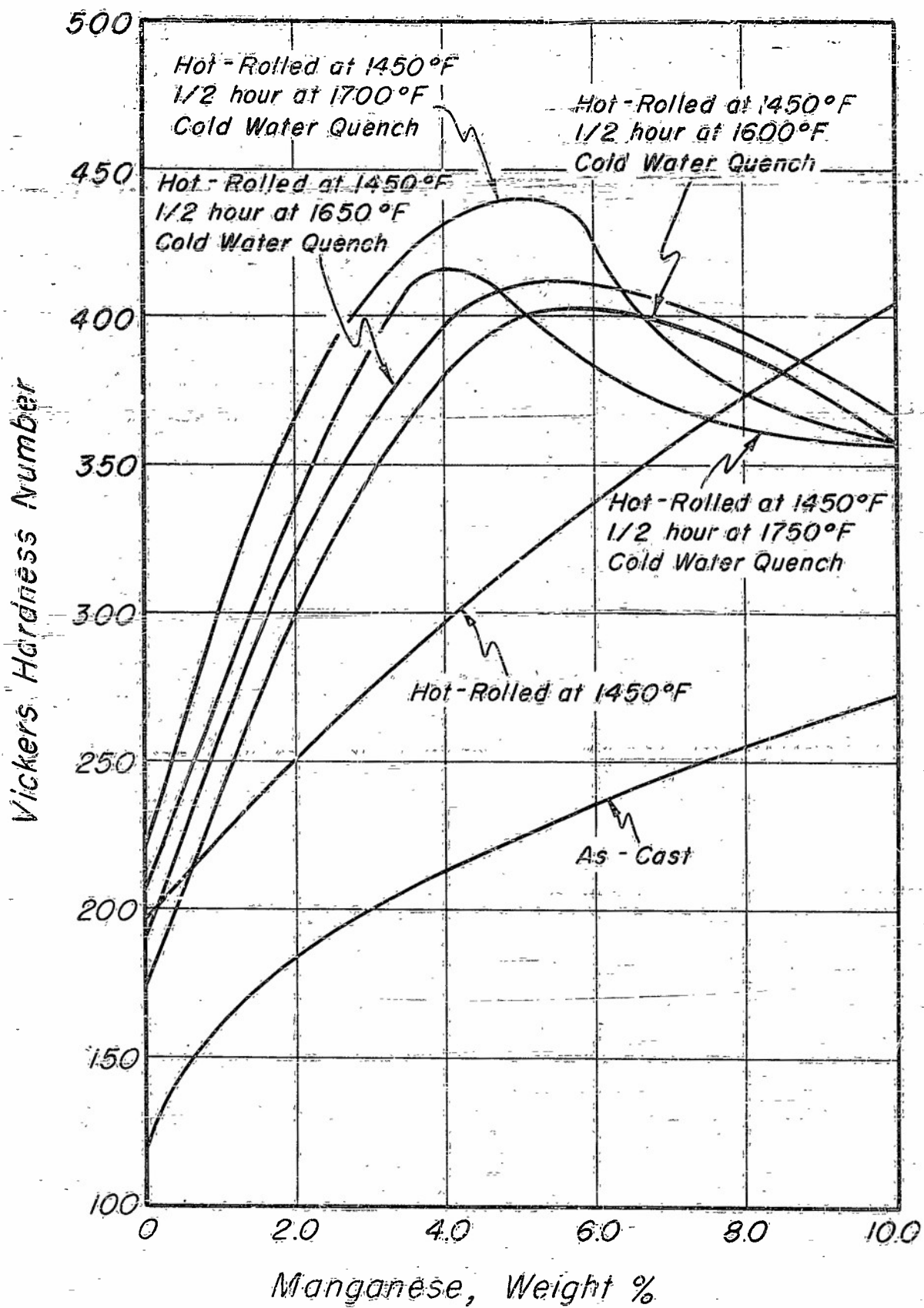


Fig. 1 — EFFECT OF HEAT TREATMENT
ON Ti - Mn ALLOYS (Battelle)

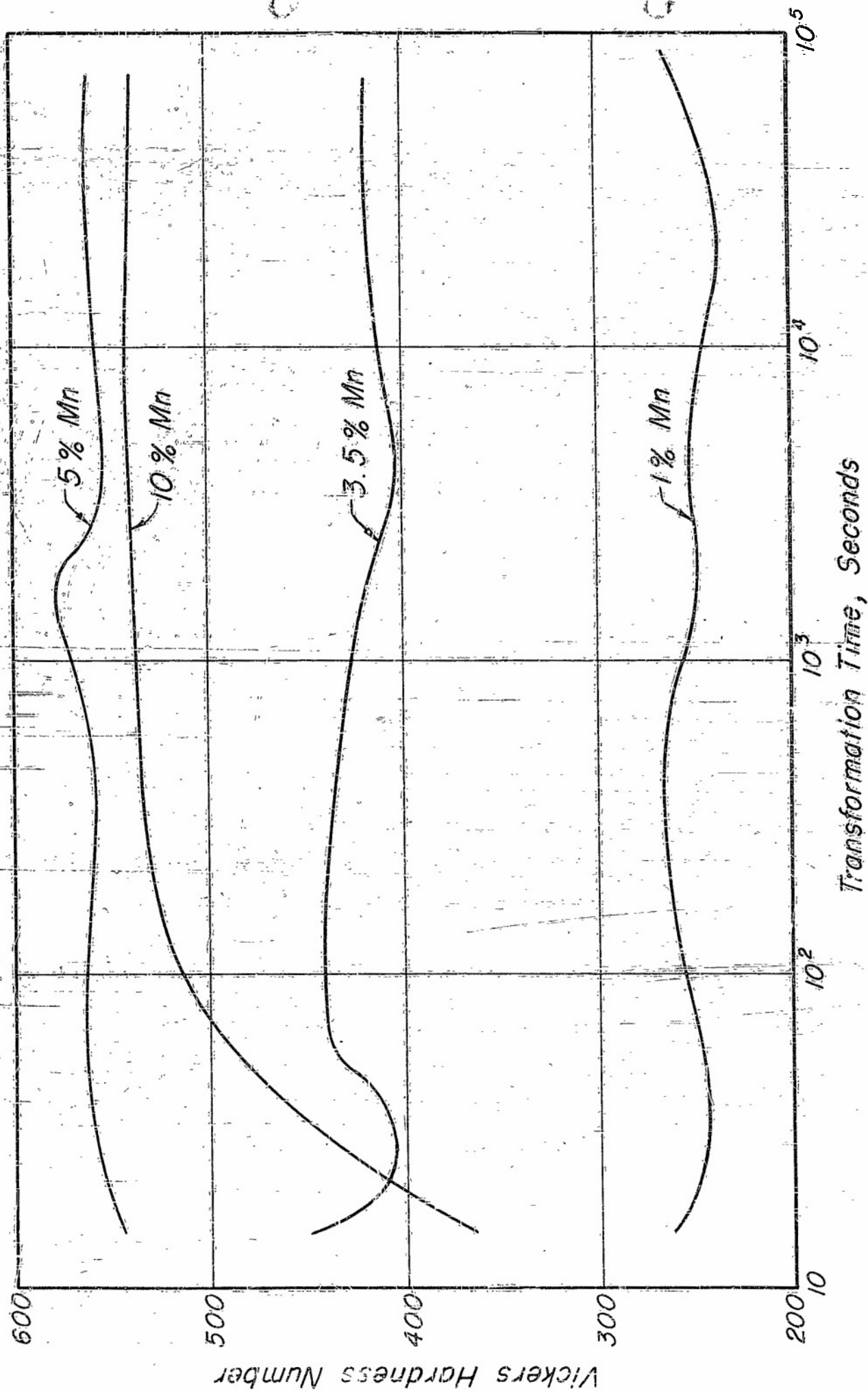


Fig. 2 - ISOTHERMAL TRANSFORMATION CURVES OF Ti-Mn ALLOYS (Battelle)

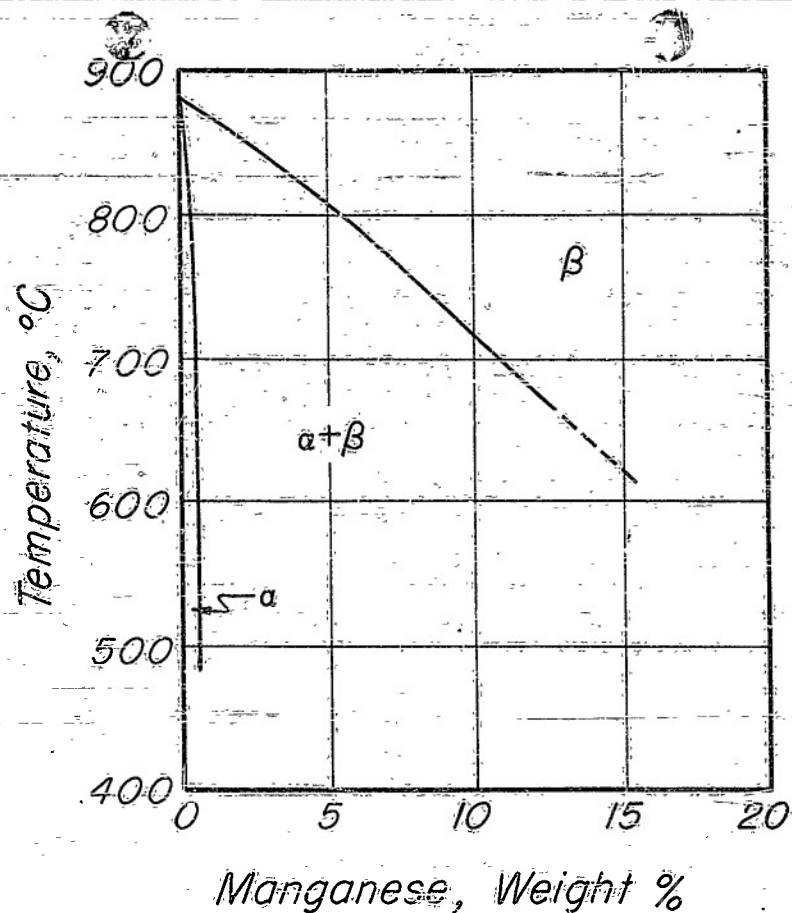


Fig. 3 - Titanium-Rich Portion of the Tentative Ti-Mn Equilibrium Diagram (Battelle)

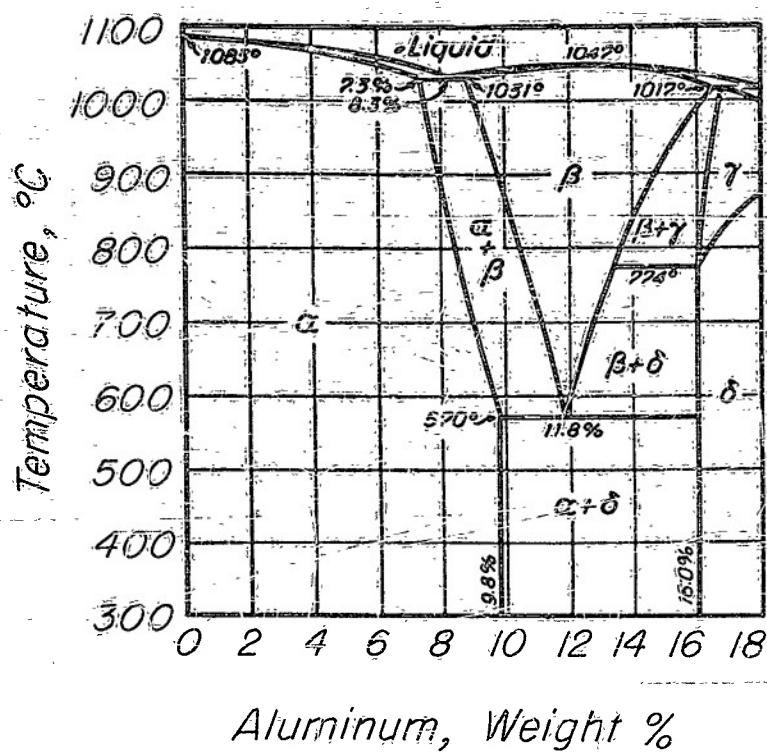


Fig. 4 - Copper-Rich Portion of the Cu-Al Equilibrium Diagram (National Metals Handbook)

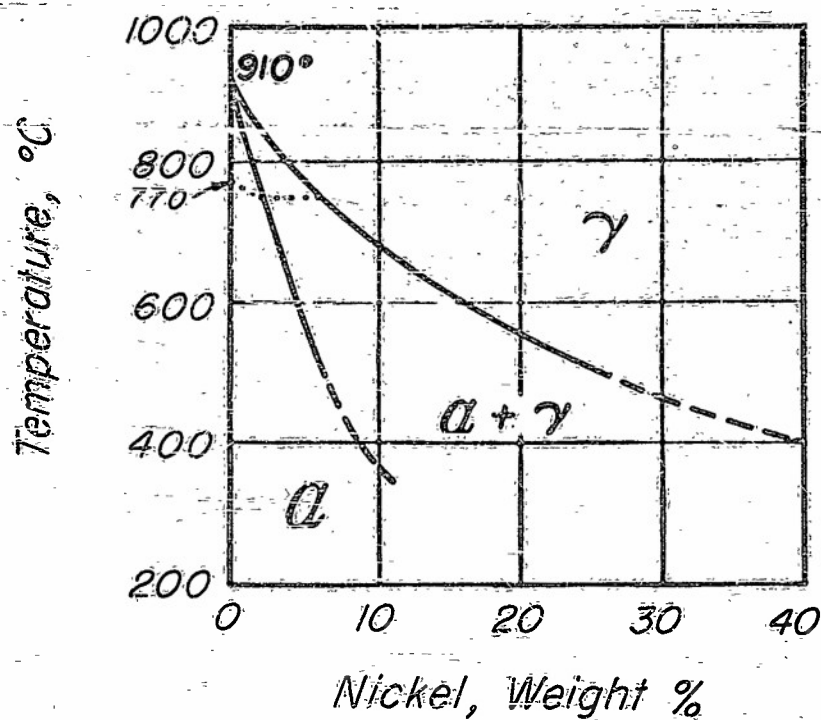


Fig. 5 - Iron - Rich Portion of the Fe - Ni Equilibrium Diagram (Metals Handbook 1948 Edition)

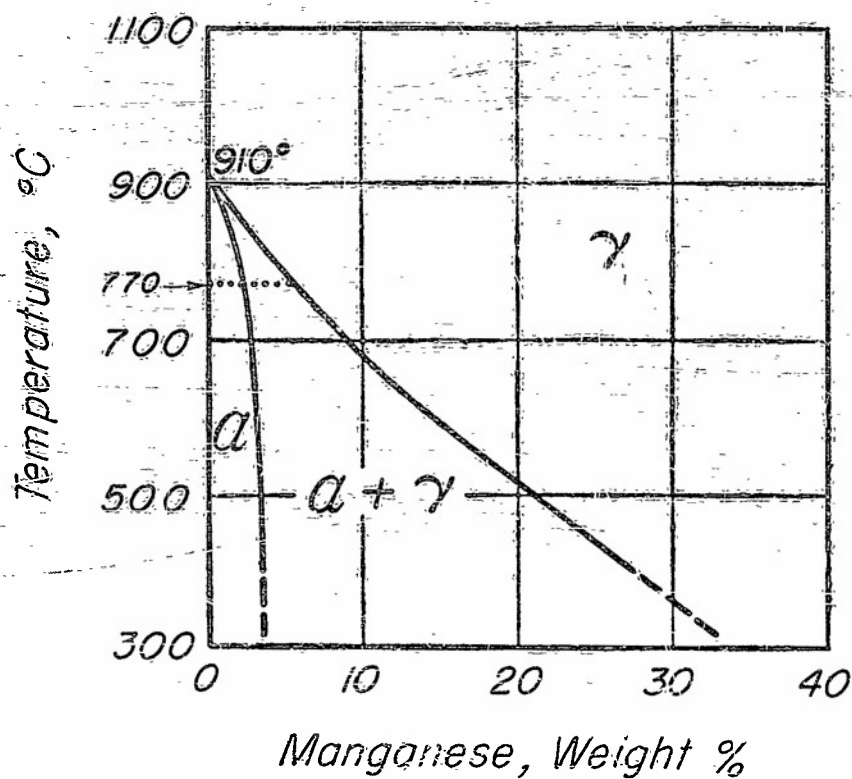


Fig. 6 - Iron - Rich Portion of the Fe - Mn Equilibrium Diagram (Metals Handbook 1948 Edition)

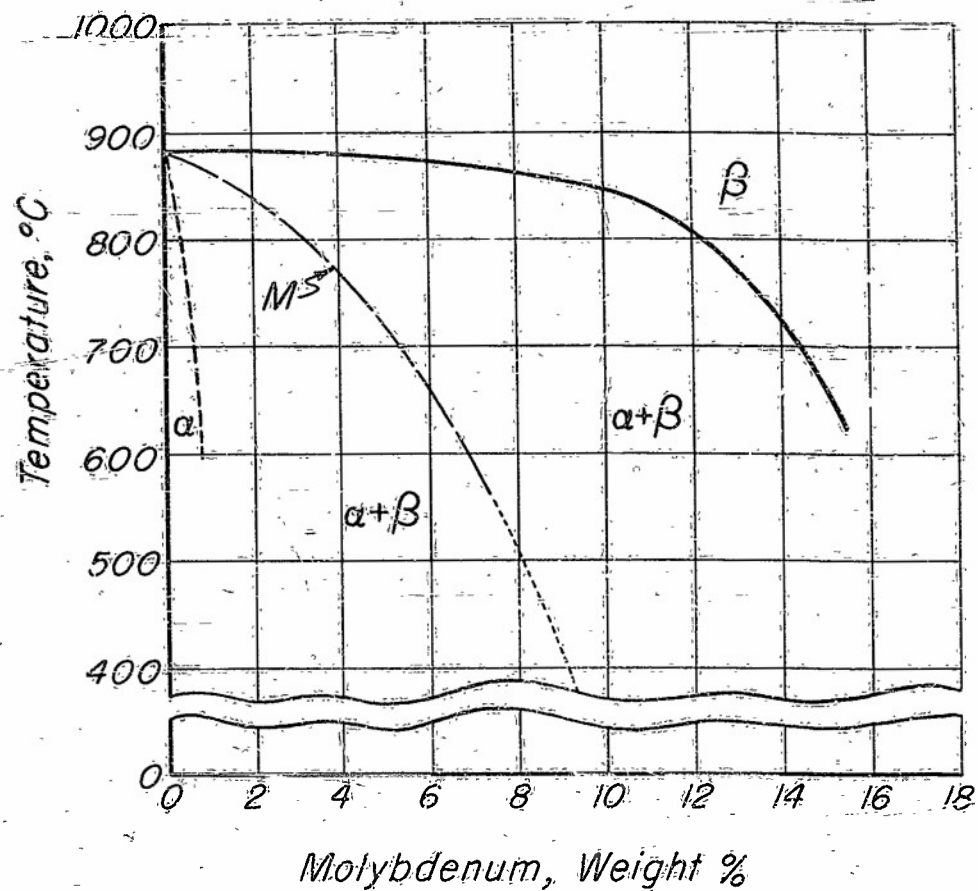


Fig. 7 — Titanium - Rich Portion of the Ti - Mo Equilibrium Diagram (Duwez)