AD-783 235

DEHYDRIDING OF TITANIUM ALLOY (Ti-6246) HYDRIDED POWDER

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Cincinnati University

Prepared for:

Air Force Materials Laboratory

June 1974

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SECURITY CLASSIFICATION OF THIS PAGE (*When Data Entered*)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFML-TR-74-31	2. GOVT ACCESSION NO	3. RECIPIENT'S CATALOG NUMBER AD-783 235
4. TITLE (*and Subtitle*) DEHYDRIDING OF TITANIUM ALLOY (Ti-6246) HYDRIDED POWDER		5. TYPE OF REPORT & PERIOD COVERED Final Report March 1973 - October 1973
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) N. Birla V. DePierre		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS University of Cincinnati Cincinnati, Ohio 45221		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Materials Laboratory Air Force Systems Command Wright-Patterson Air Force Base, Ohio 45433		12. REPORT DATE June 1974
		13. NUMBER OF PAGES 35
14. MONITORING AGENCY NAME & ADDRESS (*if different from Controlling Office*)		15. SECURITY CLASS. (*of this report*) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (*of this Report*)

Approved for public release, distribution unlimited.

17. DISTRIBUTION STATEMENT (*of the abstract entered in Block 20, if different from Report*)

18. SUPPLEMENTARY NOTES

NATIONAL TECHNICAL
INFORMATION SERVICE
[illegible]

D D C
AUG 14 1974
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19. KEY WORDS (*Continue on reverse side if necessary and identify by block number*)

Dehydriding, Titanium, Time, Temperature

20. ABSTRACT (*Continue on reverse side if necessary and identify by block number*)

A systematic investigation of temperature - pressure - time variables for dehydriding titanium alloy (Ti-6246) powder was made to determine practical processing conditions for reducing the hydrogen content of the powder to levels below 100 ppm. Analyses of the experimental results of tests made between 1200°F and 1500°F indicate that 1500°F is the optimum dehydriding temperature. The controlling process for dehydriding can be expressed by second order reaction 2H(gas) = H_2 (gas) with an activation energy of 21.260±1940 cal. Below 0.088

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micron partial pressure of hydrogen, the type of vacuum system used for dehydriding is believed to be the major factor for removal of these molecules.

FOREWORD

This report was prepared by the Metals and Processing Branch, Metals and Ceramics Division, Air Force Materials Laboratory (LLM). The work was initiated under Project No. 7351, "Metallic Materials," Task No. 735108, "Processing of Metals." The research was conducted in the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, by Dr. N. Birla of the University of Cincinnati, Cincinnati, Ohio and Mr. Vincent DePierre of the Air Force Materials Laboratory (LLM).

This report covers work performed from March 1973 to October 1973. It was submitted by the authors in December 1973.

The authors gratefully acknowledge the assistance of Mr. F. Gurney and T. E. Jones of the Westinghouse Electric Corporation, Astronuclear Laboratory, Wright-Patterson Air Force Base, Ohio for execution of the experimental program.

SUMMARY

A systematic investigation of temperature-pressure-time variables for dehydriding titanium alloy (Ti-6246) powder was made to determine practical processing conditions for reducing the hydrogen content of the powder to levels below 100 ppm. Studies made between 1200°F and 1600°F established the following conditions for obtaining the desired hydrogen contents:

Temperature °F	Pressure microns	Time minutes	Hydrogen Content ppm
1450	0.028	700	71
1500	0.02	730	57
1600	0.04	820	62

Analyses of the experimental results of tests made between 1200°F and 1500°F indicate that between 1 and 0.088 micron partial pressure of hydrogen, the dehydriding process is controlled by the second order reaction 2H (interstitial) = H_2 (gas) with an activation energy of 21,760±1840 cal. Below 0.088 micron partial pressure of hydrogen, the process appeared to be controlled by the rate of removal of H_2 molecules clinging to the powder surfaces. The type of vacuum system used for dehydriding is believed to be the major factor for removal of these molecules.

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SECTION I

INTRODUCTION

Titanium alloys have very attractive service properties (such as high strength, toughness, low density and corrosion resistance) for aircraft applications. However, the high unit costs for finished titanium alloy parts limit the full utilization of these materials because of economic considerations. Major factors controlling the cost of titanium alloy parts are raw materials and processing (such as machining) costs, and methods for reduction of these costs are of vital interest to the U. S. Air Force. The use of titanium alloy powders, instead of ingot material, for the production of metal products is a promising cost reduction method. The potential cost benefits from metal powder use are less machining costs and less scrap generation (5-10% of original weight for powder compared to 70-80% for ingot material). (Reference 1).

Several manufacturing processes have been used to produce titanium alloy powders. These are hydride-dehydride, rotating electrode process, atomization, electrolysis, "Durarc", mechanical attrition, and self attrition. At present only two of the above processes (rotating electrode process and hydride-dehydride) are established commercial processes. The hydride-dehydride process has the potential for producing titanium alloy powders at lower costs than the rotating electrode process because the hydride-dehydride process can utilize both scrap and new material of any geometry while the rotating electrode process requires prepared bar stock for producing powders. Therefore, from cost reduction considerations, the hydride-dehydride process for making titanium alloy powders was considered worthwhile for investigation.

Since hydrogen drastically reduces the mechanical properties of titanium alloys, an important step in the hydride-dehydride process is the removal of the hydrogen from the titanium alloy powder. A search of the literature showed very little detailed information on the temperature-time-pressure variations required for dehydriding titanium alloys.

The need of this data for proper evaluation of hydride-dehydride titanium alloy powders as a cost reduction method for making aircraft parts instigated studies in this area. The investigation described in this report was undertaken to determine practical processing conditions (temperature-time-pressure) for reducing hydrogen contents of hydrided titanium alloy (6246) powders to levels below 100 ppm.

SECTION II

THEORY

Many investigators (Reference 2) have studied the titanium-hydrogen system and obtained equilibrium diagrams, represented by Figure 1, at one atmosphere pressure. Reference 3 furnishes a temperature-pressure-concentration (Figure 2) of the system. No information was found in the literature on kinetics involved in hydrogen removal from or solution in titanium.

From the above limited guidelines, the following assumptions were made. On thermal decomposition in vacuum of hydrided Ti alloy 6246, the interstitial atoms of hydrogen will be eliminated as molecular hydrogen. In the temperature region of 649 to 871°C (1200 to 1600°F) both α and β phases will be formed and the elimination of hydrogen will be accompanied by a gradual reduction of the unit cell dimensions of both these phases. The evolution of hydrogen follows the general reaction:

$$\text{solid} \rightarrow \text{solid} + \text{gas} \tag{1}$$

The decomposition involves the following successive processes:

1. Lattice diffusion of interstitial hydrogen atoms at the solid-gas interface surface.

2. Combination of hydrogen atoms at the surfaces to give adsorbed hydrogen molecules.

3. Desorption of hydrogen molecules from the surface.

The overall velocity of a reaction is determined by the velocity of the slowest unit process of the reaction and establishes the time required for completion. Therefore, in this investigation, an attempt was also made to determine the dehydriding mechanism which controls the completion time for dehydriding Ti alloy 6246.

SECTION III

EXPERIMENTAL PROCEDURE

1. MATERIALS

The -45 mesh size hydrided powder of Ti-6246 alloy was obtained from Titanium Metals Corporation of America, the composition of which is given in Table I.

2. DEHYDRIDING

The schematic diagram of the complete set up for dehydriding is shown in Figure 3. The furnace used was a Hevi-Duty box electric furnace of Type G-153010-CUBH, which had a temperature control within ±10°F in the temperature range of 1200°F (649°C) to 1600°F (871°C). A 15 gm sample from hydrided alloy powder was weighed and kept in a 2-3 inch long titanium tube, the ends of which were crimped. This crimped titanium tube was kept in the quartz tube, which, in turn, was pushed into the constant temperature zone of the furnace. The temperature of the powder was measured by a chromel-alumel thermocouple, the tip of which was kept touching the titanium tube and the temperature was recorded on a Honeywell Recorder. The vacuum system had a 2 inch diameter diffusion pump and a Kinney mechanical pump Model KC-8 of capacity 8 cu.ft./min (227 liters/min). To measure pressure, an NRC thermocouple and emission regulated ion gauge control Type 710B was used. The whole system could reach a vacuum of 1.8×10^{-5} torr by leaving it overnight. Each time, the system was evacuated overnight before putting the quartz tube into the furnace. To stop the dehydriding, the quartz tube was pulled out of the furnace and was allowed to cool in air.

3. CHEMICAL ANALYSIS

The hydrogen analysis was performed by the chemical laboratory of the Air Force Materials Laboratory.

4. X-RAY DIFFRACTION

To study the phase change, if any, and change in size of unit cell of the phases as a function of hydrogen concentration, X-ray diffraction Debye-Scherrer patterns were taken using 114.6 mm camera and copper radiation with a Ni filter. Lattice parameters were calculated with the help of a computer program.

5. METALLOGRAPHY

Standard technique for mechanical polishing the samples, followed by chemical etching (10 ml Hf, 30 ml HNO_3, 50 ml water) was used.

SECTION IV

RESULTS

In the beginning of the present investigation, the dehydriding at different temperatures was done up to 0.1 micron pressure and the samples after dehydriding were analyzed for hydrogen concentration. The results of the hydrogen concentration as a function of temperature from these first series of experiments are shown in Table II from samples No. 1 to 8. It can be seen in Table II that as the temperature increases the concentration of hydrogen decreases up to 1500°F for the same value of partial pressure; for example, at 1207°F (653°C) the concentration of hydrogen is 0.0424% whereas at 1500°F (815.5°C) the concentration of hydrogen drops down to 0.0151%. This set of data indicated that to get the desirable hydrogen concentration in the alloy (less than 100 ppm) further dehydriding up to a value lower than 0.1 micron partial pressure of hydrogen is needed. Therefore, a new set of data were obtained at temperatures from 1350°F (732°C) to 1600°F (871°C) and were left overnight so as to attain minimum partial pressure of hydrogen obtainable with the vacuum system. The results of hydrogen concentration are shown in Table II from sample No. 14 to 19. Sample dehydrided at 1500°F up to 0.02 micron gives the minimum hydrogen concentration, i.e., 0.0057 wt. %. The dehydriding curves as a function of time at 1207°F and 1255°F from the first series of experiments and at 1350°F, 1500°F, and 1600°F from the second series of experiments are shown in Figure 4. In Figure 4, it is found that the higher the temperature the lesser the time required to reach the same partial pressure of hydrogen except at 1600°F, which shows a kind of inversion.

By "decomposition in vacuum" it is meant that the evolution of hydrogen begins in vacuum but that, as the reaction proceeds, the pressure of molecular hydrogen builds up, which in the experiments described was more than 1000 microns at the temperatures under consideration and then follows the decrease in pressure as a function of time. Figure 5 shows that within experimental error there is a linear relationship between x/(a-x) and the time; a is the initial interstitial hydrogen concentration (107 H atoms/100(Ti+Zr) atoms) and (a-x) the concentration after time t. The data points in Figure 5 are at and below 1.5 micron partial pressure of hydrogen. Though the hydrogen concentration was determined analytically at least at two or more partial pressures of hydrogen at one temperature, to determine more points, it was assumed that Sievert's law (concentration of hydrogen $\alpha \sqrt{pH_2}$ for dilute solutions) is obeyed, which is a valid assumption in the hydrogen concentration range of 0.0908 to 0.0057 wt. % hydrogen.

Table III summarizes the results obtained for dehydriding in vacuum; k, the second order velocity constant is given in units of (H atoms/100(Ti+Zr) atoms)$^{-1}$ sec^{-1} for different temperatures. These results were calculated by using the least squares program. The rate of decomposition in vacuum increases rapidly with increasing temperature up to 1500°F, and as shown by Figure 6, the plot of ln k against 1/T is approximately linear. The slope of the ln k vs 1/T line and the application of Arrhenius equation gives a value of 21,760 + 1840 cal. for the experimental activation energy, E.

To follow up the changes in the phases and their crystal structure and sizes at different hydrogen concentrations. Samples were taken by stopping the dehydriding at different partial pressures of hydrogen at 1255°F, i.e., at 25, 12, 1, 0.5 and 0.1 micron pressures. The hydrogen concentration at these partial pressures of hydrogen are shown in Table II, i.e., samples No. 9 to 13, and the results of room temperature X-ray diffraction patterns are shown in Table IV. In this table, for the sake of comparison, the results of the starting material and for the sample having only 0.0062 wt. % hydrogen (1600°F) are also included. Figure 6 shows the changes in size of unit cells of both α and β phases as a function of hydrogen concentration in the alloy. Also the change in lattice parameter of β-phase (bcc) is given in Figure 7. This change follows a straight line within experimental error. As the hydrogen concentration is decreased, the size of the unit cell in both α and β phases shrinks.

Figure 8 shows the microstructures of powders containing 224 ppm and 908 ppm of hydrogen in the alloy. Both the micrographs show α and β-phases but no hydride needles were observed.

SECTION V

DISCUSSION

If it is assumed that the rate-determining process in the thermal decomposition of Ti-6Al-2Sn-4Zr-6Mo hydride at 1207°-1500°F is the combination of hydrogen atoms at the solid surface,

$$2H\,(\text{interstitial}) \rightarrow H_2(\text{gas}) \qquad (2)$$

Then the experimental observation that the reaction is of the second order with respect to the interstitial hydrogen concentration is explained. It follows that, in making this assumption, we associate the experimental activation energy, E, with the process given by Equation 2. The energy of activation (Reference 4) may also be estimated by Eyring's empirical rule that this energy is about one-quarter of the energy of the bonds broken in forming the activated complex. The bonds broken in this case are the bonds from two hydrogen atoms to the surrounding titanium and the other atoms of the alloying elements. Assuming that the ΔH for $H_2(\text{gas}) = 2H(\text{in Ti})$ is zero, then the ΔH for $2H(\text{gas}) = H_2(\text{gas})$ is the bonding energy, i.e., 104.207 Kcal (Reference 5). One-fourth of this is about 26051 cal., in good agreement with the observed value of 21,760±1840 cal., considering the assumption made and dynamic vacuum being maintained on the system. From the values of diffusion coefficients for hydrogen in α- and β-phases in titanium, it appears that diffusion of hydrogen in the lattice is not a controlling factor in determining the kinetics of dehydriding of Ti-6Al-2Sn-4Zr-6Mo hydride powder.
The values of diffusion coefficient (Reference 6) (cm^2/sec) in titanium phases are:

$$D_\alpha = 1.8 \times 10^{-2} \exp\left(- \frac{12,380 \pm 680}{RT} \right) \qquad (3)$$

and

$$D_\beta = 1.95 \times 10^{-3} \exp\left(- \frac{6,640 \pm 500}{RT} \right) \qquad (4)$$

which shows that it takes about 14 seconds for hydrogen atoms to diffuse up to the surface of a particle of 100 microns diameter in the β-phase of titanium and about 22 seconds in the α-phase of titanium at 1500°F and at 1200°F, it takes 60 seconds in the α-phase and 25 seconds in the β-phase of titanium for the diffusion of hydrogen for the same size of particle. Though the values of diffusion coefficients will not be the same in the alloy under consideration, it seems unlikely to reduce the diffusion rates to an extent that it will become a controlling factor in kinetics. Therefore, diffusion of hydrogen is not a controlling factor in the kinetics of dehydriding the Ti-6Al-2Sn-4Zr-6Mo hydride alloy powder. However, at lower partial pressures, in Figure 5, the data points seem to deviate from straight line, for example, at 1207°F and 1255°F all the data points fall on the straight line (1 to 0.1 microns), at 1350°F, the last data point does not fall on the same line. At 1500°F, the data point below 0.045 micron does not fall on the same line, and similarly at 1600°F, data points below 0.075 micron deviate from the straight line. This indicates that at lower partial pressures of hydrogen, the controlling factor is not the breaking of hydrogen bonds at the surface. The time at lower pressures is much more than what it would have been if it was due only to second order reaction. The reason (Reference 7) may be that at lower pressures (less than 0.1 micron) the molecules no longer behave like fluids but like hard balls hitting along the walls of the tube and it takes much longer time to remove the molecules from the system, therefore, it will depend upon the geometry and type of vacuum system used. Also, at 1600°F, it takes a longer time to attain the same hydrogen concentration as compared to the hydrogen concentration at 1500°F. It may be due to the amount of β-phase present in the alloy. The $\alpha \rightleftharpoons \beta$ transus temperature for the Ti-6Al-2Sn-4Zr-6Mo alloy is 1720±20°F depending upon the oxygen concentration in the alloy. However, the presence of hydrogen will further bring down the $\alpha \rightleftharpoons \beta$ transus temperature. Therefore, at 1600°F, it is either all β or very close to all β-phase, in which the solubility of hydrogen is higher as compared to the α-phase (Reference 8). Since it has higher solid solubility for hydrogen, it will take longer time to achieve the same partial pressure of hydrogen as at 1500°F. Considering all these factors and on the basis of data obtained, it will not be wrong to conclude that 1500°F is the optimum temperature to dehydride the Ti-6Al-2Sn-4Zr-6Mo alloy hydride powder.

As shown in Table IV, the starting material is (Ti, Zr)H_{1+x} + β-phase, which is in good agreement with the results of the ternary Ti-Zr-H phase diagram (Reference 9) isothermal section at 750°C for 107 H atoms/100 (Ti+Zr) atoms. It appears from Table IV that within a few minutes, there is no longer a hydride phase in the alloy, but α and β-phases. Even at 25 microns pressure the hydrogen is in α and β-phases. In the process of dehydriding. hydrogen is removed from both the α- and β-phases, thereby giving rise to a decrease in the sizes of unit cells of both the phases as is shown in Figure 7.

In a single α-phase alloy, hydrogen is known to precipitate out at room temperature in the form of hydride needles because of the negligible solid solubility of hydrogen at room temperature. However, in the present alloy, no hydride needles were observed as shown in Figure 8. However, the amount of β-phase is higher in the powder which has 908 ppm hydrogen as compared to the alloy which has only 224 ppm hydrogen. For comparison, in Figure 9, the hydride needles can be seen in the Ti-6246 hydride particles.

SECTION VI

CONCLUSIONS

1. It has been established that Ti-6Al-2Sn-4Zr-6Mo alloy hydride powder can be dehydrided to a desired level of hydrogen concentration within a practical time (overnight) and the optimum temperature is 1500°F to dehydride. At 1500°F and 0.02 micron partial pressure of hydrogen, 57 ppm hydrogen concentration in the alloy has been obtained. Therefore, if the impurities are kept under control, the H/DH process will prove to be an economical process to reclaim scrap.

2. An attempt to determine the mechanism of dehydriding in dynamic vacuum between partial pressures of 1 micron and .088 micron shows that the kinetics is, most probably, controlled by the process:

$$2H(\text{interstitial}) = H_2(\text{gas})$$

and not by the diffusion of hydrogen in the lattice, which is very fast. The activation energy for the process between 1207° and 1500°F and partial pressures between 1 micron and .088 micron is 21,760±1840 cal. However, at lower partial pressures, the kinetics may be controlled by the geometry and type of vacuum system used.

3. The $(Ti,Zr)H_{1+x}$ phase in the starting material decomposes within a few minutes of the starting of the experiment at temperatures between 1207°F and 1500°F and then there is a decrease in size of the unit cells of both α- and β-phases with decrease in hydrogen concentration in the alloy.

4. The available titanium-hydrogen equilibrium data serve as a very useful basis for studying dehydriding of Ti alloy 6246.

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TABLE I

THE RESULTS OF CHEMICAL ANALYSIS ON HYDRIDED Ti-6Al-2Sn-4Zr-6Mo ALLOY POWDER

Element	Wt.%	Element	Wt.%
Copper	0.005	Carbon	<.01 not detected
Tin	1.71	Molybdenum	5.21
Zirconium	3.48	Hydrogen	1.9
Aluminum	5.48	Oxygen*	0.109
Iron	0.07	Nitrogen*	0.013
		Titanium	rest

*Oxygen and nitrogen could be determined after removing hydrogen from the alloy

TABLE II

RESULTS OF CHEMICAL ANALYSIS FOR HYDROGEN IN Ti-6Al-2Sn-4Zr-6Mo ALLOY AFTER DEHYDRIDING AT DIFFERENT TEMPERATURES AND PRESSURES

S. No.	Temp. °F	Hydrogen Partial Pressure in Microns	Hydrogen Concentration in	
			Wt.%	atoms/100(Ti+Zr) atoms
1	1207	0.1	0.0424	2.43
2	1255	0.1	0.0359	2.045
3	1300	0.1	0.0285	1.64
4	1350	0.1	0.0225	1.28
5	1400	0.1	0.0224	1.28
6	1450	0.1	0.0230	1.30
7	1500	0.1	0.0151	0.86
8	1600	0.15	0.0234	1.33
9	1207	0.5	0.0973	5.54
10	1255	25.0	>.15	-
11	1255	12.0	0.134	7.632
12	1255	1.0	0.0908	5.172
13	1255	0.5	0.0582	3.425
14	1350	0.088	0.0207	1.18
15	1350	0.038	0.0123	0.7
16	1400	0.026	0.0099	0.564
17	1450	0.028	0.0071	0.388
18	1500	0.02	0.0057	0.325
19	1600	0.04	0.0062	0.353

TABLE III

SLOPE IN $\left(\frac{\text{H atoms}}{\text{100(Ti+Zr) atoms}}\right)^{-1}$ SEC^{-1} AS A FUNCTION OF TEMPERATURE

Temperature °F	(k) slope in $\left(\frac{\text{H atoms}}{\text{100(Ti+Zr)}}\right)^{-1}$ sec^{-1}
1207	2.292×10^{-5}
1255	4.070×10^{-5}
1350	5.466×10^{-5}
1500	12.885×10^{-5}
1600	6.322×10^{-5}

TABLE IV

LATTICE PARAMETERS FOR THE PHASES AS A FUNCTION OF HYDROGEN CONCENTRATION IN Ti-6Al-2Sn-4Zr-6Mo ALLOY

Partial Pressure of Hydrogen		25 µm	12 µm	1 µm	0.5 µm	0.1 µm	0.04 µm*	Hydride Powder
Hydrogen Concentration in Wt.%		.15	0.134	0.908	0.0582	0.0359	0.0062	1.9
$\frac{H}{100(Ti+Zr)}$	Ratio	-	7.632	5.172	3.425	2.045	0.353	107
α-Phase	a	2.9506±.0019	2.9402±.0025	2.9399±.0022	2.9330±.0015	2.9328±.0024	2.9313±.0022	Shows TiH$_{1+x}$ phase with body centered tetragonal str. (10) with a_o = 3.12 c_o = 4.18 + β-Ti(bcc)
	c	4.6998±.0054	4.6900±.0035	4.6861±.0048	4.6995±.0025	4.6900±.0035	4.6844±.0043	
	V	35.436±.046	35.111±.046	35.075±.047	35.010±.025	34.935±.040	34.858±.040	
β-Phase	a	3.2999±.0013	3.2692±.0033	3.2669±.0013	3.2653±.0006	3.2606±.0025	3.2594±.0023	
	V	35.934±.043	34.939±.055	34.868±.042	34.816±.018	34.666±.079	34.628±.073	

*This sample taken after dehydriding at 1600°F

(10) H. Jaffee, J. Metals, 8,861 (1956)

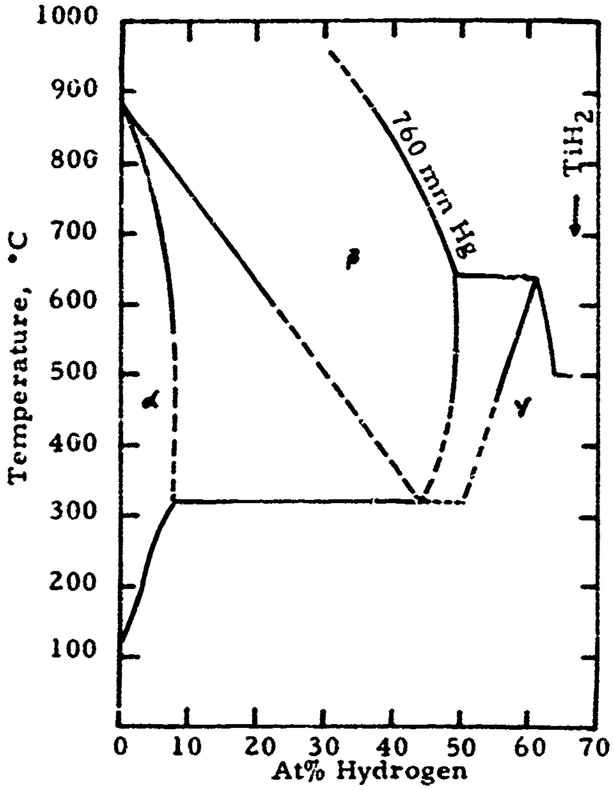


Figure 1. Ti-H Phase Diagram

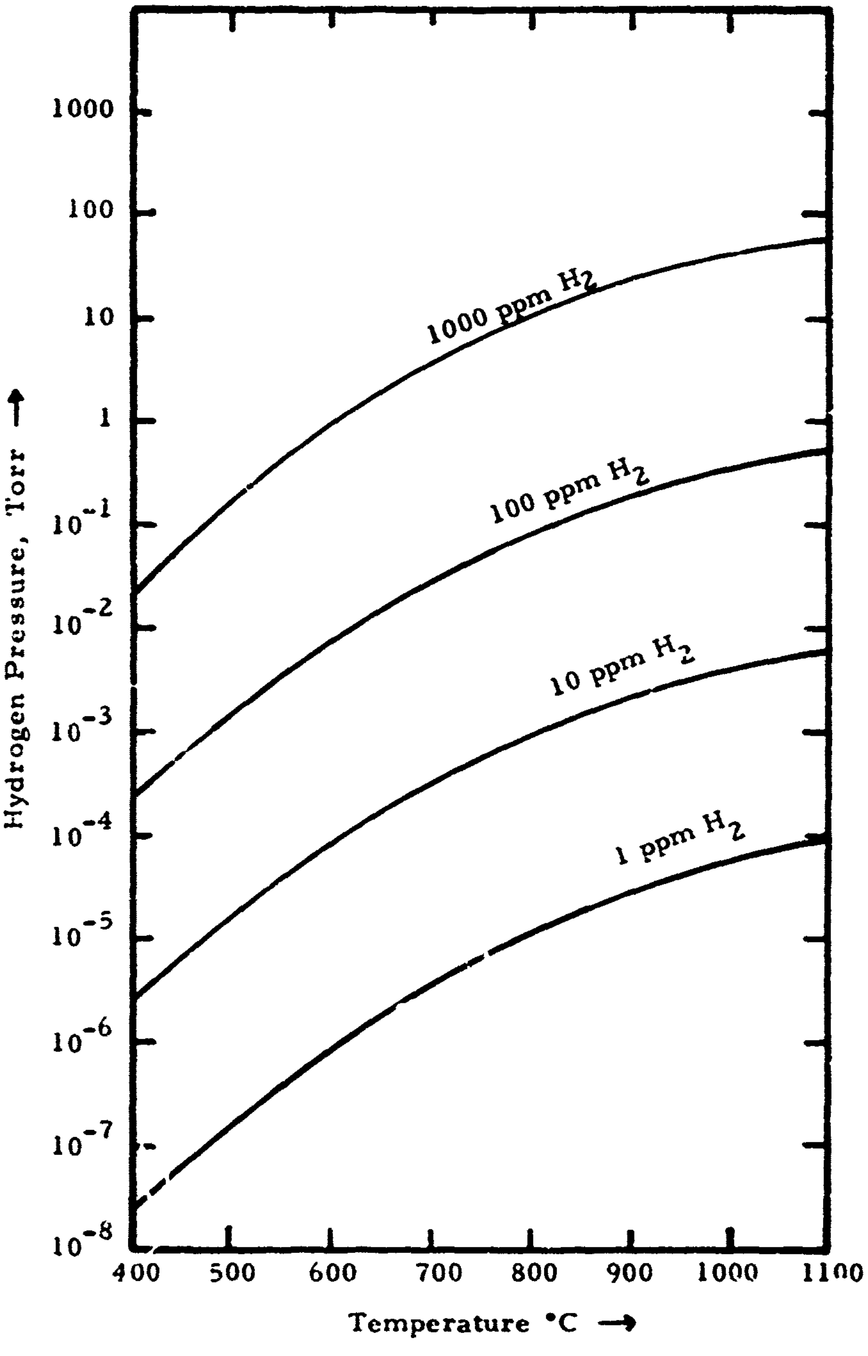


Figure 2. Equilibrium Pressure/Temperature/Concentration Behavior of Hydrogen in Pure Titanium

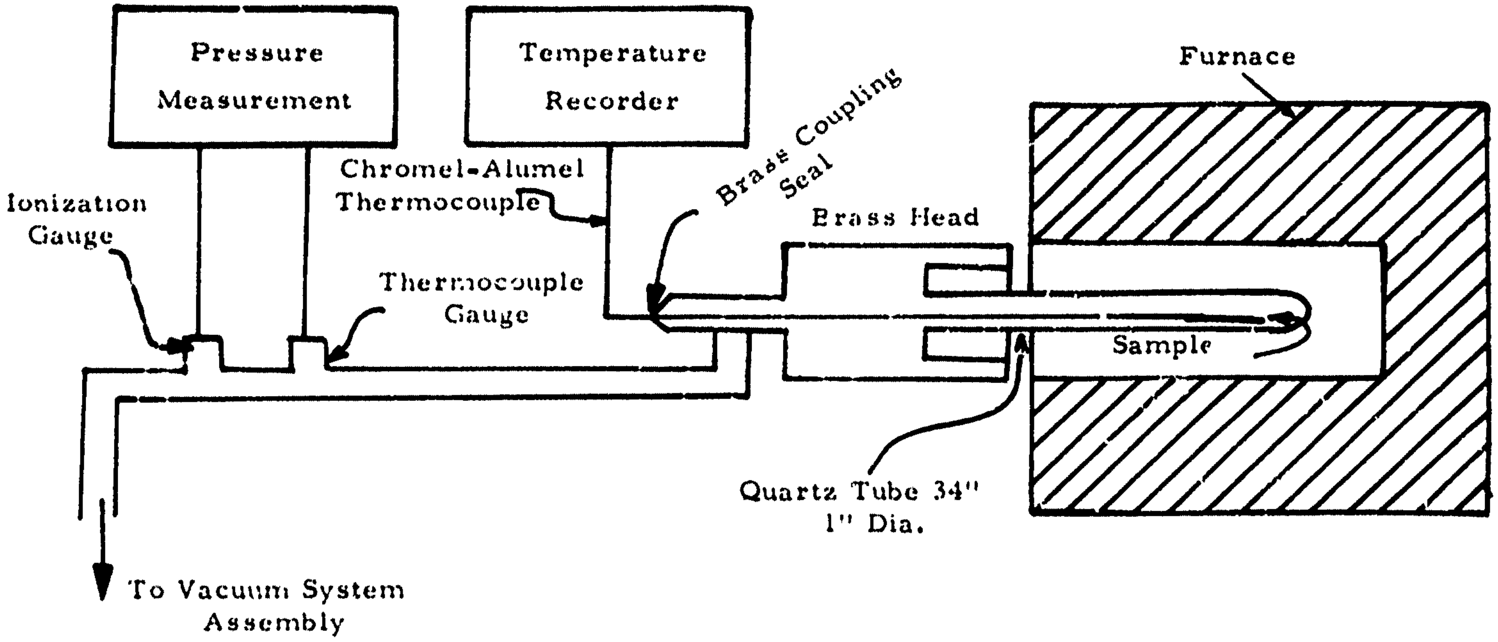


Figure 3. Schematic Diagram of Dehydriding Apparatus

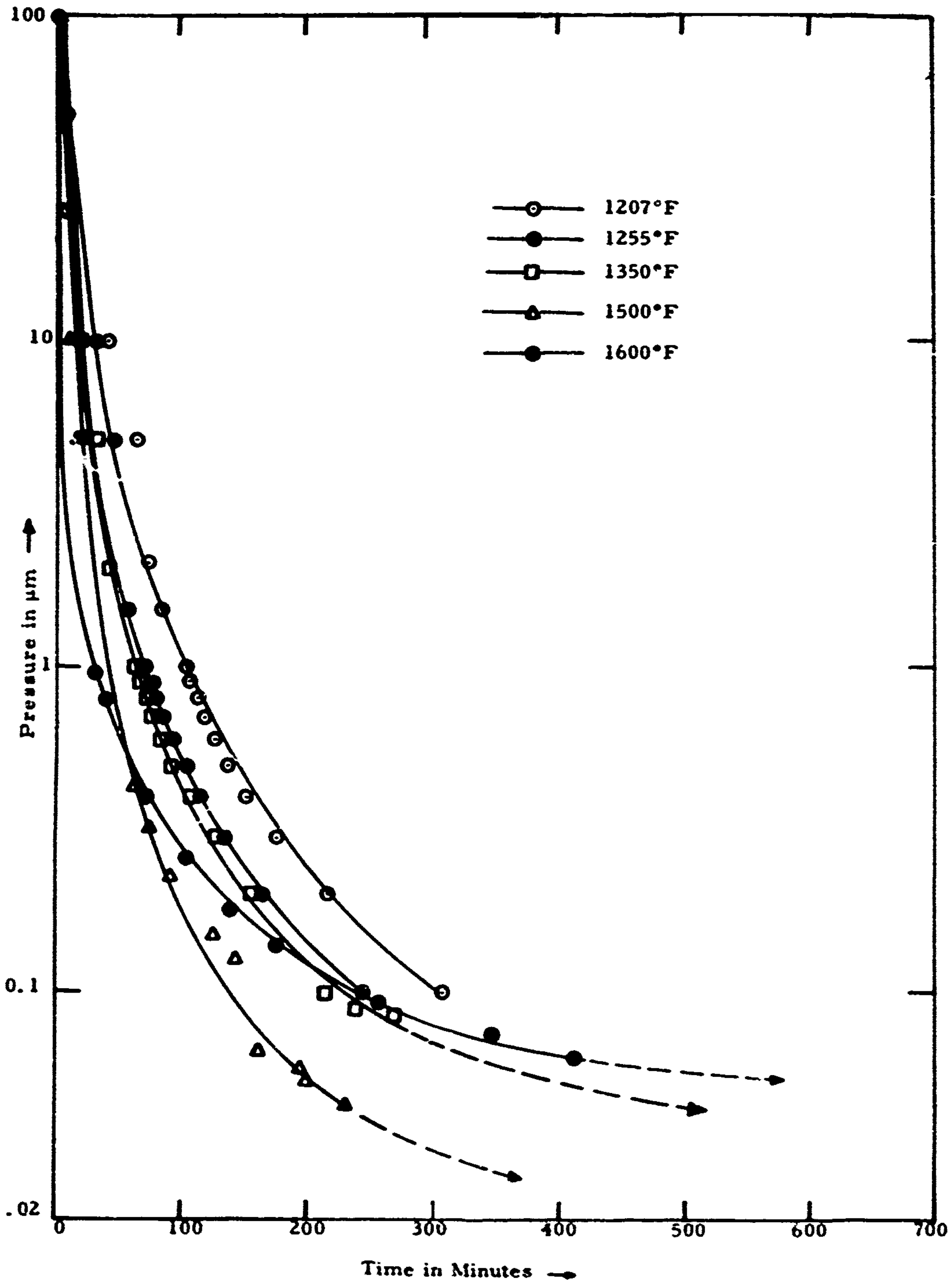


Figure 4. Partial Pressure of Hydrogen as a Function of Time at Constant Temperatures

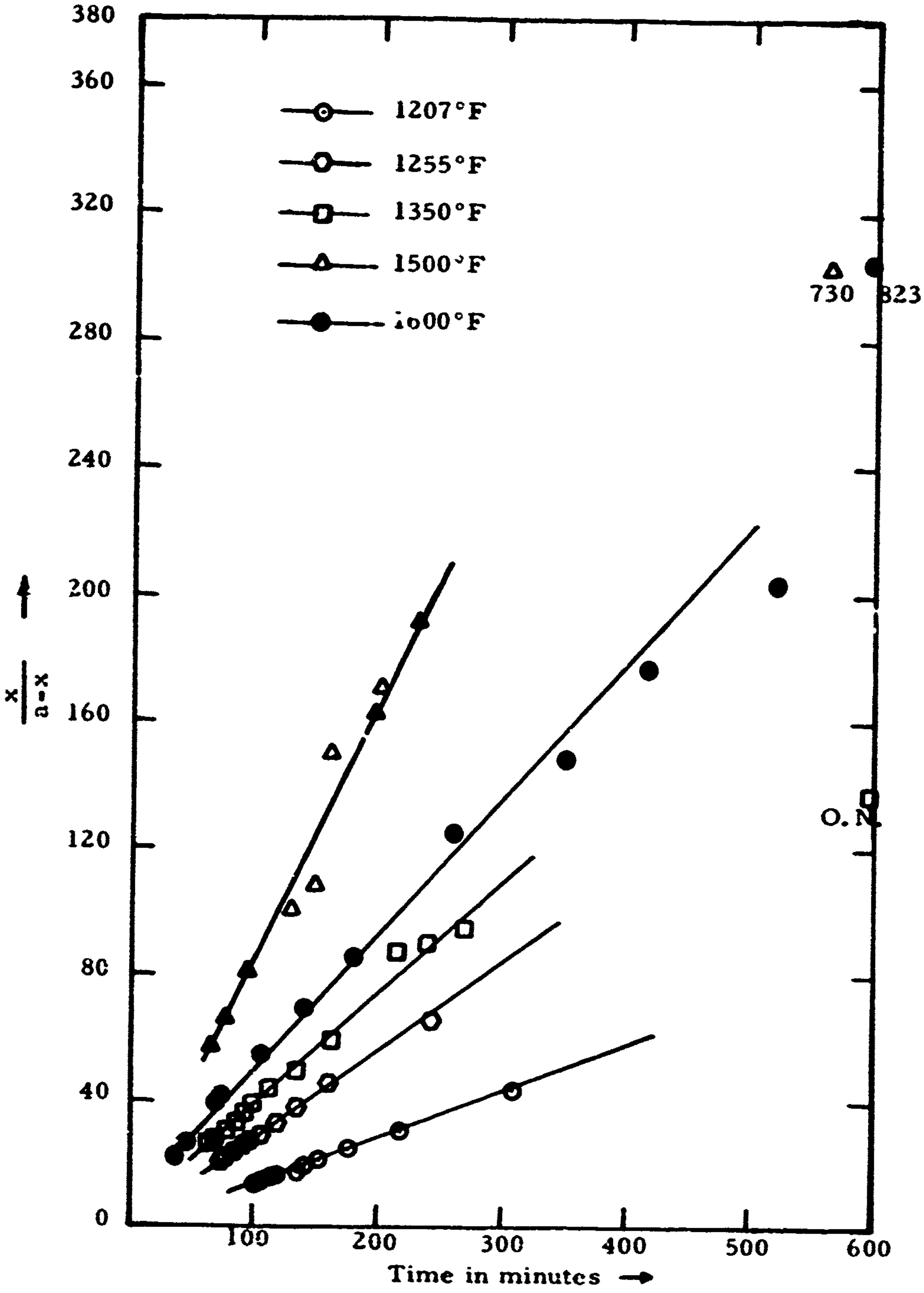


Figure 5. x/a-x Vs Time for Dehydriding of Ti-6Al-2Sn-4Zr-6Mo Hydride Powder (O.N. = Overnight). The Data Points on the Right Hand y-Axis Show the Time Required to Reach That Value of x/(a-x)

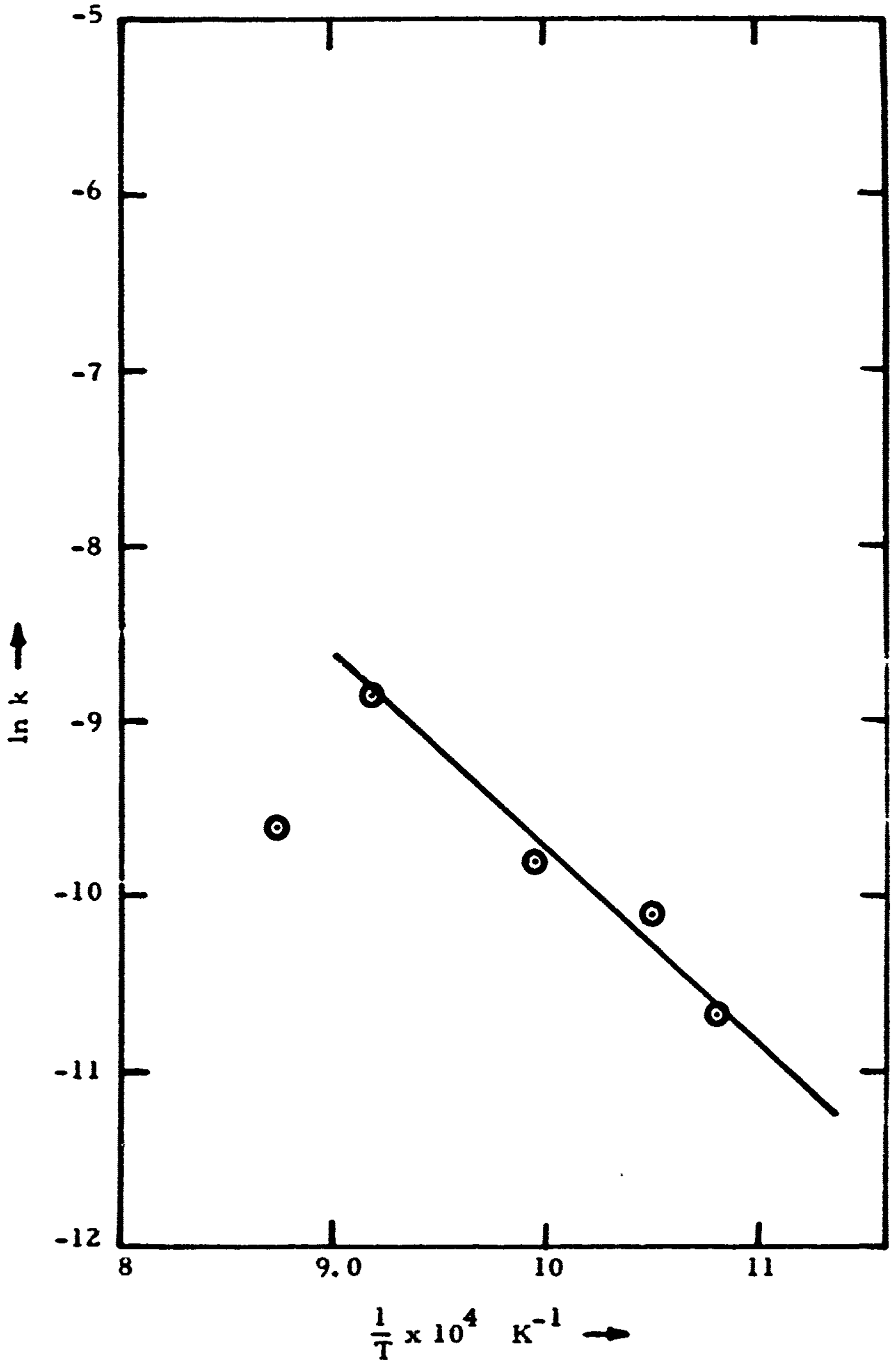


Figure 6. ln k Vs 1/T for Dehydriding of Ti-6Al-2Sn-4Zr-6Mo Hydride

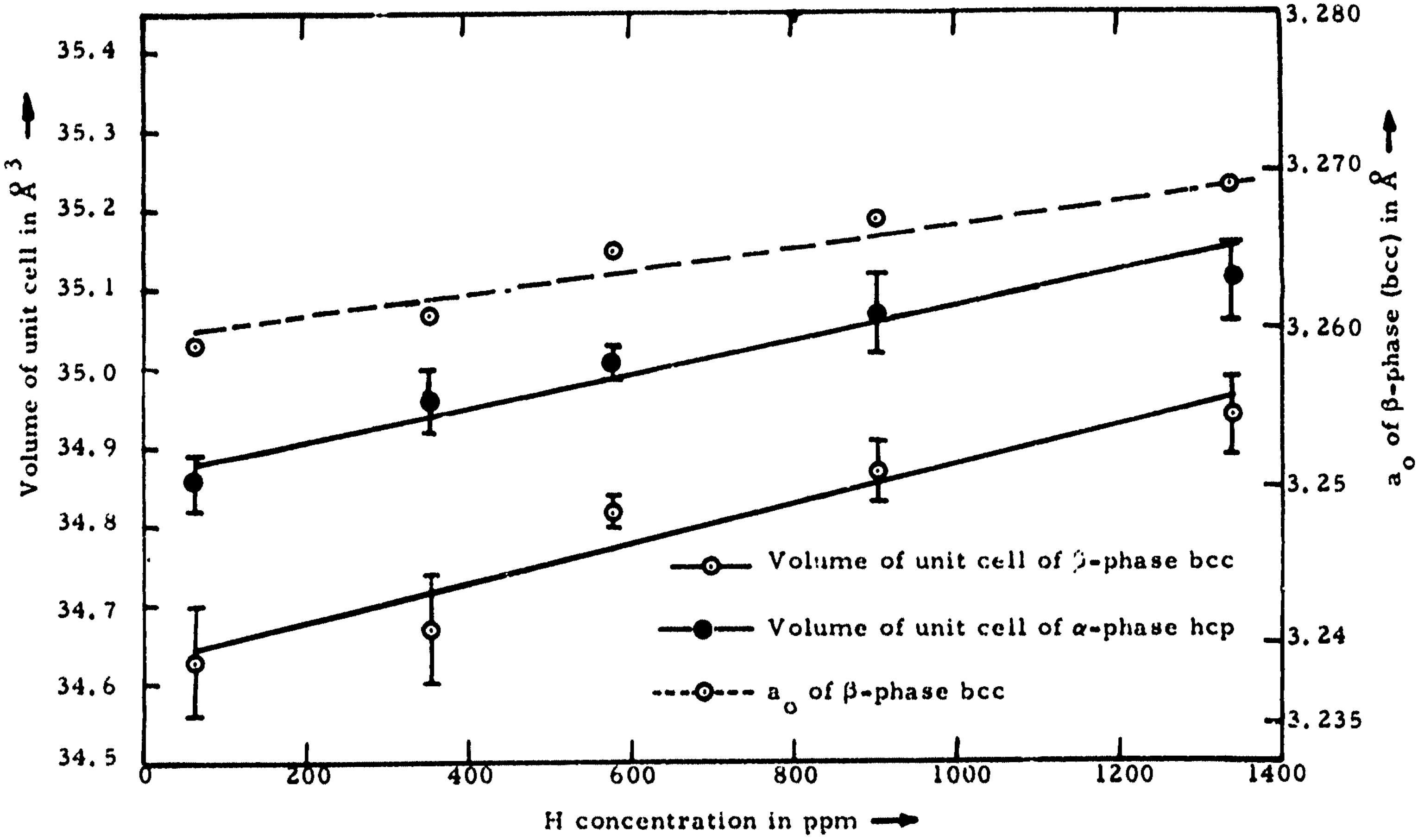
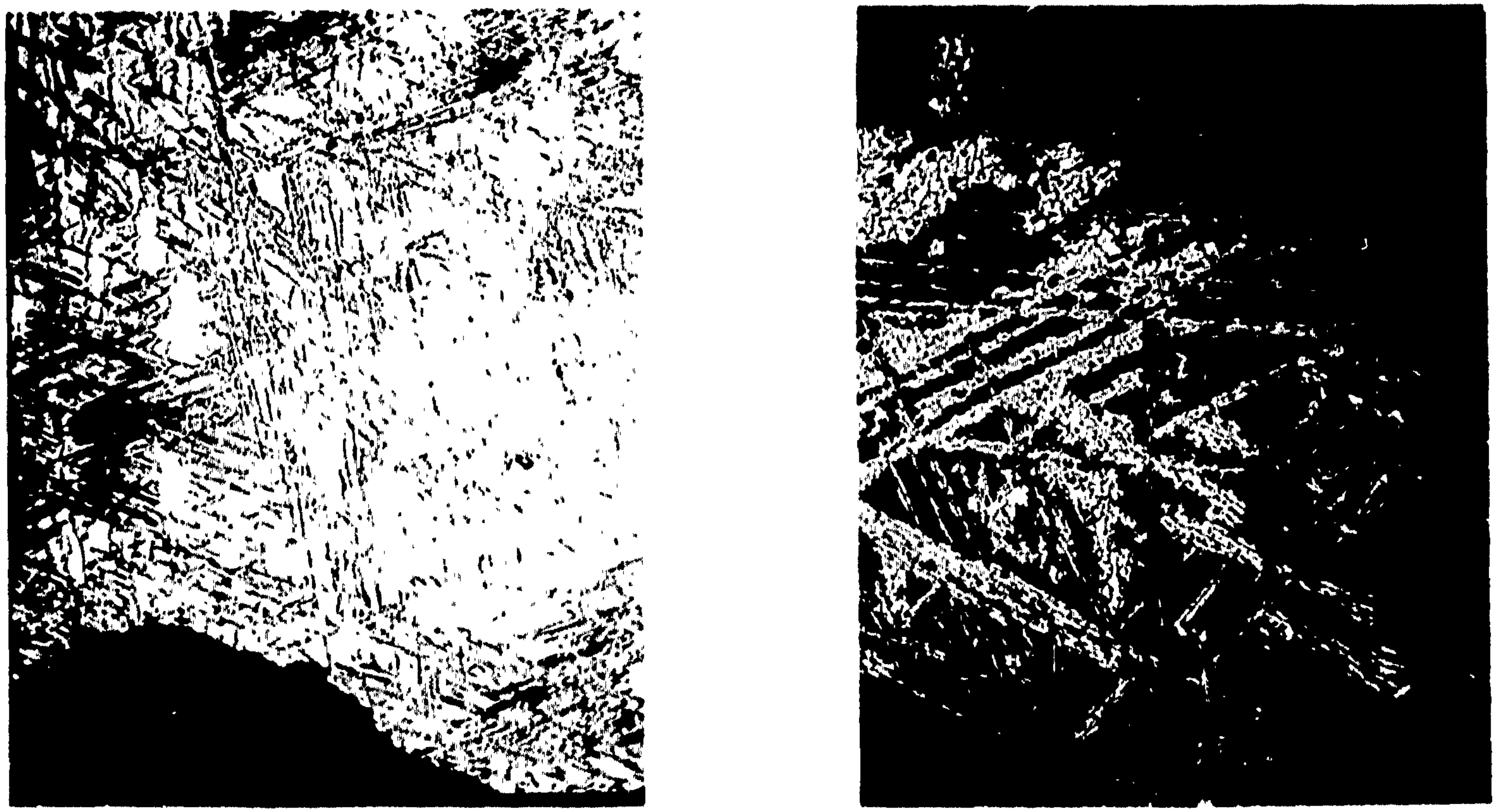


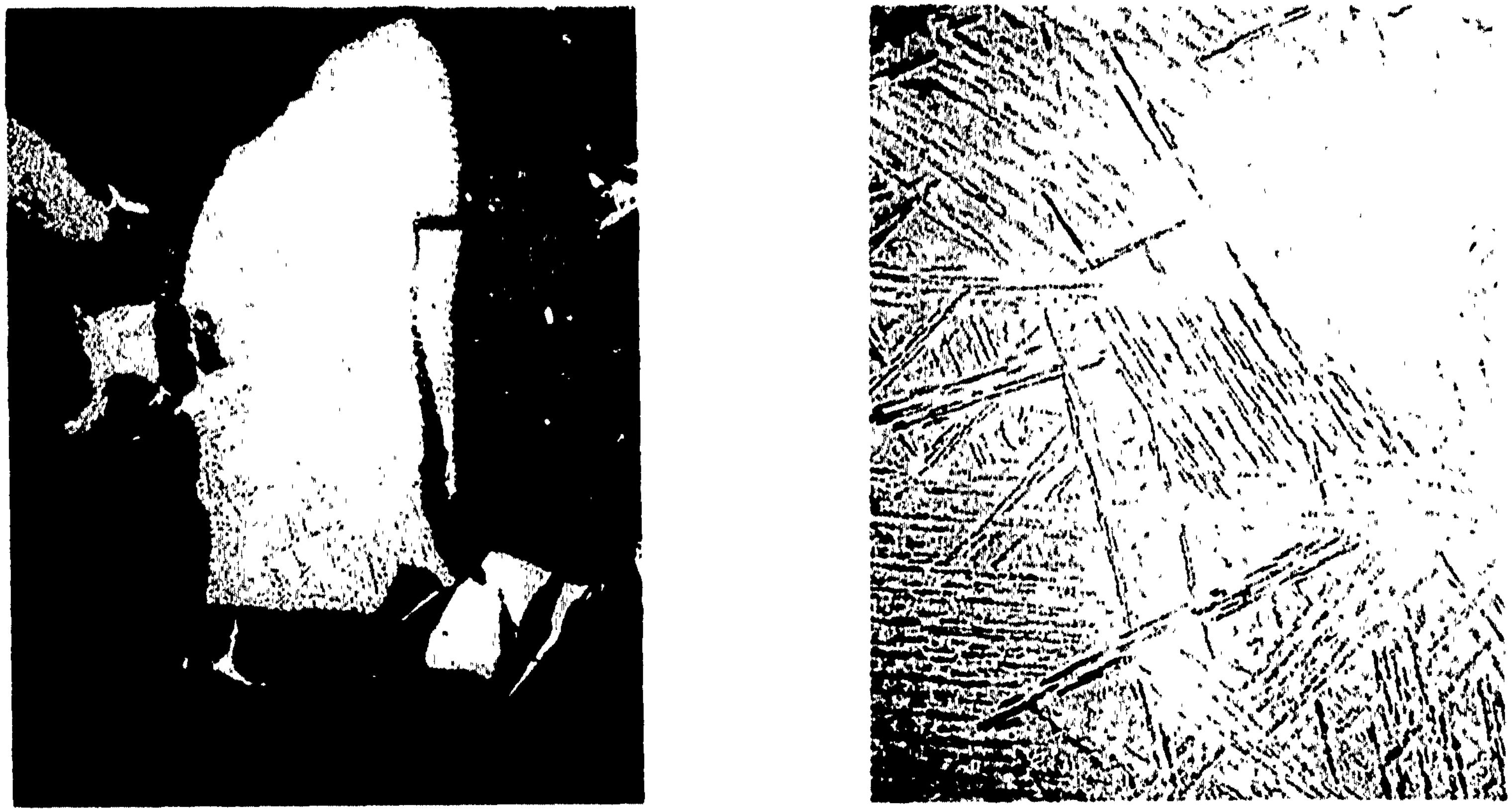
Figure 7. Unit Cell Volume and Lattice Parameter as a Function of Hydrogen Concentration in Ti-6Al-2Sn-4Zr-6Mo Alloy



(a)

(b)

Figure 8. Microstructures of Dehydrided Ti-6Al-2Sn-4Zr-6Mo Powder; (a) Containing .0224 Wt.% Hydrogen, (b) Containing .0908 Wt.% Hydrogen 500X



(a) 100X

(b) 500X

Figure 9. Microstructures of Hydrided Ti-6Al-2Sn-4Zr-6Mo Powder

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