

IRON TITANIUM HYDRIDE: ITS FORMATION,  
PROPERTIES, AND APPLICATION

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Iron and titanium form two known stable intermetallic compounds, FeTi and Fe<sub>2</sub>Ti.<sup>1</sup> It is also generally accepted that a third compound, FeTi<sub>2</sub>,<sup>2</sup> exists above 1000°C, decomposing to FeTi and Ti below that temperature. We have briefly noted previously<sup>3,4</sup> that one of these compounds, FeTi, will react directly with hydrogen to form an easily decomposed hydride which may be useful as a hydrogen storage medium. Our primary purpose here is to discuss the Fe-Ti-H system in some detail with particular emphasis on the reaction of FeTi with hydrogen and the formation and properties of two ternary hydrides, FeTiH<sub>~1</sub> and FeTiH<sub>~2</sub>. A further, though secondary, purpose is to discuss the application of iron titanium hydride as a storage medium for hydrogen fuel.

I. FORMATION AND PROPERTIES

Experimental

The Fe-Ti alloys were prepared from zone-refined Fe and Ti in an arc furnace under an argon atmosphere, although it should be noted that no significant differences were observed when commercial grade Fe and Ti were substituted for the zone-refined starting material. Initially we had prepared the alloys in an induction furnace; however, it appeared that the resulting products were contaminated by the alumina crucible material, which had an inhibiting effect upon their reaction with hydrogen. Contamination of iron titanium alloys by alumina crucibles has been noted previously.<sup>1</sup> All the alloys were

quite brittle and could be easily crushed to pass through a 10-mesh screen. It was not necessary to carry out the crushing step in an inert atmosphere. The samples, weighing ~10 g, were introduced into a high pressure hydriding reactor, the construction of which has been previously discussed in detail.<sup>5</sup>

Our procedure for hydriding metals which form unstable hydrides, as in this instance, has also been described,<sup>6</sup> and only a brief synopsis will be given here. The reactor was loaded with the granular alloy samples, sealed, evacuated and then heated to 400-450°C while outgassing continuously. Upon reaching the cited temperature range, hydrogen was admitted to the reactor until the pressure was ~100 psia. After ~30 minutes the reactor was evacuated and cooled to room temperature and H<sub>2</sub> was admitted to the reactor until the pressure was ~950 psia. Usually the metal-hydrogen reaction proceeded immediately with the evolution of heat. If no reaction took place over a course of ~15 minutes the above procedure was repeated. It should be noted that if the alloy is in ingot form, rather than granular, the initiation of the reaction is somewhat more difficult and may require several such treatments. In order to obtain a highly active metal substrate, the sample was hydrided and dehydrided several times. Dehydriding was accomplished by outgassing and heating to ~200°C.

The procedure for obtaining the pressure-composition isotherms presented here is essentially the same as that described previously.<sup>5,6</sup> Briefly, it consisted of equilibrating the metal hydride with hydrogen at >900 psia at a pre-determined temperature. Hydrogen was then withdrawn in a measured amount from the system by venting to an evacuated reservoir of known volume, after which equilibrium was allowed to become re-established. This step was repeated until the equilibrium pressure was below 1 atmosphere, at which point the sample

was heated to  $>400^{\circ}\text{C}$  and any further hydrogen that evolved was measured. Finally, the sample was cooled to room temperature, removed from the reactor and analyzed for Fe, Ti and residual hydrogen. Occasionally, the reverse procedure was followed in order to determine hysteresis effects. In these runs, the starting material was an alloy sample that had been activated by previous hydriding and dehydriding; and the points on the pressure vs. composition curve were obtained by adding successive small increments of hydrogen.

All the x-ray data were obtained using a 114.59 mm dia. Norelco powder camera (Debye-Scherrer type) and with Cu K  $\alpha$  radiation.

### Results and Discussion

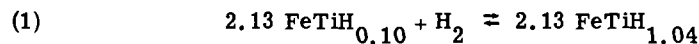
The Fe-Ti-H system was explored between the approximate limits, by weight, of 70% Fe-30% Ti and 37% Fe-63% Ti, corresponding to the atomic proportions  $\text{Fe}_2\text{Ti}$  and  $\text{FeTi}_2$ . At the iron-rich end of this range no hydrogen was absorbed, but all compositions richer in Ti than  $\text{Fe}_2\text{Ti}$  did take up hydrogen to some extent. The stable intermetallic compound, FeTi, reacted readily, and a series of pressure-composition isotherms for the FeTi-H system is shown in Figure 1. The composition of the starting alloy was 53.6% Fe and 46.7% Ti. The metal phase present should be only that of FeTi, which was confirmed by an x-ray diffraction pattern taken of the sample. In the graphs the equilibrium dissociation pressure of the hydride is plotted against the hydrogen content of the alloy expressed as the ratio of hydrogen atoms to that of the total number of metal atoms ( $\text{H}/(\text{Fe} + \text{Ti})$  or simply  $\text{H}/\text{M}$ ). The shape of the isotherms can be interpreted as follows: on the left, where the isotherms rise steeply as the hydrogen content of solid increases, is the region of solid solution of hydrogen in the FeTi metal lattice. This solid solubility region may be designated as the  $\alpha$  phase of the FeTi-H system. As the hydrogen

content of the solid is further increased the equilibrium pressure remains constant and forms, so to speak, a plateau. The composition at which the plateau begins marks the point at which a new phase appears and also marks the maximum solubility of hydrogen in the  $\alpha$  phase. At room temperature that composition corresponds to  $\text{FeTiH}_{0.10}$  ( $\text{H}/\text{M} = 0.05$ ). The new phase is the monohydride or  $\beta$  phase of the FeTi-H system. Both the  $\alpha$  and  $\beta$  phases coexist until the solid composition corresponds to  $\text{FeTiH}_{1.04}$  where the isotherms begin a steep ascent. At this point the  $\alpha$  phase has disappeared. (For the present we shall ignore the dip shown in the lower temperature isotherms just below this composition.) As the hydrogen content of the  $\beta$  phase is increased a new phase appears, the  $\gamma$  or dihydride phase. Its exact point of inception is temperature dependent and is somewhat difficult to determine since the upper plateaus are narrow and the breaks in the isotherms quite gradual. The  $55^\circ$  isotherm shows only a vestigial plateau structure and it appears that this temperature is quite close to the critical temperature, above which two discrete solid hydride phases cannot coexist and the monohydride is transformed continuously into the dihydride phase. The  $70^\circ$  isotherm shows no evidence of a plateau in this region. These data and observations are summarized in the phase diagram of the FeTi-H system shown in Figure 2.

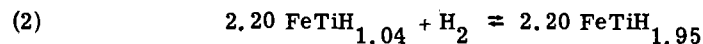
The effect of hysteresis in the FeTi-H system is illustrated in Figure 3. It is worth noting that the system almost forms two loops, the reason being that in the region of the pure  $\beta$  phase hysteresis is reduced substantially, which is the usual case in single phase regions. The loops are not closed since the composition range over which only the  $\beta$  phase is present is quite narrow. As the  $\gamma$  phase appears hysteresis again increases. It is also of interest to point out that the dip occurring in the lower temperature desorption

isotherms at H/M 0.5 does not occur in the absorption isotherm. This situation appears to be analogous to that occurring in the uranium-hydrogen system below 400°C, where a similar dip in the desorption isotherm was noted by Spedding et al.<sup>7</sup> and by Wicke and Otto.<sup>8</sup> Our results indicate that at higher temperatures the dip is less pronounced and finally disappears altogether in the 70° isotherm. This behavior is, again, similar to that of the uranium-hydrogen system, where Libowitz and Gibb<sup>9</sup> found no dips in isotherms determined at high temperatures (450°+). Wicke and Otto have suggested that the phenomenon is due to the supersaturation of hydrogen vacancies in the hydride phase, while Flotow et al.<sup>10</sup> have proposed that it is an effect caused by the state of subdivision of the solid.

The reaction taking place in the lower plateau region (H/M 0.10 to H/M = 0.52) may be written as follows



which is followed by



The variation of the log of the equilibrium dissociation pressure with the reciprocal temperature for several solid compositions is shown in Figure 4. The relationship is linear and obeys the van't Hoff equation in the form of  $\ln P = \frac{A}{T} + B$  where A and B are constants and T is the absolute temperature. Thermodynamic values for the iron titanium-hydrogen system were derived from these data and are shown in Table 1. They are given as relative partial molal quantities,  $(\bar{X}_H - \frac{1}{2}X_{H_2}^O)$  where  $\bar{X}_H$  is the partial molal enthalpy (entropy or free energy) of hydrogen (as atoms) in the solid relative to hydrogen in its standard state as a pure diatomic ideal gas at a pressure of 1 atm.

The products of reactions 1 and 2 are gray metal-like solids, essentially not different in appearance from the granular starting alloy. They are very brittle but are not pyrophoric in air; on the contrary, exposure of these materials to air tends to deactivate them. Even though both hydrides have dissociation pressures appreciably above one atmosphere at 25°, they will decompose relatively slowly in air. Once exposed to air they may be reactivated by repeating the procedure described in the experimental section, but a precautionary note should be added here; the remaining hydrided material, though apparently inert, will decompose quite rapidly as elevated temperatures are reached (200°-300°) and should not be heated to such temperatures in closed systems unless the free volume is sufficient to accommodate the evolved hydrogen without the buildup of excessive pressure.

The existence of the ternary compound  $\text{TiFeH}_1$  is strongly supported by evidence gathered from x-ray diffraction patterns of material whose composition lies in the lower plateau region of the P-C isotherm. In order to obtain a suitable sample for x-ray analysis, an amount of  $\text{FeTiH}_{\sim 1}$  was cooled to -70° and the hydrogen remaining in the gas phase was quickly evacuated; the sample was then contacted with air and allowed to warm to room temperature. This, in effect, "froze" the hydrogen content of the solid at a high level since such a procedure deactivates the hydride by air contact, thus preventing its rapid decomposition even at relatively high temperatures under conditions far removed from equilibrium. A portion of this material was taken and its x-ray diffraction pattern determined. The remaining material was analyzed for hydrogen by heating and decomposing it. While it is quite possible that the x-ray sample was not of the same composition as the bulk of material, or suffered some subsequent decomposition after sampling, as long as its composition

was within the lower plateau region, the pattern due to the  $\beta$  hydride phase should be discernible. We have followed this technique with material of a composition corresponding to  $\text{FeTiH}_{0.89}$ . Upon subtracting the lines due to  $\text{FeTi}$  ( $\alpha$  phase), the ternary hydride phase  $\text{FeTiH}_1$  was indexed as having tetragonal symmetry with  $a = 3.18^\circ\text{A}$  and  $c = 8.73^\circ\text{A}$ , giving a  $c/a$  ratio of 2.74. The observed and calculated d lines are given in Table II.

In addition, the density of another sample having composition corresponding to  $\text{TiFeH}_{0.80}$  was measured under benzene and found to be 6.003. Since this is a mixture of two phases, hydrogen saturated  $\text{TiFe}$  and  $\text{TiFeH}_1$ , and the density of  $\text{TiFe}$  is known (6.50), the density of  $\text{TiFeH}_1$  was calculated to be 5.88. Knowing the density and using the lattice parameters given above, the number of molecules in a unit cell were calculated to be 2.99 or 3. That the calculated value is so very close to an integral number is substantial evidence that the indexing treatment is correct.

The situation with solids of higher hydrogen contents, i.e. in the  $\beta$ - $\gamma$  region is more complex and has not been resolved. A sample of the higher hydride was treated the same as outlined above; however, this material is more unstable than the lower hydride and decomposes more rapidly. We have taken an x-ray pattern at room temperature of material whose bulk composition was  $\text{FeTiH}_{1.75}$ , but it is quite possible that some decomposition took place in the process of obtaining the diffraction pattern. Only 5 lines were observed, which are listed in Table III. Three of these (2, 3, 4) are lines which are also observed with  $\text{FeTiH}_1$  and one (5) is a line observed with unhydrided  $\text{FeTi}$ . On this basis no identification can be made. However, it does appear that the crystal structure is quite similar to that of the monohydride phase; a presumption which is supported by the low critical temperature of  $\beta$ - $\gamma$  two phase region.

For unequivocal results low temperature diffraction studies would have to be carried out.

The FeTi phase is homogeneous in the composition region extending from 45.9% Ti to 48.2% Ti. The equiatomic composition is 46.17% Ti; thus an appreciable amount of Ti can be dissolved in the intermetallic phase. This fact may be responsible for the behavior illustrated in Figure 5, in which the starting alloy was enriched in Ti to the extent that its initial composition, 49.3 wt/% Ti and 50.7 wt/% Fe, was slightly above that corresponding to the single phase region. The isotherm (B) has been significantly distorted, the equilibrium dissociation pressure markedly decreased and the boundary between the lower and higher hydride almost indistinguishable. Such a situation is not without precedent and a somewhat similar effect occurs in the LaNi<sub>5</sub>-hydrogen system when excess nickel is added to the starting alloy,<sup>11</sup> i.e. the dissociation pressure of the hydride is increased by a factor of over three upon increasing the nickel content of the alloy from LaNi<sub>4.90</sub> to LaNi<sub>5.5</sub>. Thus, in order to obtain reproducible behavior it is advisable to control the intermetallic composition as closely as possible. Upon increasing the Ti content to 63.2 wt/%, the pressure-composition isotherm (C) is greatly distorted as also shown in Figure 5. This alloy was annealed at 1000° for 12 hours, and then quenched in an unsuccessful attempt to prepare metastable FeTi<sub>2</sub>; but only FeTi and Ti were produced. After hydriding, an x-ray diffraction pattern of the product indicated the presence of FeTiH<sub>~2</sub>, TiH<sub>~2</sub>, TiFe and Ti. The increased amount of residual hydrogen in the solid is undoubtedly due to the presence of the stable titanium hydride.

Upon departing from the single phase region in the opposite direction, i.e. that of higher iron content, there appears to be no significant effect other than



a reduction in the amount of hydrogen sorbed as shown in Figure 5. The starting alloy was a two-phase mixture,  $\text{Fe}_2\text{Ti}$  and  $\text{FeTi}$ , having an overall composition of 60.5 wt/% Fe and 39.5% Ti. The isotherm (A) is essentially congruent with that obtained with  $\text{FeTi}$ , indicating little interaction between the two phases or solid solubility of Fe in  $\text{TiFe}$ ; an observation which is in accord with the known homogeneity range of  $\text{FeTi}$ . The amount of hydrogen actually sorbed is somewhat less than that expected from the proportionate amount of  $\text{FeTi}$  present in the alloy, which may be due to the mere physical presence of  $\text{Fe}_2\text{Ti}$ .

## II. APPLICATION TO HYDROGEN STORAGE

We have noted previously the advantage of using certain metal hydrides as a storage medium for hydrogen fuel.<sup>3, 4, 12</sup> Iron titanium hydride was included in this group and, as we became more familiar with its properties, it appeared to be an outstanding candidate as a hydrogen storage medium when measured against the following criteria: cost, hydrogen content and availability, and safety.

A simplified diagram in Fig. 6 shows a hydrogen storage reservoir of  $\text{FeTiH}_{\sim 2}$  integrated with a hydrogen burning energy converter. The heat of decomposition is supplied from the waste heat of the energy converter by circulating its coolant through a heat exchanger in contact with the metal hydride. The heat, of course, must be supplied at a rate consistent with the fuel demand of the converter. For example, if the reservoir temperature can be maintained at  $\sim 25^\circ\text{C}$  by the waste heat, the hydride is readily capable of delivering  $\text{H}_2$  fuel continuously, at high flow rates and at pressures in excess of one atmosphere. If the heat supply is insufficient the bed will progressively cool and the  $\text{H}_2$  flow will decrease until a balance is struck between the decomposition rate and the available heat. When the bed is exhausted it can be regenerated by following the opposite procedure, i. e., the bed is contacted with hydrogen at

a pressure substantially above the dissociation pressure, making due allowance for hysteresis (Fig. 3) and a coolant is circulated through the bed to remove the heat of reaction. In practice, the composition limits may be expected to range from  $\text{FeTiH}_{\sim 0.10}$  to  $\text{FeTiH}_{\sim 1.85}$  to give a total of 1.67 wt % available hydrogen.

We have demonstrated in the laboratory the feasibility of the scheme outlined above using a small Wankel engine and two metal hydride reservoirs each containing 1.5 Kg of  $\text{FeTiH}_{\sim 2}$ . The engine was a Sachs type KM48 with a maximum output of 8 H. P.; it was purchased from Sachs Motors Ltd., Pointe-Claire Dorval 700, Quebec, Canada. The alloy, FeTi, was part of a 73 Kg heat supplied by Cannon-Muskegon Corporation of Muskegon, Michigan. In lots of  $\sim 100$  Kg the price was \$5.15/Kg. The material contained about 7000 ppm oxygen which apparently affected the properties of the hydride in two important respects, i.e. the maximum hydrogen content was reduced and its equilibrium dissociation pressure was increased. However, for our specific purpose these changes were not of a critical nature. The reservoirs (Fig. 7) were constructed of stainless steel and had an i.d. of 1-3/4 in. and an overall length of 14 $\frac{1}{4}$  in. They were made from two 500 ml gas sampling cylinders (Hoke #4HS500) by fitting each cylinder with an outlet connection and sintered metal filter disk to prevent particle entrainment in the gas stream. Other ancillary equipment included a pressure regulator and rotometer, throttling valve, several pressure gauges and a dynamometer.

We have previously noted the performance of the Wankel engine using hydrogen fuel<sup>4</sup>; our purpose here is merely to demonstrate the application of iron titanium hydride as the hydrogen storage medium. We did not optimize the storage system in any way or quantitatively weigh such factors as kinetics,

heat transfer or thermal conductivity of the bed material, but merely chose a configuration which we qualitatively judged would be adequate to prove our thesis. The system, a photograph of which is shown in Fig. 8, differs from the ideal in one respect. For the sake of simplicity the waste heat of the engine was not extracted to provide the heat of decomposition of the hydride; rather it was supplied by immersing the hydride reservoirs in hot water.

The FeTi alloy was activated by the procedure outlined in Section 1. Following activation and several hydriding-dehydriding cycles, a composition corresponding to  $\text{FeTiH}_{\sim 1.8}$  could readily be attained. During the hydriding step, the reservoirs were exposed to hydrogen at a pressure of 60 atm and were cooled by immersion in an ice bath. After hydriding, the ice bath was replaced with hot water ( $\sim 50^\circ\text{C}$ ) and the reservoirs were connected to the engine intake manifold through the intermediate  $\text{H}_2$  flow control and monitoring devices. The hydrogen delivery pressure was regulated at about 25 psia which insured good throttling capability. The pressure at the engine intake manifold was slightly below atmospheric and no air regulation or supercharging was required. Lubricating oil, normally mixed with the gasoline fuel, was aspirated into the engine by the gaseous fuel mixture.

The total amount of hydrogen in the reservoir was about 54 g of which about 50 g were readily and continuously available. At a delivery pressure of 25 psia the maximum sustainable flow rate of hydrogen was  $\sim 40$  lSTP/min. Higher flow rates were possible but could only be maintained for short periods; this is attributed to a limitation to the rate of heat transferred to the reservoirs. It may be pointed out in this regard that the hydrogen evolution rate, per unit weight of alloy, is undoubtedly much higher than it would be in practice for a large, working hydrogen storage system. Nevertheless, if desired, an

increased decomposition rate could easily be realized through the adoption of a more efficient heat exchanger and/or a higher temperature heat source. Engine speed, which ranged from 1000-4000 RPM, was regulated by throttling the  $H_2$  flow. The dynamometer load was varied from 0 to 2 H. P.

Although the work described above is of a qualitative nature, there is no doubt that its essential purpose was successfully realized. Indeed, the results have given an added impetus to a program of a more ambitious scope, which has recently been initiated as a cooperative effort by Public Service Electric and Gas of New Jersey and Brookhaven National Laboratory. Its objective is to design and build a hydride storage reservoir capable of storing and delivering a minimum of 4.5 Kg of hydrogen. The reservoir will be part of a pilot unit designed to examine the feasibility of storing electrical energy via the production, storage and reversion of hydrogen. This will be accomplished by producing hydrogen electrolytically using off-peak power, storing the hydrogen in iron titanium hydride and finally decomposing the hydride to supply hydrogen to a fuel cell which will supply power for on-peak loads. The reservoir will have an internal diameter of 12 in. and will be ~6 ft long and will contain ~409 Kg of FeTi to give a maximum available hydrogen content of about 6.8 Kg. The unit will have an internal heat exchanger through which hot (~45°C) and cold (~17°C) water will circulate. The hydride will go through a complete sorption-desorption cycle once a day. Sorption will be carried out at a pressure of approximately 40 atm at a rate of 141 l STP/min. During the desorption portion of the cycle hydrogen will be delivered to the fuel cell at ~141 l STP/min regulated at a pressure of 3 atm.

Acknowledgment

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Table 1

Relative Partial Molal Quantities Per Gram Atom of Hydrogen

<u>Composition</u>	$(\bar{H}_{\text{H}-\frac{1}{2}\text{H}_2^0})$ <u>Kcal</u>	$(\bar{S}_{\text{H}-\frac{1}{2}\text{H}_2^0})$ <u>e. u.</u>	$(\bar{F}_{\text{H}-\frac{1}{2}\text{H}_2^0})$ <u>Kcal</u>
FeTiH <sub>0.1</sub> -FeTiH <sub>1.04</sub>	-3.36	-12.7	+0.42
FeTiH <sub>1.20</sub>	-3.70	-14.4	+0.57
FeTiH <sub>1.40</sub>	-3.98	-15.6	+0.65
FeTiH <sub>1.60</sub>	-4.03	-15.8	+0.68

Table II

d Spacings for  $\text{FeTiH}_1$ 

<u>Relative Intensity*</u>	<u>d obs.</u>	<u>d calc.</u>	<u>hkl</u>
40	2.2488	2.2488	110
20	2.1931	2.1931	004
100	2.1454	2.1472	103
10	1.9950	1.9990	112
50	1.5676	1.5644	201
20	1.2840	1.2840	204
20	1.2553	1.2474	007

\* by visual inspection



Table III

d Spacings Observed with  $\text{FeTiH}_{>1.04}$ 

<u>Line No.</u>	<u>Intensity*</u>	<u>Observed d Spacing, Å</u>
1	10	2.391
2	50	2.320
3	20	2.243
4	100	2.186
5	20	2.090

\* by visual inspection

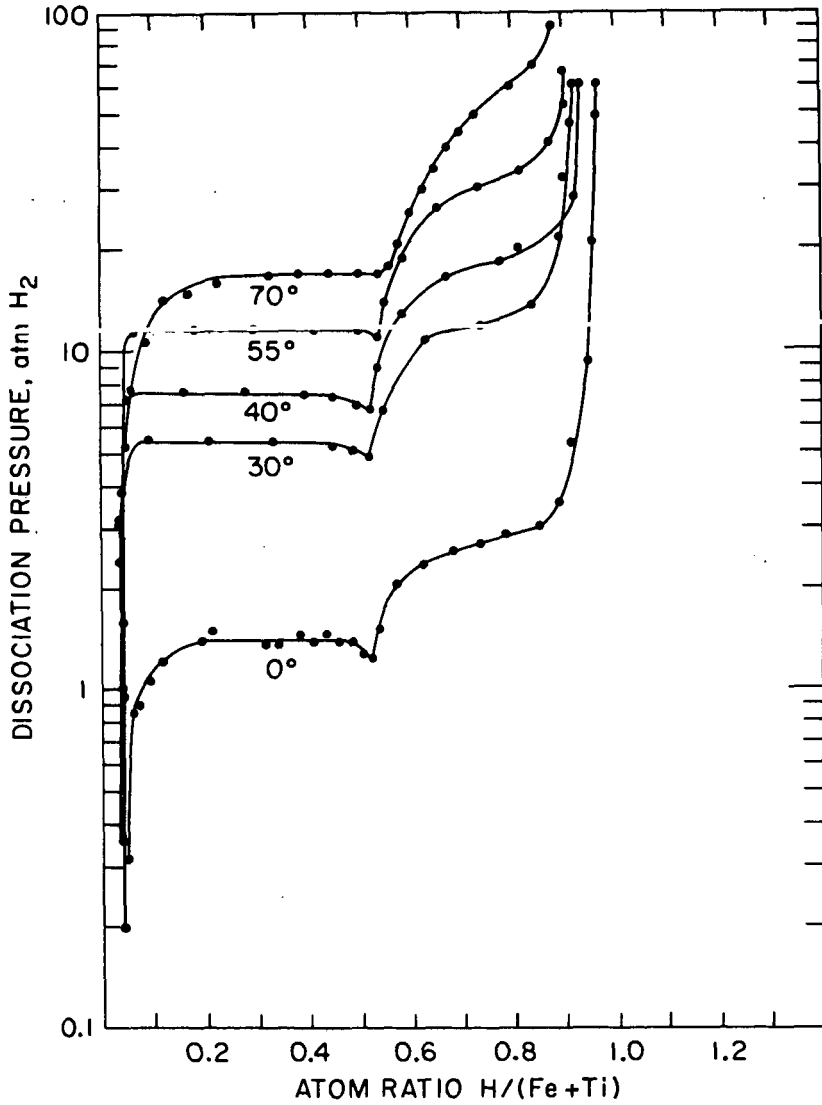


Figure 1 - Pressure-composition isotherm for the FeTi-H system. The initial alloy composition was 53.6% Fe and 46.7% Ti.

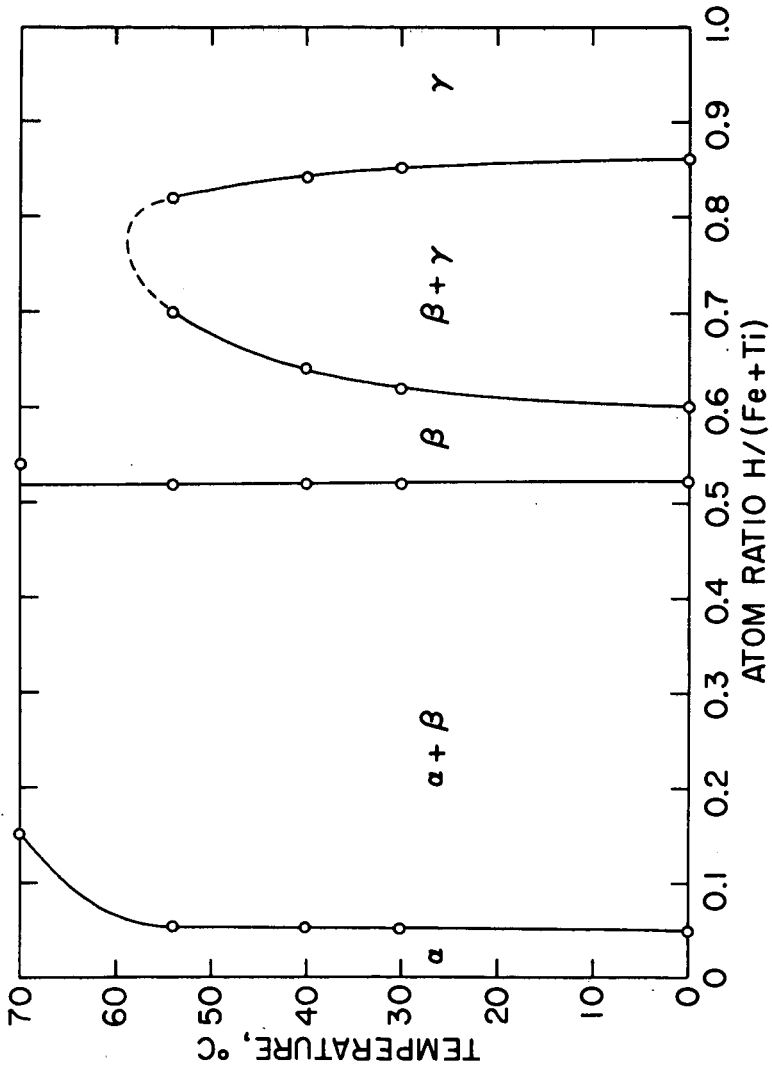


Figure 2 - Phase diagram of the FeTi-H system.

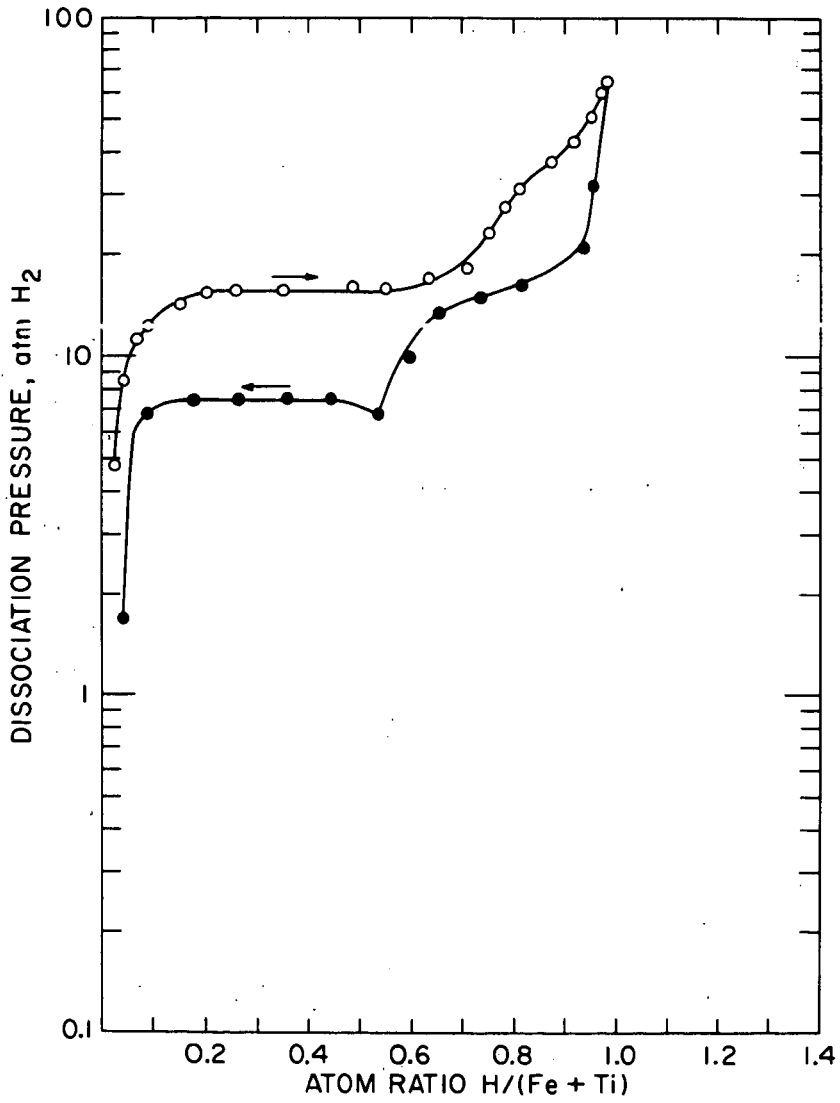


Figure 3 - Hysteresis in the FeTi-H system.

40°C

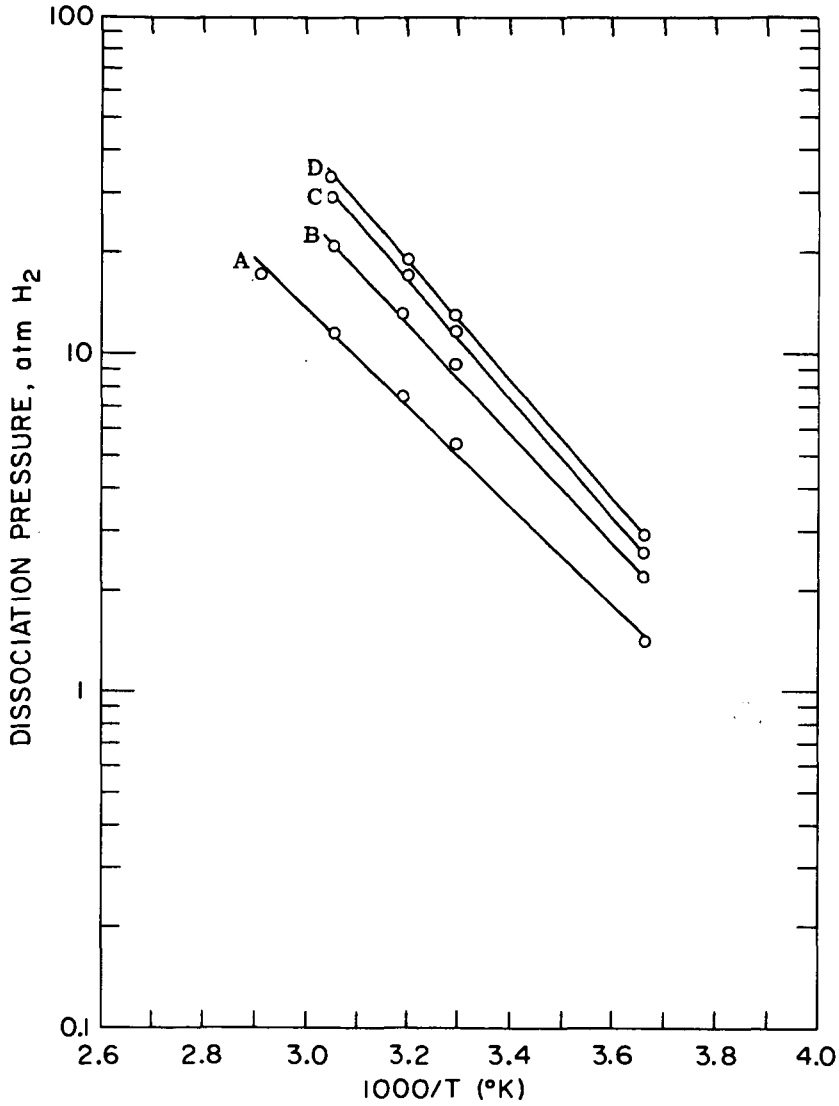


Figure 4 - Equilibrium dissociation pressure vs. reciprocal temperature for  $\text{FeTiH}_x$ .  
 A)  $\text{FeTiH}_{0.1}$ - $\text{FeTiH}_1$ ; B)  $\text{FeTiH}_{1.2}$ ;  
 C)  $\text{FeTiH}_{1.4}$ ; D)  $\text{FeTiH}_{1.6}$ .

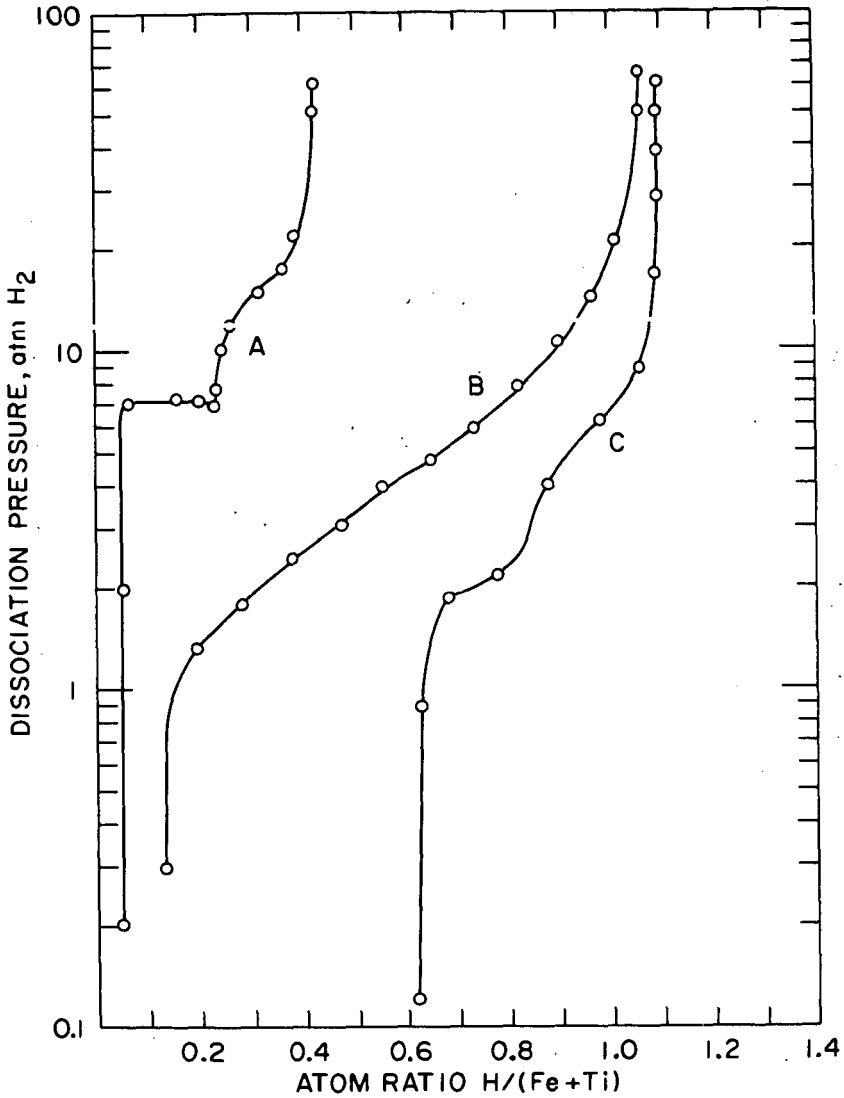


Figure 5 - Pressure-composition isotherms for alloys of various Fe/Ti ratios at 40°C. A) 60.5 wt % Fe, 39.5 wt % Ti; B) 50.5 wt % Fe, 49.2 wt % Ti; C) 36.7 wt % Fe, 63.2 wt % Ti.

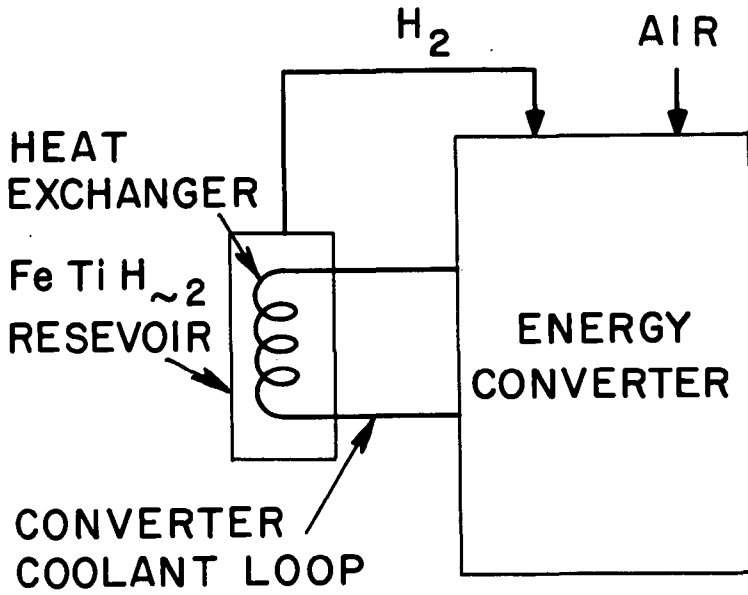
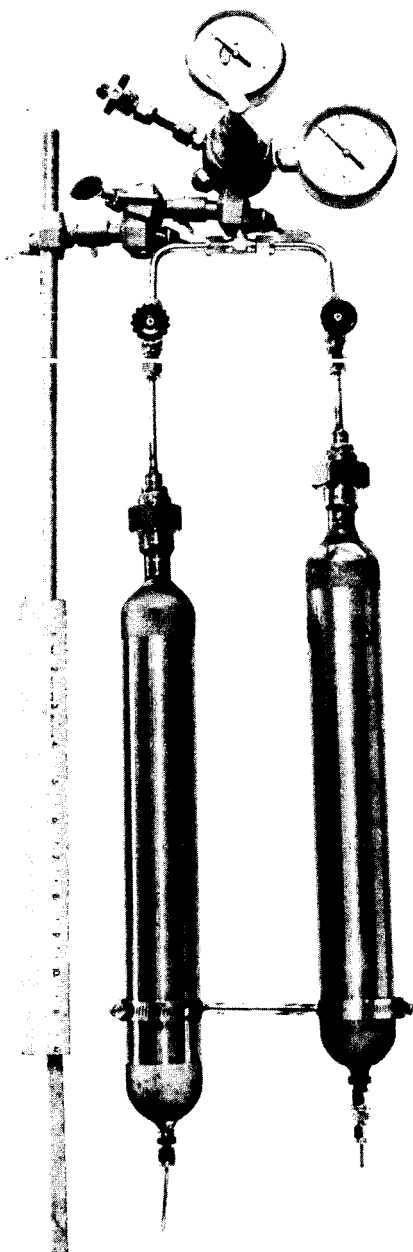


Figure 6 - Schematic of an Integrated Metal Hydride-Energy Converter System.



**Figure 7 - Metal Hydride Reservoirs - Each Reservoir Contains 1.5 Kg of  $\text{FeTiH}_{\sim 1.8}$**



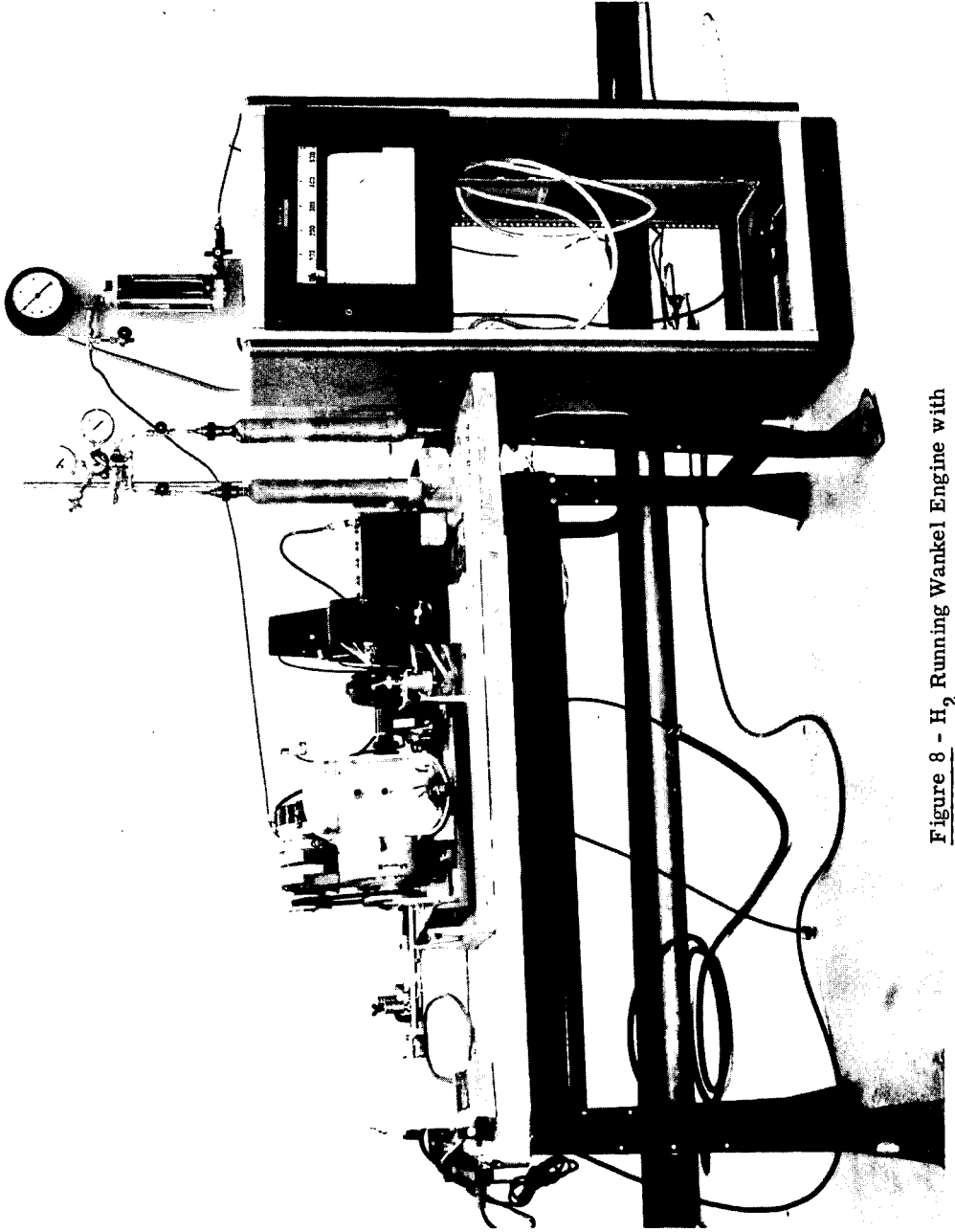


Figure 8 - H<sub>2</sub> Running Wankel Engine with Hydride Reservoirs.