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ELECTROLYSIS-BASED HYDROGEN STORAGE SYSTEMS

**SEMIANNUAL REPORT
JULY 1 TO DECEMBER 31, 1977**

JANUARY 1978

Work Supported by the
DIVISION OF ENERGY STORAGE SYSTEMS
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**BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.**

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SEMIANNUAL REPORT
JULY 1 TO DECEMBER 31, 1977

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JANUARY 1978

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ABSTRACT

The work described in this report was accomplished during the period July 1 - December 31, 1977, on a DOE-sponsored program aimed at improving the cost and efficiency of electrolytic hydrogen production and at developing the technology of metal-hydride hydrogen storage for stationary and transportation applications. The related work of organizations having sub-contracts with BNL is included, and the effort on systems studies and project management for DOE are summarized.

Work in the hydrogen production area includes hardware development and cell-materials testing for both acid and alkaline water electrolyzers. Also reported is related work on development and analysis of the reversible H_2-Cl_2 electrochemical cell, which is the key component in an electrical energy storage system proposed for utility use. Some results on a techno-economic assessment of this system are also given.

In the area of hydrogen storage, progress is reported on solutions to the hydride expansion problem, design of the Hydrogen Technology Advanced Component Test System, construction of two hydrogen reservoirs, characterization and production of FeTi-based hydrides, improved hydrides, and the Hydrogen Homestead.

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EXECUTIVE SUMMARY

Accomplishments are reported for the period July 1 - December 31, 1977, on a DOE sponsored program aimed toward improving cost and efficiency of electrolytic hydrogen production and developing technology of metal-hydride hydrogen storage for stationary and transportation applications. The related work of organizations subcontracting to BNL as well as systems studies and project management activities are summarized.

AREA 1: ELECTROLYTIC HYDROGEN PRODUCTION

1.1 Solid Polymer Electrolyte Water Electrolyzers (General Electric)

- Primary objective is the design and fabrication of 6 - 12 cell stack with active electrode area of 2.5 ft^2 .
- Consistent with cost reduction objective, major thrust has been realized via development of molded carbon and phenolic separator/current collector. This configuration permits use of gasketless seals further reducing costs.
- New design has been tested at 1 Amp/cm^2 (1000 Amps/ft^2) for over 1000 hrs.
- Electrocatalysts costs are reduced by use of lower cost anode catalyst and lowering noble metal loading by 50% at cathode side.
- Small cells (0.5 ft^2 area) have been tested at 150°C for > 5000 hrs exhibiting predicted improvement in efficiency.
- Fabrication of 12 cell module is in progress.

1.2 Advanced Alkaline Water Electrolyzers (Teledyne Energy Systems)

- The 5 cell test rig has been designed and fabrication will be completed by mid-January 1978.
- BNL studies indicate superior performance of nickel boride and nickel cobalt oxide over nickel catalysts for the hydrogen and oxygen evolution reactions.
- Potassium titanate and Nafion show excellent performance as separators although Nafion will require cost trade-off consideration.
- Nickel doped with lithium on mixed oxides may override time variation of performance at anode while sintered nickel impregnated with nickel hydroxide shows promise as electrocatalyst for oxygen evolution.

1.3 Advanced Concepts

- Studies continue to be aimed toward identification of low cost - high performance catalysts via examination and correlation of physical, electrochemical and performance characterization of a range of materials.

AREA 2: HYDROGEN STORAGE SUBSYSTEMS

2.1 Engineering Reference Designs and Process Systems

- Bechtel Corporation has initiated conceptual design and comparative cost studies on Hydrogen-Halogen Energy Storage System for electric load leveling/peaking application.
- Hydrogen storage costs are found comparable for compressed gas and FeTi hydride at <\$2.00/lb. FeTi from ilmenite ore can result in a cost breakthrough.

- Cost and performance estimates of $H_2 - Cl_2$ and $H_2 - Br_2$ systems show that $H_2 - Br_2$ systems are capable of providing ~5 hrs of electric energy storage on a 1/2 acre site versus 10 hrs for $H_2 - Cl_2$ system at similar cost.
- Foster Wheeler Energy Corporation has completed a preliminary design and cost study for Fe-Ti-Mn hydrides of a type suitable for Hydrogen-Halogen Energy Storage System.
- Three operating and/or design approaches to overriding metal-hydride expansion in bulk storage systems have been considered:
 - (1) hydrogen pulsing at start of charge
 - (2) use of heat transfer panels as shallow trays
 - (3) use of 3"-4" tubular hydride containers with heat transport fluid external to tubes.
- Construction has begun at FWEC on Variable Parameter Test Unit (VPTU), a 24" diameter x 84" long carbon steel vessel that will accommodate and permit test of the selected hydride storage configuration.

2.2 Process Test Beds

2.2.1 Utility Applications

- Additional tests with the 3" diameter metal hydride test bed (~5 diameters deep in vertical orientation) verifies that height of the bed is a critical parameter in overriding bed compaction due to expansion as expected.
- Apparatus has been set up to evaluate thermal conductivity enhancement by addition of various forms of aluminum or copper.
- Tests show that water vapor (108 ppm) in hydrogen reduces storage capacity by 60% after 36 cycles.

2.3 Hydrogen Technology Advanced Component Test System

- HYTACTS is an engineering-scale service system that will provide operational equipment to test: (1) reversible electrochemical storage/peaking systems; (2) storage systems for automotive applications; (3) characterization of advanced hydrides and their associated storage systems.
- A 2.5' long x 2' diam. carbon steel vessel designated as VPTU-1 is nearing completion at BNL. This test vessel will be used to evaluate and select the operating procedures and design configuration for metal hydride-hydrogen storage systems.
- Equipment has been placed on order, fabrication and assembly of piping assemblies is ongoing and HYTACTS flow diagram is being updated.

2.4 Other Contract Work

- Teledyne Continental Motors has submitted its final report (draft) identifying an in-plant fork lift truck as the lead candidate for hydrogen fuel and internal combustion engine and Fe-Ti hydride storage system.
- Stevens Institute of Technology was awarded a Special Research Support Agreement to continue work in thermal energy storage via investigation of benzene and hydrogen/cyclohexane reaction.
- Billings Energy Corporation was awarded a contract for construction of Fe-Ti based hydride-hydrogen storage system for the Hydrogen Homestead in Provo, Utah.

AREA 3: HYDRIDE MATERIALS DEVELOPMENT

3.1 Materials Characterization and Tests

3.1.1 Subcontracts

- International Nickel Company reports that alumino thermic reduction of ilmenite ore represents the lowest cost for FeTi production ($\sim \$1.60/\text{lb}$). Presence of 5-10 atom percent of Al reduces absorption capacity substantially. Good hydriding quality FeTi is obtained by direct melt of Ti and Fe at a cost of $\sim \$2.25/\text{lb}$.
- INCO reports on effects of contaminants on FeTi hydride storage capacity (mainly silicon and aluminum).

3.1.2 BNL Research

- Non-annealed manganese-substituted FeTi alloys have been characterized. All these materials show improvements in ease of activation.
- Ability to activate in situ with no heating translates to capital cost reduction of storage systems.
- Selection was made of $\text{TiFe}_{.085}\text{Mn}_{0.15}$ as manganese alloy best suited to H_2 -Halogen Electric Energy Storage System.

3.2 Improved Hydrides

3.2.1 Subcontract Work

- Denver Research Institute initiated program of applying DRI predictive techniques to identify and characterize improved hydrides.

- Air Products and Chemicals of Allentown, Pennsylvania, will soon be awarded contract following evaluation of responses to RFP "Development of New and Improved Hydrogen Storage Systems." Objectives of the program are to develop materials with ≥ 3 wt% storage capacity capable of delivering H_2 at 1 atmosphere at temperatures $\leq 175^\circ C$.
- Teitel Associates will be awarded a contract in response to RFP cited above to examine economics of hybrid microcavity/hydride storage system for automotive application.

AREA 4: TECHNOLOGY UTILIZATION

4.1 Hydrogen-Halogen

- GE work is virtually completed on .05 ft² cell with a chlorine/HCl storage subsystem capable of operating at elevated temperature and pressure. Phase I Summary Report was delivered.
- GE has carried out studies in small H_2 /Bromine cells indicating encouraging results for low-cost electrode/current collector configurations.
- BNL has conducted studies aimed toward characterizing H_2 /Halogen systems via heat and mass balance analysis as well as performance characterization of Nafion membranes.
- The data from the studies suggest low coulombic inefficiencies due to self discharge and overall system electric-electric efficiency $> 70\%$.
- Decision points will be addressed in FY 78 considering:
 - : H_2/Cl_2 vs. H_2/Br_2
 - : Techno-economic and safety factors
 - : Storage

AREA 5: SYSTEMS STUDIES

5.1 Natural-Gas Supplementation

- PSE&G of New Jersey has completed its study in support of Ad Hoc Committee mission and the report is in press.

5.2 Hydrogen Injection in Natural Gas

- PSE&G are examining gas blends, conservation testing and leakage testing.
- Evaluations to date do not indicate significant technological barriers to the concept of hydrogen supplementation of natural gas. The concept implementation calls for modification of distribution subsystems that should be tested "hands-on" in order to develop confidence in the industry as well as the consumer sector.

5.3 Characterization of U.S. Falling Water Resources

- Institute of Gas Technology (IGT) is conducting a short-term (3 mos.) study to assess the extent of falling water resource as may be associated with hydrogen production.
- Output of this study will be applied to evaluating the need for demonstration.

5.4 International Energy Agency Agreement on Electrolytic Hydrogen Production

- Accomplishments are identified as they relate to agreements and manpower commitments.

AREA 6: PROJECT MANAGEMENT

- Project planning and liaison activities are described.
- Two RFP's processed: (1) Development of New and Improved Hydrogen Storage Systems for Automotive Hydrogen Fuel Storage; (2) Underground Hydrogen Storage.

- Nine unsolicited proposals/preproposals were evaluated. Of these, six have been rejected two are pending and one (Clarkson College) was recommended for funding.
- Charts are submitted outlining budgetary distribution, areas of program emphasis and major milestones.

AREA 1: ELECTROLYTIC HYDROGEN PRODUCTION

1.1 Development of Solid Polymer Electrolyte Water Electrolyzers

The main objectives of the current program at the General Electric Company are the design and fabrication of a 6-12 cell stack with an active area of the electrode of $2\frac{1}{2}\text{-ft}^2$. The power input for a 12-cell module is approximately 50 kW. The task in the GE program, which is supported by DOE, Empire State Electric Energy Research Corp. (ESEERCO), and Niagara Mohawk Power Corp., is aimed at minimizing the capital cost and achieving high efficiencies. The greatest part of the cost reduction has been realized by the development of a molded carbon and phenolic separator/current collector to replace the transition-metal screens and separator sheet. Use of gasketless seals with this configuration further reduces the cell costs. Cells with this new design have been tested for over 1000 hrs at 1 Amp/cm^2 . The electrocatalyst costs have been reduced by the use of lower cost anode catalyst and the lowering of noble-metal loading from 4 mg/cm^2 to 2 mg/cm^2 on the cathode side. The causes for performance decay in cells with sulfonated trifluorostyrene as the electrolyte have been identified and corrective measures taken.

Small cells (electrode area $1/2\text{ ft}^2$) have been operated at 150°C for over 5000 hrs with the expected improvement in energy efficiency. More than 20 potential anode catalysts have been screened, the three most promising ones being subjected to longer term testing. The $2\frac{1}{2}\text{-ft}^2$ cell design has been completed and the fabrication of components for a 12-cell module is in progress. The development of $2\frac{1}{2}\text{-ft}^2$ membrane and electrode assemblies has been successfully completed. Samples cut out from the M+E assemblies were tested in small ($1/20\text{ ft}^2$) SPE water electrolysis cells and showed performance comparable to or better than the

base line laboratory cells (Figure 1.1). A mockup of the 50 kW-module is shown in Figure 1.2. This module will be in test by the middle of FY 1978.

BNL studies show a direct correlation between the optical properties of oxide films and their electrocatalytic activities for the oxygen-evolution reaction (Figure 1.3). Ruthenium dissolution occurs at 1.45 Volts versus the potential of the reversible hydrogen electrode (RHE). A method of stabilizing the ruthenium oxide is by the use of binary or ternary alloys of ruthenium as the electrocatalyst.

1.2 Development of Advanced Alkaline Water Electrolyzers

Teledyne Energy Systems (TES) completed the testing of polyphenylene sulfide, in fiber form made into a mat, as the separator material. The 5-cell test rig was designed, and fabrication is in the final stage (expected to be completed by the middle of January). This rig will be tested for three months at TES and then shipped to BNL for further testing.

In long-term studies at BNL, it was shown that nickel boride and nickel cobalt oxide are superior to nickel as electrocatalysts for the hydrogen- and oxygen-evolution reactions (Figures 1.4 and 1.5). Potassium titanate and Nafion also showed excellent performance as separator materials in these long-term studies. The time variation problem of anodes may be minimized by doping nickel with lithium or mixed oxides (NiCo_2O_4). Sintered nickel impregnated with nickel hydroxide shows promise as a good electrocatalyst for the oxygen-evolution reaction.

1.3 Advanced Concepts

The study of $\text{Li}_{0.08}\text{Ni}_{0.92}$ and anodized nickel as electrocatalysts for oxygen evolution was carried out over a wide range of temperatures (25-280°C). It was found that prior to oxygen evolution, a chemical transformation from a lower oxide to a higher oxide should occur in the surface film. Studies on the

correlation between the magnetic properties of electrode materials and their electrocatalytic activities for oxygen evolution were extended to $\text{Ni}_x\text{Ti}_{3-x}\text{O}_4$ spinels and Ni-Cu alloys. In the Ni-Cu alloys, a definite break in the transfer coefficient was observed near the Curie temperature of each alloy (Figure 1.6). The magnetization of the electrode probably caused only a secondary effect on the kinetics of the oxygen-evolution reaction.

Figure 1.1 Water electrolysis 48-hour performance comparison

Figure 1.2 Mockup of 50 kW Electrolysis Module

Figure 1.3 Variation of ellipsometric and reflectometric parameters with increasing anodic potential of a ruthenium electrode.

Figure 1.4 Variation of electrode potential with time for hydrogen evolution on nickel boride and nickel screen in 30% KOH at 90°C.

Figure 1.5 Variation of electrode potential with time for oxygen evolution on Teflon bonded NiCo_2O_4 and Ni screen in 30% KOH at 90°C.

Figure 1.6 Temperature dependence of the transfer coefficient for oxygen evolution on two Ni-Cu alloys.

AREA 2: HYDROGEN STORAGE SUBSYSTEMS

2.1 Engineering Reference Designs and Process Systems

A preliminary conceptual design and comparative cost study, based on BNL's reference design, was initiated with Bechtel Corporation on the Hydrogen-Halogen Energy Storage System for electric load leveling/peaking applications at the substation level. It was found that the cost of storing hydrogen as FeTiH_x was less than the cost of compressed-gas storage when the hydride is $< \$2.00/\text{lb}$. If a practical method of producing FeTi alloy from low-cost ilmenite ore ($12¢/\text{lb}$) can be developed, the cost is likely to be appreciably below $\$2.00/\text{lb}$. Another finding was that the use of either chlorine or bromine as the halogen has little effect on the cost. However, the $\text{H}_2\text{-Br}_2$ system is capable of storing only ~5 hours of electrical energy on existing half-acre substation sites, and is thus limited to a daily cycle.

Foster Wheeler Energy Corporation (FWEC) completed a preliminary design and cost study for a hydride storage subsystem (Fe-Ti-Mn type) based on BNL's hydride materials and test bed data and on reference process designs. This subsystem is the type suitable for an electric storage system such as the Hydrogen-Halogen Energy Storage System. Costs were based on the production of 25 plants per year and storage vessel/heat exchangers that are truck-transportable when loaded with the hydride. The following three approaches to a solution of the critical hydride expansion problem were considered and are not significantly different in cost: 1) periodic loosening of a deep bed ($>2\text{-ft}$ diam) by the use of hydrogen pulsing at the start of a charging run; 2) the use of heat-transfer panels as shallow trays having a depth less than that at which bed packing occurs (vertical vessel); and 3) the use of 3- to 4-in.-diameter horizontal tubes as hydride containers, with the heat-transport fluid external to these tubes.

Construction was started on the Variable-Parameter Test Unit (VPTU) which is a 24-in.-diameter carbon-steel vessel capable of accommodating any of the above three internal assemblies.

2.2 Process Test Beds

2.2.1 Utility Applications

Additional experiments were made with the 3-in.-diameter test bed in studying how to prevent vessel damage due to hydride expansion. When the vertically mounted vessel was vigorously vibrated to compact the bed (~5 diameters deep), thus simulating its condition after transportation to the site of use, it was not possible to loosen the bed by the usual hydrogen pulsing technique using a 580-psia pressure pulse. Evidently, the density at the vibrator zone (bottom) was high enough to require a more energetic pulse. It seems probable that the test conditions were considerably more severe than those which might be incurred. Previously it had been demonstrated that a hydride bed which had become packed due to cycling could be loosened by a 500-psia pulse.

With the vessel positioned horizontally (80% full) and the bed at its maximum density after cycling, the bed was loosened by a 100-psia hydrogen pulse. No expansion of the bed had occurred, and none is expected provided ~20% free space is allowed for expansion.

A simple apparatus was set up for use in evaluating the enhancement of bed thermal conductivity due to the addition of various forms of aluminum or copper; an appreciable increase in the overall heat-transfer coefficient is expected.

In a test on the effect of water vapor on FeTiH_x , it was shown that hydrogen containing 108 ppm of water vapor reduced the storage capacity by 60% after 36 hydride-dehydride cycles.

2.3 Hydrogen Technology Advanced Component Test System (HYTACTS)

This engineering-scale service system will eventually provide all of the operational equipment for the testing of:

a) an energy storage system utilizing a reversible electrochemical cell coupled to a hydrogen reservoir containing a metal hydride; and b) energy storage systems for highway vehicles, using metal hydrides or other hydrogen storage media.

The HYTACTS flow diagram was revised and equipment was ordered to provide for the high hydrogen flow rate required for loosening a self-packed hydride bed, thereby preventing container damage due to hydride expansion. A diaphragm-type compressor (20 SCFM) was purchased for use in recycling of hydrogen will be bought as commercial grade (99.8%) and purified to 99.999% in the system.

The first (2.5-ft long, 2-ft diam.) carbon-steel vessel, designated VPTU-1, is nearing completion at BNL and will be used for testing the three methods of solving the hydride expansion problem. A sketch of the vessel set up for Method 1 is shown in Figure 2.1. Most of the piping assemblies for the flow-metering section were fabricated, but are not yet assembled. The 7-ft. long vessel (VPTU-2), now being fabricated at FWEC will be used for testing the preferred method of accommodating hydride expansion.

2.4 Other Contract Work

Teledyne Continental Motors (TCM) identified an in-plant fork-lift truck as the best candidate for evaluating an internal combustion engine fueled by hydrogen stored as an Fe-Ti based hydride. A preliminary reservoir design was completed, and the necessary engine modifications were identified in their final report (draft).

A Special Research Support Agreement was awarded to Stevens Institute of Technology to continue work on their investigation of the benzene + hydrogen/cyclohexane reaction for use in thermal energy storage. The work will be monitored by BNL and Sandia Laboratories (SL) so that it can be coordinated with the heat-pipe program of General Electric Company.

Billings Energy Corporation was awarded a contract for construction of a hydride (Fe-Ti based) storage system for use in their Hydrogen Homestead in Provo, Utah. The kitchen range, fireplace log, barbeque, tractor and automobile will be powered by hydrogen produced by an electrolyzer using hydroelectric power generated locally. The Homestead was inaugurated on November 10, 1977, and the storage system was near completion at the end of December.

Figure 2.1 Sectional views of variable-parameter test unit 1 showing major components

AREA 3: HYDRIDE MATERIALS DEVELOPMENT

3.1 Materials Characterization and Testing (Ti Systems)

3.1.1 BNL Research

Non-annealed manganese-substituted ferrotitanium alloys of the general formula $\text{Ti-Fe}_x\text{Mn}_y$ have been completely characterized as to their p-c-T, initial activation, and charging properties for the Reference Design Conditions of the regenerative Hydrogen-Chlorine Energy Storage System. The specific alloys investigated were as follows: TiFe(NL-2A) , $\text{TiFe}_{0.9}\text{Mn}_{0.1}$, $\text{TiFe}_{0.85}\text{Mn}_{0.15}$, $\text{TiFe}_{0.80}\text{Mn}_{0.20}$, $\text{TiFe}_{0.70}\text{Mn}_{0.30}$, $\text{TiFe}_{0.932}\text{Mn}_{0.094}$ (INCO #5), and $\text{TiFe}_{0.90}\text{Mn}_{0.096}$ (INCO #4). All of the manganese materials could be activated much more easily than FeTi with little or no heating, as illustrated in Table 3.1. This behavior is a major advantage for such alloys, in comparison to FeTi, because they can be activated in situ with no heating, thus leading to a reduction in capital costs for bed construction materials. The specific manganese alloy which seems to best meet the needs of the $\text{H}_2\text{-Cl}_2$ unit is $\text{TiFe}_{0.85}\text{Mn}_{0.15}$. It has the highest storage capacity for the Reference Design Conditions, as shown in Table 3.2; and a fully activated sample can be charged to its maximum hydrogen capacity in a matter of hours, as indicated in Table 3.3. Absorption and desorption isotherms for this alloy are shown in Figures 3.1 and 3.2. It should also be pointed out that if the Reference Design charging pressure were changed from 500 to 300 psia, the maximum storage capacity of FeTi would be reduced much more than that of $\text{TiFe}_{0.85}\text{Mn}_{0.15}$ because of differences in the stability and hysteresis of the two systems.

3.1.2 Subcontract Work

Two interim reports were received from the International Nickel Company. The first of these reports covered the economics and thermodynamics of the large-scale production of FeTi. The

lowest cost for the production of FeTi was determined to be aluminothermic reduction of ilmenite. The cost would be approximately \$1.60/lb; however, the resulting FeTi would be contaminated with 5-10 atom % Al, which seriously reduces its absorption capacity. The most cost-effective production method for good hydriding-quality FeTi was the direct melting of Ti and Fe, resulting in a cost/lb of \$2.25.

The second interim report dealt mainly with effects of contaminants on the hydrogen storage capacity of FeTi. It was found that both silicon and aluminum were particularly detrimental. The Ti-Fe-Mn system also requires further work because subtle variations in composition drastically affect ease of activation and isotherm shape for large-scale commercial melts.

3.2 Improved Hydrides

3.2.1 BNL Research

Several different manganese-substituted chrome-titanium alloys were prepared and investigated by p-c-T determinations. The alloys were all derivatives of the parent TiCr_2 system which has been defined and found to form a hydride of composition $\text{TiCr}_2\text{H}_{3.7}$. The stability of the hydrides formed from TiCr_2 are low, however, and their practical use is therefore limited. An alloy composition in the range $\text{Ti}_{1.20}\text{Cr}_{0.80}\text{Mn}$ to $\text{TiCr}_{0.80}\text{Mn}_{1.20}$ yields a hydride with an equilibrium storage capacity of ~2 wt % and has more attractive p-c-T properties. Other substituted AB_2 systems are also being investigated.

3.2.2 Subcontract Work

The Denver Research Institute initiated its program which is intended to use DRI predictive techniques to identify and characterize improved hydrides. The technique involves the correlation of the free energy of formation of a hydride with the interstitial hole size of the starting alloy. To date, work has primarily concentrated on the following: (1) experimental

studies of AB_2 Laves phases (distinct from those being investigated at BNL), including an analysis of the general structural and geometric characteristics of the C(14)- and C(15)-type phases; (2) initiation of hydriding studies on titanium-base, beta-solid-solution alloys; and (3) initiation of hydriding studies on ANi_5 compounds where Ni is partially replaced by two neighboring elements.

A contract will shortly be signed with Air Products and Chemicals of Allentown, Pennsylvania, in response to an RFP titled "Development of New or Improved Hydrogen Storage Systems for Automotive Hydrogen Fuel Storage." The main thrust of their work will deal with the development of hydride materials with ≥ 3 wt % storage capacity and capable of delivering such hydrogen at 1 atmosphere pressure with temperatures not to exceed 175°C . A second contract will also be signed in response to the above RFP with R. J. Teitel Associates to investigate the economics of a hybrid microcavity (high-pressure hydrogen in glass microspheres)/hydride storage system for automotive application.

Table 3.1

LOW-TEMPERATURE ACTIVATION EXPERIMENTS FOR
 TiFe_xMn_y ALLOYS

<u>Alloy Composition</u>	<u>Maximum Temperature (°C)</u>	<u>Elapsed Time At 500 PSIA (hr)</u>	<u>Final H/M</u>	<u>% Reaction</u>
TiFe	100	72	0	0
TiFe _{0.90} Mn _{0.10}	25	27	0.24	25
TiFe _{0.85} Mn _{0.15}	25	6	0.97	100
TiFe _{0.80} Mn _{0.20}	25	45	0.58	60
TiFe _{0.70} Mn _{0.18} (INCO #1)	25	25	0.91	91
TiFe _{0.70} Mn _{0.20}	50	3.5	0.94	94
TiFe _{0.932} Mn _{0.094} (INCO #5)	25	68	0.81	100
TiFe _{0.90} Mn _{0.096} (INCO #4)	25	19	0.86	96
TiFe _{0.77} Mn _{0.09}	25	27.5	0.98	100
TiFe _{0.73} Mn _{0.13}	25	19	0.98	100

Table 3.2

EQUILIBRIUM HYDROGEN STORAGE CAPACITIES FOR TiFe_xMn_y
 ALLOYS UNDER PROJECTED $\text{H}_2\text{-Cl}_2$ CELL OPERATING CONDITIONS *

<u>Alloy Composition</u>	<u>Effective Equilibrium H_2 Storage Capacity Wt %</u>
TiFe	1.55
$\text{TiFe}_{0.90}\text{Mn}_{0.096}$ (INCO #4)	1.50
$\text{TiFe}_{0.90}\text{Mn}_{0.10}$	1.65
$\text{TiFe}_{0.85}\text{Mn}_{0.15}$	1.80
$\text{TiFe}_{0.80}\text{Mn}_{0.20}$	1.60
$\text{TiFe}_{0.70}\text{Mn}_{0.30}$	0.90
$\text{TiFe}_{0.932}\text{Mn}_{0.094}$ (INCO #5)	1.35

*Charged to 500 psia, bed temperature = 40°C
 Discharged to 45 psia, bed temperature = 60°C

Table 3-3

CHARGING TESTS OF 5 HOURS DURATION FOR COMPLETELY ACTIVATED
 TiFe_xMn_y ALLOYS AT 500 PSIA AND 25°C

<u>Alloy Composition</u>	<u>Final H/M</u>	<u>% Reaction</u>
$\text{TiFe}_{0.93}\text{Mn}_{0.094}$ (INCO #5)	0.81	100
$\text{TiFe}_{0.85}\text{Mn}_{0.15}$	0.98	100

Figure 3.1 Pressure-composition desorption isotherms for the $\text{TiFe}_{0.85}\text{Mn}_{0.15}\text{-H}_x$ system at several temperatures.

Figure 3.2 Hysteresis in the $\text{TiFe}_{0.85}\text{Mn}_{0.15}\text{-H}_x$ system at 40°C

AREA 4: TECHNOLOGY UTILIZATION

4.1 Hydrogen-Halogen Program

Work at GE on the Hydrogen-Halogen Program was resumed in the last week of June, 1977. Since then, most of their work was focused on Task 1 which is the delivery of a 0.05-ft^2 cell with a chlorine/hydrochloric acid storage subsystem capable of operating at elevated pressures and temperatures. The unit will be completely instrumented. Work on this unit is essentially complete. Delays occurred because of late delivery of some items and mechanical problems with pumps. GE delivered a Phase I Summary Report covering activities from June 23, 1977 to October 24, 1977. This report also includes operating procedures and schematics for the deliverable items.

GE also carried out cell studies in small hydrogen/bromine cells. The purpose of these studies was to evaluate halogen electrode/current collector configurations. The results are encouraging. Low-cost electrode/current collector materials showed good performance and no degradation after operating times of 600 hours.

A heat-and mass-balance analysis for a 1-MW/10-MWh Hydrogen-Chlorine Energy Storage System was carried out at BNL. Typical results are shown in Figure 4.1. The system cools during charge, and the temperature increases during discharge. The degree of heating and cooling is a function of cell overvoltage (η), the method of reactant storage, and the degree of heat exchange between the electrolyte and reactant storage subsystems. Cell cooling during charge can affect the cell membrane resistance.

An investigation of Nafion membrane conductivity as a function of electrolyte concentration and temperature was completed. The data are plotted in Figure 4.2. These results, together with those from the heat-and mass-balance analysis, were used to

calculate a number of cell-voltage parameters, which are shown in Figure 4.3. An overall voltage efficiency of >75% can be expected from the system.

A method was devised to determine chlorine diffusivity and permeation rates through Nafion membranes. The rate of chlorine transport through the membrane was determined coulombically using the experimental setup shown in Figure 4.4. The data were used to calculate the expected self-discharge current due to chlorine migration through the membrane, and the results are shown in Figure 4.5. Indications are that the expected self-discharge rate will be low and will lead to coulombic inefficiencies of the order of 0.75-3%.

A setup for testing the GE system at BNL was designed, and all the necessary equipment was ordered.

Bechtel completed their preliminary techno-economic assessment of Hydrogen-Halogen Energy Storage Systems. Their principal conclusion was that the choice of chlorine or bromine and the method of chlorine storage had little effect on the cost of the system. Hydrogen storage as a compressed gas in mild-steel containers is competitive with hydride storage, if the installed and activated hydride costs more than \$2/lb. Storage as a compressed gas at 2000 psi in U.S. Steel's T-1 containers is competitive with hydride storage if the installed and activated hydride costs more than \$1.40/lb.

Figure 4.1 Temperature fluctuations in an electrochemically-regenerative hydrogen-chlorine energy storage system. Total capacity = 10 MWh; charge and discharge current = 10^6 Amp. Cell overvoltages are indicated on the figure.

Figure 4.2 Resistivity of a Nafion membrane as a function of HCl concentration and temperature. Broken lines are HCl resistivities.

Figure 4.3 Cell voltage parameters. It was assumed that all polarization losses were iR losses in the separator. The maximum temperature drop during charge was assumed.

Figure 4.4 Schematic of cell for determination of chlorine diffusivity and permeation rate through Nafion membranes. The inset is a schematic of the permeation current transients.

Figure 4.5 Calculated self-discharge currents due to chlorine migration through a 0.010-in. Nafion membrane as a function of temperature at three pressures.

AREA 5: SYSTEMS STUDIES

5.1 Natural-Gas Supplementation

The work done by Public Service Electric and Gas Company of New Jersey (PSE&G) in support of the Ad Hoc Committee's mission was reported in "Hydrogen Supplementation System Evaluation Study," now in press.

5.2 Hydrogen Injection in Natural Gas

The study by the Public Service Electric and Gas Company of New Jersey examines:

1. Gas blends
2. Conservation testing
3. Leakage testing

The contract has been extended to April 30, 1978 in order to complete the original Scope of Work. The program is within the original funding estimate. The delay was due to company-connected emergency work which had to be done by program personnel. The work completed to date is highly satisfactory. Volumes I and II of the final report, "Gas Blends Flow in the Distribution System," and "Conservation Tests of Blends," were received. Volume III, "Gas Blends Leakage Tests," is still due.

Discussions were begun for a follow-on effort. During a review meeting at PSE&G on January 12, it was agreed that the follow-on effort will emphasize the necessary ground work for hydrogen injection on an experimental basis when a commitment is made by DOE.

5.3 Characterization of U.S. Falling-Water Resources

The primary objective of this short-term study (3 months) is to assess the extent of falling-water resources associated with gas utility systems. The work is a joint effort by the Institute of Gas Technology (IGT) and the Pennsylvania Gas and Water Company. It involves an in-depth study of the

Pennsylvania area, a less detailed study of the Northeast U.S., and a cursory assessment of suitable falling-water resources in the U.S. Upon completion, the study will have examined the technical and economic feasibility for producing hydrogen from such falling-water resources. This effort is very dependent on information available from such resources as the Corps of Engineers, New York State ERDA, and DOE Geothermal Division. The specific question on the value of producing hydrogen from these resources is being addressed, as well as the question of the need for a demonstration effort.

The study will be completed in January 1978, and a final contract review meeting is scheduled for February 7, 1978.

5.4 International Energy Agency Agreement on Electrolytic Hydrogen Production

A meeting of the Working Committee on Electrolytic Procedures for Production of Hydrogen was held in Paris, France, on November 7, 1977; and the participants made commitments to the proposed cooperative effort. The meeting resulted in the preparation of two draft Annexes,

Annex IV - Advanced-Alkaline and Solid-Polymer Water Electrolysis;

Annex V - Solid-Oxide Water-Vapor Electrolysis, and the specification of manpower commitment. The primary accomplishments of the Working Committee are summarized below.

Accomplishments of the Working Committee as of November 7, 1977

1. Received formal input regarding the prospective contributions of the participating countries, including projected manpower.
2. Prepared draft Annexes IV and V.
3. Tentatively accepted the U.S. draft proposal on Intellectual Property, subject to further review.

4. Identified the U.S. and Germany, respectively, as the prospective Operating Agents for Annexes IV and V. It is anticipated that the responsibilities will most likely be carried out by the respective governments through the Brookhaven National Laboratory in the U.S., and the Julich Research Center in Germany.
5. Representatives agreed to review draft Annexes IV and V, particularly the section on Intellectual Property, and have comments to IEA, Paris, by January 15, 1978.
6. It was agreed that the proposed Operating Agents will review and polish the draft Annexes and have responsibility for the preparation of program plans.

AREA 6: PROJECT MANAGEMENT

6.1 Project Planning and Liaison

The major planning activity completed during the period was the FY 78 Program Operating Plan for the BNL-Managed Hydrogen Project. This 100-page document provides the details on program goals and objectives, budget allocations, schedule milestones, and includes data/fact sheets on each contract or project area. The plan will serve as the working document for the program and the basis for generating long-range or multi-year plans.

Some administrative support was provided to JPL for conducting the Annual Hydrogen Contracts Review Meeting held at Hunt Valley, Maryland, in mid-November 1977. All of the contractors managed by BNL and representatives of the BNL in-house program, presented progress reports at the meeting. The proceedings are being assembled by DOE/NASA-JPL and should be published shortly. Initial planning is being done by BNL for conducting next year's meeting.

The following activities relate to planning the direction of the technical work and allocation of funds: Two Request for Proposals (RFP) were processed during the period. The first RFP was concerned with "Development of New or Improved Hydrogen Storage Systems for Automotive Hydrogen Fuel Storage." Over 100 copies of the RFP were distributed and nine proposal responses were received. After the formal evaluation procedure, which included technical experts from other laboratories and outside organizations, two contracts were awarded; one to Air Products and Chemicals, and the other to R. J. Teitel Associates.

The second RFP, which was issued in November, was for work in "Underground Hydrogen Storage." Over 150 copies of the RFP were distributed, and there has been considerable interest in

the procurement. The proposal evaluation process will be conducted during February and March 1978 with a contract award expected by fourth quarter, FY 1978.

A total of nine unsolicited proposals/preproposals were evaluated during the period. The evaluations consisted of obtaining and compiling technical reviews from cognizant individuals in the specific research area and relating the proposed work to the program goals. Of this total, six proposals were rejected, two are pending and one proposal from Clarkson College was recommended for funding. These figures do not include follow-on proposals for work in progress.

6.2 Contract Management and Technical Monitoring

The BNL Conservation Program Management Group (CPMG) managed and provided technical monitoring for 17 contracts amounting to about \$1.8 million connected with the Hydrogen Program in FY 77. The largest of these contracts was with General Electric (\$900,000) for work on large-scale water electrolysis technology development. The primary activity, in addition to the administrative tasks associated with the placement of the contracts, was the monitoring of contractors' technical progress by BNL personnel. This function included discussions with principal investigators, site visits, review of monthly reports and supporting the hardware demonstrations. An example of this latter activity was the participation in the inaugural of the Billings Hydrogen Homestead Project which incorporated a DOE/BNL contracted metal-hydride, hydrogen storage reservoir. A listing of the contractors, the approximate contract value and the R&D area is provided in Table 6.1. A chart of the program budgetary distribution is shown in Figure 6.1, to indicate areas of program emphasis. Another chart, Figure 6.2, shows the major milestones scheduled in the program.

Table 6.1
CONTRACT ACTIVITIES FY 1977-78

<u>Contractor</u>	<u>Work Area</u>	<u>Approx. Contract Value</u>
General Electric (2)	SPE Water Electrolysis	\$900K
Yeshiva University	Electrolysis Research	15K
Teledyne Energy Sys.	Alkaline Electrolysis	125K
Univ. of Virginia	Electrolysis Materials	15K
Teledyne Motors	Automotive Storage	75K
Foster Wheeler	A/E Hydrogen Storage	50K
Billings	Hydride Reservoir	125K
Stevens Inst. of Technology	Hydrogen Storage/Conversion	45K
Denver Research Institute	Hydride Research	100K
INCO	Hydride Metallurgy	40K
Air Products	Improved Hydrides (Auto- motive)	125K
Public Service Electric & Gas (2)	Natural-Gas Supplementation	60K
R. J. Teitel Assoc.	Hydrogen Storage	60K
Bechtel	A/E Hydrogen Energy Storage	50K
EDA	Halogen Storage	40K

Figure 6.1 Budgetary distribution of BNL Hydrogen Project

TECHNOLOGY AREA	SCHEDULE					MILESTONES
	FY78	FY79	FY80	FY81	FY82	
ELECTROLYTIC HYDROGEN PRODUCTION			*	-	▲	DELIVER 5-MW PRODUCTION PROTOTYPE OF SPE WATER ELECTROLYSIS SYSTEM *DECISION ON SCALE-UP 2.5 TO 10 FT ² CELLS
HYDROGEN STORAGE UTILITIES		*	-	-	▲	DELIVER 20-MWh METAL HYDRIDE STORAGE SYSTEM (VESSEL > 3-FT DIAMETER, UP TO 40-FT LONG) FOR USE WITH 2-MW HYDROGEN-HALOGEN CELL IN BEST *DECISION ON SCALE-UP
AUTOMOTIVE				▲		COMPLETE TEST AND EVALUATION OF STORAGE SYSTEM FOR AUTOMOTIVE APPLICATION
MATERIALS			▲			IDENTIFY MOST PROMISING MATERIAL/METHOD FOR HYDROGEN STORAGE FOR AUTOMOTIVE APPLICATION
TECHNOLOGY UTILIZATION						
HYDROGEN-HALOGEN ENERGY STORAGE SYSTEM			*	-	▲	DELIVER 2-MW PRODUCTION PROTOTYPE MODULE FOR TESTING IN BEST *DECISION ON SCALE-UP
NATURAL-GAS SUPPLEMENTATION			▲	-	*	COMPLETE CURRENT TECHNOLOGY DEVELOPMENT *DECISION TO RE-EVALUATE CONCEPT
HYDROGEN AS A PROPANE SUBSTITUTE			▲			COMPLETE EVALUATION OF HYDRIDE VESSEL IN HYDROGEN HOMESTEAD
						DETAILS ON EVENTS AND ACTIVITIES SUPPORTING THESE MILESTONES CAN BE FOUND IN THE 5-YEAR PROGRAM.

Figure 6.2 Major technology milestones and deliverables

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