Present Status and the Perspective of New Hydrogen Energy Project

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Abstract

A research and development project, named as 'New Hydrogen Energy', has started in Japan in November, 1993, to confirm the excess heat generation during electrolysis with Pd-LiODsystem as the first priority objective. The Project has been supported by the Ministry of International Trade and Industry(MITI) and major industries in Japan, and new laboratory was established in Sapporo.

Present status and the perspectives of the project will be reported together with main technical results. Two types of electrolysis cells, namely, open type cell system from IMRA-Europe, Inc. and the fuel cell type cell systems from IMRA-Japan, Inc., were installed in NHE Sapporo laboratory and the experiments have started from February 1994 to demonstrate the NHE-phenomena.

Correlationships among maximum attainable D/Pd ratio, deuterium absorption and desorption and the characteristics of various palladium sources and treatments have been studied and analysed based on the resultsof the material observations and instrumentational analysis such as O/M, SEM, XRD, AES, SIMS and EPMA.

The laboratory will be reinforced by introducing of mass flow calorimetry systems, and reaction products detection systems towards an interim review scheduled in late 1995.

This R&D program has been conducted under the consignment of New Energy and Industrial Technology Development Organization(NEDO).

1. Introduction

Fleischmann and Pons revealed the excess heat generation phenomena by electrolysis of heavy water using a palladium electrode in 1989. Since then, there have been many researchers who observed this heat generation and the amount of heat sometimes cannot be explained by chemical reaction, and there are some theoretical models which may possibly explain the phenomena. Since the heat generation is essentially based on abundant deuterium, there arose an expectation to develop this phenomena as a non-oil energy source.

A R&D project has started in Japan on November 1993, in which the excess heat generation is regarded as "New Hydrogen Energy". The aim of this R&D project is to clarify the potentiality and possibility to use a future energy source and to control heat generation quantitatively by its demonstration and understanding of the reaction mechanism.

This "NHE-Project" had been first announced and introduced at ICCF-4 held in Maui by one of the author, Matsui.

2. Organization Structure

Ministry of International Trade and Industry (MITI) has decided to subsidize New Energy and Industrial Technology Development Organization (NEDO). NEDO have contracted with the Institute of Applied Energy (IAE) to conduct this research and development project of four years as period from late 1993. Figure 1 shows the R&D execution structure.

Three committees were established in NEDO with noticeable experts, some from the academic world and the other from industries; the first committee is responsible to determine a basic plan and action programs, the second to evaluate R&D results and the third to plan and manage the action programs.

The NHE R&D project consist of a national project and multi-client industry supported project. The national project conducts mainly to examine excess heat generation for confirmation and demonstration. The latter project conducts basic researches. The framework of the NHE R&D project is illustrated in figure 2. These two projects keep close relationships and are mutually supported and cooperated. Details of latter project will be described later by Prof. Okamoto of Tokyo Institute of Technology.

The guidelines of the NHE-R&D project are as follows,

- (1) The NHE research and development program is to be recognized as one of national basic research plans of industrial science and technology in long term energy issues, and to be executed in focal point manner with researchers of private sector who have significant experiences in related fields.
- (2) The researches by universities with financial support from private sector will concentrate more basic researches, such as clarification of reaction mechanisms in order to support the national project.
- (3) Furthermore, reinforcement of international networking with foreign research institutes and exchange of basic research information through international symposiums and exchange program to support identification of possibility for future energy source.
- 3. Research and Development Programs
 - (1) Demonstration of excess heat generation
 - 1) Measurement of excess heat during electrolysis

Based on several prior arts of experiments, measurements of excess heat are to be demonstrated to confirm the NHE phenomena, and the excess heat generation is to be quantified together with its reproducibility, controllability, amplification and durability.

- 2) Reaction products accompanied with excess heat The specifications of kinds and amount of reaction products is to be instrumented with insitu observation, and correlation with heat generated is to be surveyed to support reaction modeling.
- 3) Conjunction of electrolysis and vacuum method

To take advantages of both electrolysis(wet) and vacuum method(dry), electrode material and reaction products are to be analyzed during excess heat generation at high deuterium loading to find out the control factor of the reaction.

- (2) Material analysis and development
 - 1) Material analysis

At first, it is essential to find out basic features of electrode material required to generate excess heat. Two types of materials; one with excess heat generation and the other without, and another with high loading and the other with low, are to be observed and analyzed to identify the differences.

2) Material development

Controlled factors of material characteristics are to be found out for the high loading and the heat generation, and guidelines for material development are to be formulated.

(3) Information exchange

Information and data exchange are to be encouraged to formulate developmental guidelines in order to conduct effectively research and development programs of which master schedule and research items are shown in figure 3.

4. Progress of NHE-Project.

The milestones of this national project are shown as follows,

FY1993

- Nov. 01, 1993: Establish R&D Center for NHE(Tokyo)
- Dec. 01, 1993: 1st NHE Steering Committee(Start of the Project)
- Dec. 01, 1993: Establish NHE Laboratory(Sapporo) in NHE Center

Dec. 06, 1993: ICCF-4(Maui, Hawaii), 1st announcement of the project

- Feb. mid., 1994: Start of Excess Heat Experiments with Two Electrolysis Systems
- Feb. 23, 1994: 1st NHE National Symposium in Sapporo

Mar. end, 1994: Annual Report(FY1993)

FY1994

May. 17, 1994: 2nd NHE Steering Committee

Sep. 05, 1994: 1st International NHE Workshop(Lake Kawaguchi)

Oct. mid., 1994: Start Development of Mass Flow Calorimetry Components and System

- Dec. 13, 1994: 3rd NHE Steering Committee
- Mar. end, 1995: Annual Report(FY1994)

FY1995

Apr. 10, 1995: ICCF-5, 3 Presentations from NHE Project.

Jun. 02, 1995: 4th NHE Steering Committee

- Oct. mid, 1995: 2nd International NHE Workshop(Trino)
- Dec. mid, 1995: Interim Review of NHE Project

Mar. end, 1996: Annual Report(FY1995)

5. Major Activities and the Results

According to the basic plan which is described above, R&D activities have been conducted in NHE-Center. Major experimental activities and the results which have been performed hitherto, substantially during FY 1994, in NHE-Laboratory are summerized. Figure 4 shows the milestone chart of NHE-R&D items in FY1993 and FY1994.

5-1 Demonstration of excess heat generation

(1) Experiments on fuel cell type electrolysis system

Measurement of the D/Pd ratio and excess heat of Pd from various sources have been carried out using fuel cell type electrolysis systems. Various Pd include 3-nine and 4-nine purity, meltedin atmosphere and vacuum, as cold worked and annealed, polycrystal and single crystallized, and Pd-5%Rh and -10%Rh alloys were examined to seize the suitable material characteristics.

Major results are followings

a) Measurement of Excess heat of $9 \sim 15\%$ have been reproduced in 2 cases with vacuum

annealed pure Pd, however reproducibility issue still remained.

- b) D/Pd ratio of 0.89 and H/Pd ratio of 0.93 have been attained with vacuum annealed pure Pd and single crystallized Pd.
- c) Low D/Pd ratio of $0.79 \sim 0.82$ have been measured in as cold worked Pd having surface defects such as micro-crack.
- d) High D/Pd ratio of $0.9 \sim 0.94$ have been attained with Pd-10at%Rh alloys, however excess heat has not observed.
- (2) Experiments on open type electrolysis system

The open type cell systems, namely ICARUS-1 system which was developed by Fleischmann and Pons originally, have been introduced in NHE-Laboratory at the end of Jan. 1994, and started electrolysis with pure Pd and Pd-10%Ag.

ICARUS-1system is capable to measure the phenomena with relatively low current(~ 200 mA) electrolysis and then negligible region of evaporation effect. Necessity of the improvement on cell components and instrumentation had been recognized to reduce the measurement noise and improve precise calorimetry.

Version up to ICARUS-2 system from ICARUS-1 system to measure near boiling region phenomena and to eliminate ambiguity by accuracy and precision, has been completed in Feb. 1995.

(3)Development of mass flow calorimetry system(FCS)

A mass flow calorimrtry to confirm the excess heat generation measurement in above 2 prior systems has been developed from middle 1994. Figure 5 shows the conceptual system flow of NHE- FCS. Although various electrolysis cell types are considerable, at the first step, fuel cell type electrolysis cells which is illustrated in figure 6, has been developed.

As a result of careful modification of the FCS components, fluctuation of flow rate of < 0.5% and heat recovery rate of >98% has been attained.

On the other hand, mass flow calorimetry systems with a cell having recombiner which had been developed SRI was also introduced and installed in NHE Laboratory in Mar. 1995.

5-2) Material analysis and material developments

Microstructural observations and analysis on various source Pd have been performed to compare and evaluate the material characteristics. Surface and inner microstructure using O/M and SEM, surface impurity analysis using AES, SIMS, and EPMA, and crystal structure analysis using XRD have been carried out for Pd electrodes. And also, chemical composition analysis of electrolytes using ICP-AES, ICP-MS and isotopic composition analysis using NMR have been conducted.

As a results of the analysis and observation, understandings of correlation among material characteristics such as exisistence of surface defects and impurities, effect of annealing and annealing conditions, D/Pd loading factors, have been deepened.

From the overall evaluation of excess heat measurement, material analysis, and loading rate, standardized Pd material specifications and treating processes have been proposed as follows;

- 1) Source Pd: Vacuum melting pure Pd (Typically >3 nine) and cold working uniformly
- 2) Processing: Machining surface layer($\sim 0.2 \mathrm{mm}$) to remove surface defects and impurities
- 3) Annealing: Annealing in high vacuum($<10^{-5}$ torr) environment at 850° after chemically etched

6. Perspectives

This FY1995 is the 3rd fiscal year from the start of NHE-Project, and about 15 monthes has been passedfrom the beginning of actual experimental works. The project is now at the most important and interesting stage. There are many issues to pursue and to clarify the correlation between the heat generation and material characteristics, and excess heat and nuclear products. From this view point, the domestic and international collaboration should be emphasized to conduct effective research and development of this new scientific field because isolated research effort has its limit in human and ideas, and financial resources.

Major activities to be conducted in FY1995 are as follows;

- (1) Improvement of reproducibility for excess heat measurement in fuel cell type electrolysis systems with high loading material
- (2) Reproduce Fleischmann & Pons Phenomena at nearly boiling point of electrolyte using ICARUS-2 systems.
- (3) Confirmation of heat generation using both newly developed and SRI type mass flow calorimetry systems
- (4) Material analysis and development to attain high loading and reproducibility, and development of in-situ X-ray diffraction measurement to investigate the crystallographic change during electrolysis.
- (5) Development of reaction products detection systems and the measurement of γ -ray, tritium, and He etc. at high deuterium loading.

The NHE research activities will be reinforced by introducing of mass flow calorimetry and reaction products detection systems toward an interim review scheduled in late 1995.

7. Acknowledgement

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We would also like to thank all members of NHE-Centre and Laboratories for their collaboration and sincere research efforts.



Figure 1 Organizational Structure of NHE Project



Figure 2. Framework of NHE Research and Development

	FY 1993	FY 1994	FY 1995	FY 1996
1. Demonstration of Excess Heat				
o Reproducibility of Excess fieat				
o Verification of Reaction Products				
o Identification of Control Factors				
2. Material Science				
o Characterization of Materials				
o Development of New Materials				
3. Database and Feasibility Study				

Figure 3 Master Schedule and R&D Items of NHE Project

Fiscal Year			FY 1993			FY 1994												1
R&D Items	Month	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	1
 Demonstration Model Experiments for Excess Heat Generation a)Fuel Cell Type Electrolysis Cell												-						
											• •	,			•	· · ·		
2.Material Analysis an a)Observation and Ana Electrodes and Cell b)Basic Research on I c)Benchmark Test of I	nd Development alysis of Pd I Components Pd Electrode Pd Source Metal																	
3.Detection of Reactio	on Products					0				********			•••••					-
4. Investigation of New	w Exp. Systems							;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;		**********			_,_,_,				, _ ,_,_	

Figure 4. Milestone Chart of NHE R&D Items in FY 1993 and FY 1994



Figure 5. The Concept of Mass Flow Calorimetry System



Figure 6. Schematic Figure of Fuel Cell Type NHE-FCS Cell