Brief Summary Report of MHE Project Phenomenology and Controllability of New Exothermic Reaction...

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Brief Summary Report of MHE Project Japan for 2015 October - 2017 October

Phenomenology and Controllability of New Exothermic Reaction between Metal and Hydrogen

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Project Aim: to verify the existence of new exothermic reaction between nano-metals and hydrogen which will be applicable for future new clean energy source, and to study the controllability of generated thermal energy.
In the following, brief summary of implementation and results by MHE-group Japan is described in designated R&D issues for two years project period of 2015 October to 2017 October.

1) New MHE (metal hydrogen energy) Calorimetry Facility

In the first year (2015-2016) program, a new highly accurate oil mass-flow calorimetry system was installed at Tohoku University. The system was designed by improving performances of the already existing MHE calorimetry system (500 cc reaction chamber and many operation components) at Kobe University. We fabricated components and assembled at ELPH (Electron Photon Science Research Center) of Tohoku University. In July 2016, main body of system was constructed and started to make performance test in open room, and after two months for primary tests, the system settlement was finished in a new temperature-controlled (within ±0.1℃) room. Evaluated accuracy of the calorimetry system is satisfactory, namely less than ±1.5％error in thermal-power measurement, less than ±0.1℃ error in temperature detection by thermos-couples and RTDs, and less than ±2％ error in thermal flux measurement. The new system has started to be used for the collaboration experiments of 6-

parties-joint team since August 2016.

1) Implementation of Heat Generation Tests

Two MHE facilities at Kobe University and Tohoku University and a DSC (differential scanning calorimetry) apparatus at Kyushu University have been used for excess-heat generation tests with various multi-metal nano-composite samples. Members from 6 participating institutions have joined in planned 16 times test experiments in two years (2016-2017). We have accumulated data for heat generation and related physical quantities at room-temperature and elevated- temperature conditions, in collaboration. Cross-checking-style data analyses were made in each party and compared results for consistency. Used nano-metal composite samples were PS（Pd-SiO_2）-type ones and CNS(Cu-Ni-SiO_2)-type ones, fabricated by wet-methods, as well as PNZ（Pd-Ni-Zr）-type ones and CNZ（Cu-Ni-Zr）-type ones, fabricated by melt-spinning and oxidation method.

Results for room-temperature condition: Data of significant hydrogen absorption and heat were obtained for Pd-containing samples of PNZ-type and PS-type. In the starting (virgin sample) runs, 50～100kJ heat per 1-mol H(D) with 3.5 (PNZ) to over 1.0 (PS) H(D)/Metal hydrogen absorption were observed. Post H(D)-charged runs after baking process gave 5～30kJ per 1-mol-H(D) heat with smaller H(D)/Metal absorption values. The level of observed heat generation data at room temperature condition was discussed to be explainable by chemistry-origin reactions. In the cases of Cu-containing samples as CNZ-type and CNS-type, there were no observable H(D)-absorption and heat generation phenomenon, at room-temperature condition.

Results for elevated-temperature condition: Significant level excess-heat evolution data were obtained for PNZ-type, CNZ-type CNS-type samples at 200-400℃ of RC (reaction chamber) temperature, while no excess heat power data were obtained for single nano-metal samples as PS-type and NZ-type. By using binary-nano-metal/ceramics-supported samples as melt-span PNZ-type and CNZ-type and wet-fabricated CNS-type, we observed excess heat data of maximum 26,000MJ per mol-H(D)-transferred or 85 MJ per mol-D of total absorption in sample, which cleared much over the aimed target value of 2MJ per mol-H(D) required by NEDO. Excess heat generation with PNZ-type samples has been also confirmed by DSC experiments, at Kyushu University, using very small (0.04 to 0.1 g) samples at 200 to 500℃ condition. Optimum conditions for running temperature (around 400 degree C) and Pd/Ni ratio (around 1/7-1/10) were obtained by the DSC experiments at Kyushu University to get highest heat flow (power). We also observed that the excess power generation was sustainable with power level of 10-24 W for more than one month period, using PNZ6 (Pd_1Ni_{10}/ZrO_2) sample of 120g at around 300℃.

Reproducibility at different laboratories: Providing two divided sample powders of PNZ-type from same-batch fabricated powder, independent parallel test runs were carried out at Kobe University and Tohoku University. Results of excess heat generation data from both laboratories were very reproducible for room-temperature and elevated-temperature conditions. Thus, the existence and reproducibility of new exothermic phenomenon by interaction of nano-metal composite samples and H(D)-gas have been confirmed.

2) Analyses and Evaluation of Generated Heat Quantity

Experimental data obtained by the collaboration runs were evaluated by parallel analyses of participated members. As for consequence, we have confirmed that multi-metal composite samples by melt-span-oxidation method and wet CNS-type did generate preferably significant excess heat at elevated-temperature conditions. Excess-power reduction was made by comparing foreground runs with blank (using ZrO2 or silica) calorimetry calibration runs. Integrated excess heat data at elevated-temperature condition exceeded several MJ per ca. 100g sample, which is impossibly difficult to explain heat generation level of a few 100kJ/mol-metal (at most) by known chemical reactions. In the case of room-temperature condition for PS-type (mono-metal nano-particle) samples, experiments showed ca. 50kJ/mol-H(D) heat generation, which is considered to be within the level of chemical origin reactions. No convincing excess power data have been seen at elevated temperatures (over 100℃), for PS-type mono-Pd nano-particle samples.

Form these obtained knowledge, we will be able to conclude that Ni-based multi-metal nano-composite samples are of very required necessary condition for generating sustainable high intensity heat-power generation at elevated temperatures more than 200℃. It is encouraging towards industrial application of thermal and electric power devices.

3) Sample Material Analyses before and after Runs

Various techniques such as XRD, SOR-XRD, SOR-XAFS, TEM, STEM/EDS, ERDA, ICP-MS, and others have been used for material analyses of experimental samples before and after use.

By XRD analyses, co-existence of ZrO_2 and Zr_2Ni was identified for PNZ-type samples. It was observed that the metal phase was fundamentally the same before and after H(D)-charging runs, but the existence of hydride was seen by the lower-angle-side sift of XRD $NiZr_2$ peaks.

By STEM/EDS analyses, we could observe that nano-structure of sample did not change much before and after use. Ni and Pd atoms were seen in same localized zones in homogeneous ZrO_2 supporting zone. Sometimes, $NiZr_2$ zones were surrounded by ZrO_2 layers and particles of Pd and Ni were existing in inter-crystalline layer zones, as observed by TEM. For CNZ-type samples, crystal phases of ZrO_2 and NiO were seen before H(D)-charge runs, and after the run Ni and ZrO_2 were seen. $NiZr_2$ components were not found in CNZ-type samples. Nano-structured CuO and NiO portions were seen as randomly distributed in ZrO2 supporter zone before the run, and alloy-like Cu-Ni nano-particles were seen after the run. For PNZ6 sample which provided the highest excess power level of 24 W per ca. 100g sample, Ni and Pd were not oxidized before and after runs as observed by the

SOR-XAFS analysis.

For PS-type samples, PdO crystal phase was seen before run, and reduced Pd-metal nano-particles were seen in supporter mesoporous silica after run.

5) Oversea Trends of Study towards Industrial Applications

Information survey in over sea USA and European countries has been done. Trends of research studies was picked up by participating international meetings as ICCF20 (the 20th International Conference of Condensed Matter Nuclear Science) Sendai 2016 and IWAHLM12 (the 12th International Workshop for Anomalies in Hydrogen Loaded Metals) Italy 2017. Survey of Russian activity was done by Nissan Motors Co.. Activity in this field is now growing up worldwide. H(D)-gas loading method is now the major experimental approach in the world. However research grades in over sea groups at present do not look so high as providing highly accurate excess heat data with good reproducibility. Our progress in this MHE project seems going much ahead in the world.

Foreseeing the industrial applicability, road-map study was made by our joint-team to draw the realization of industrial devices in 5-10 years later. Direct usage devices of excess heat for warming EV cars and family rooms as well as electric power devices with thermos-electric conversion systems are looked for realization in 10-20 years later.

6) Program R&D Meetings

At the managing office Technova Inc. of this project, 9 R&D discussion/managing meetings were held in 2016-2017. In every meeting, the joint-team members from 6 institutions, MHE-project members and external science advisors have participated for reporting, discussing on latest-obtained results, next experimental plans and tactics towards national project. For starting national project class R&D activity, the joint team concept with 5 sub-groups of increment of excess heat level, material development, mechanism study, substantial industrial application study and managing/strategy.

Note: The full final report (169 pages) to NEDO is written in Japanese.

Publications

[1] Akira Kitamura, Akito Takahashi , Koh Takahashi, Reiko Seto, Yuki Matsuda, Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi, Masanori Nakamura, Masanobu Uchimura, Hidekazu Takahashi, Tatsumi Hioki, Tomoyoshi Motohiro, Yuichi Furuyama, Masahiro Kishida: Collaborative Examination on Anomalous Heat Effect Using Nickel-Based Binary Nanocomposites Supported by Zirconia, Proc. ICCF20, published in JCMNS Vol.24, 2017

[2] Y. Iwamura, T. Itoh, J. Kasagi, A. Kitamura, A. Takahashi and K. Takahashi: Replication Experiments at Tohoku University on Anomalous Heat Generation Using Nickel-Based Binary Nanocomposites and Hydrogen Isotope Gas, Proc. ICCF20, published in JCMNS Vol.24,

2017

[3] Akira Kitamura , Akito Takahashi , Koh Takahashi , Reiko Seto , Yuki Matsuda, Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi ,Masanori Nakamura , Masanobu Uchimura, Hidekazu Takahashi , Tatsumi Hioki , Tomoyoshi Motohiro, Yuichi Furuyama , Masahiro Kishida; Collaborative Examination on Anomalous Heat Effect Using Nickel-Based Binary Nanocomposites Supported by Zirconia, Proc. JCF17, 2017, JCFRS

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[1] **Anomalous Heat Generation and Nuclear Transmutation Experiments at Condensed Matter Nuclear Reaction Division of Tohoku University**

Yasuhiro Iwamura

The Satellite Symposium of 20th International Conference on Condensed Matter Nuclear Science, September 26, 2016, Xiamen, China

[2] **Collaborative Examination on Anomalous Heat Effect Using Nickel-Based Binary Nanocomposites Supported by Zirconia**

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The 20th International Conference on Condensed Matter Nuclear Science, October 4, 2016, Sendai, Japan

[3] **Replication Experiments at Tohoku University on Anomalous Heat Generation Using Nickel-Based Binary Nanocomposites and Hydrogen Isotope Gas**

Y. Iwamura, T. Itoh, J. Kasagi, A. Kitamura, A. Takahashi and K. Takahashi

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[4] **Heat evolution from silica-supported nano-composite samples under exposure to hydrogen isotope gas**

Akira Kitamura, Akito Takahashi1, Koh Takahashi1, Reiko Seto, Takeshi Hatano,Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi, Masanori Nakamura, Masanobu Uchimura, Hidekazu Takahashi, Shunsuke Sumitomo, Tatsumi Hioki, Tomoyoshi Motohiro, Yuichi Furuyama, Masahiro Kishida, Hideki Matsune

The 17th Meeting of Japan CF-Research Society, March 19, 2017, Tokyo, Japan

[5] **Anomalous Heat Generation Experiments Using Metal Nanocomposites and Hydrogen Isotope Gas**

Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi,Akira Kitamura, Akito Takahashi, Koh Takahashi2, Reiko Seto, Takeshi Hatano, Tatsumi Hioki, Tomoyoshi Motohiro,Masanori Nakamura, Masanobu Uchimura, Hidekazu

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Tatsumi Hioki, Akihisa Ichiki and Tomoyoshi Motohiro
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[8] **Effect of Supporter Material on Heat Evolution from Ni-based Nano-Composite Samples under Exposure to Hydrogen Isotope Gas**
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[11] **Reproducibility on Anomalous Heat Generation by Metal Nanocomposites and Hydrogen Isotope Gas**
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The 18th Meeting of Japan CF-Research Society, November 24, 2017, Sendai, Japan

[12] **Measurement of Anomalous Heat Generation in Hydrogen Flow by Differential Scanning Calorimetry**

Masahiro Kishida, Hideki Matsune, Akira Kitamura, Akito Takahashi, Koh Takahashi, Reiko Seto, Takeshi Hatano, Tatsumi Hioki, Tomoyoshi Motohiro, Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi, Masanori Nakamura, Masanobu Uchimura

The 18th Meeting of Japan CF-Research Society, November 24, 2017, Sendai, Japan

[13] **In-situ XRD and XAFS Analyses for Metal Nanocomposites Used in Anomalous Heat Generation Experiments**

Tatsumi Hioki, Kazuya Nakazawa, Akihisa Ichiki, Tomoyoshi Motohiro, Akira Kitamura,Akito Takahashi, Koh Takahashi, Reiko Seto, Takeshi Hatano, Yasuhiro Iwamura, Takehiko Itoh, Jirohta Kasagi, Masanori Nakamura, Masanobu Uchimura, Hidekazu Takahashi, Shunsuke Sumitomo, Yuichi Furuyama, Masahiro Kishida, Hideki Matsune

The 18th Meeting of Japan CF-Research Society, November 24, 2017, Sendai, Japan