The 20th Meeting of Japan CF-Research Society

JCF20 ABSTRACTS

December 13-14, 2019

Reference Hakata Eki-Higashi Rental Room in Fukuoka

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Program of JCF20 Meeting
Japan CF-Research Society

Date: December 13-14, 2019
Place: Reference Hakata Eki-Higashi Rental Room, Fukuoka, Japan
Paper presentation: Oral presentation 20 min. + Discussion 5 min.
Language: English or Japanese
Book of Abstract: Only available at JCF home page (http://jcfrs.org/)

December 13 (Fri), 2019

12:00-13:00 Registration

13:00-13:10 Opening Address M. Kishida (Kyushu U.)

Session-1 Chair: S. Narita (Iwate U.)

13:10-13:35 JCF20-1 S. Murakami et al. (Clean Planet Inc.) Excess Energy Generation Experiments using a Nano-sized Multilayer Metal Composite and Hydrogen Gas
13:35-14:00 JCF20-2 M. Saito et al. (Clean Planet Inc.) Elemental Analysis for Elucidation of the Anomalous Heat Generation Phenomena
14:00-14:25 JCF20-3 T. Itoh et al. (Tohoku U.) Low Energy Photon Measurements in Anomalous Heat Generation Experiments using Nano-sized Metal Composite and Hydrogen Gas
14:25-14:35 Break

Session-2 Chair: K. Tsuchiya (NIT, Tokyo College)

14:35-15:00 JCF20-4 A. Takahashi et al. (Technova Inc.) Enhancement of Excess Thermal Power in Interaction of Nano-Metal and H(D)-Gas
15:00-15:25 JCF20-5 M. Uchimura et al. (Nissan Motor Corp.) Irregular Oscillation Pattern during Heat Generation from Ni-Zr Based Alloys and Hydrogen Gas
15:25-15:50 JCF20-6 K. Ooyama (Ooyama Power Inc.) Start-up of Metal Crystal Confinement Fusion Reactor
15:50-16:00 Break
Session-3  Chair: M. Kishida (Kyushu U.)

16:00-16:25  JCF20-7  H. Numata
Microstructure of Pd Rod Electrode during Repeated Cathodic and Anodic Electrolysis in Glycerin-phosphoric acid: Hydrogen Electrode :  H/Pd > β min with Vacancy

16:25-16:50  JCF20-8  K. Tsuchiya (NIT, Tokyo College)
A Theoretical Study on the Possible Change of the Phonon Dispersion Relation due to the Nuclear Reaction in Two-dimensional Lattice II

16:50-17:15  JCF20-9  H. Miura
Computer Simulation of Transition Energy of H atoms in Cu, Ag and Au Metal Lattices

17:15-17:45  JCF Annual Meeting

18:30-20:30  Reception

December 14 (Sat), 2019

Session-4  Chair: K. Tanabe (Kyoto U.)

9:30 - 9:55  JCF20-10  Y. Satoh et al. (Kyushu U.)
Verification of Anomalous Heat Detected by Differential Scanning Calorimetry from Palladium-Nickel-Zirconium Alloy in Hydrogen Flow (2)

9:55 -10:20  JCF20-11  M. Takeya et al. (Tohoku U.)
Calibration of Sendai mass-flow-calorimeter and heat measurements of recycled samples

10:20 -10:45  JCF20-12  M. Endo et al. (Iwate U.)
Deuterium Desorption Experiment Using Surface Coated Pd Foil with Fine-structured Interface

10:45-11:00 Break

Session-5  Chair: Y. Iwamura (Tohoku U.)

11:00-11:25  JCF20-13  T. Kobayashi et al. (Waseda U.)
Development of Reaction System with Small Chamber for Fundamental Experiments Measuring Anomalous Heat Effect

11:25-11:50  JCF20-14  T. Uchikoshi et al. (Kyoto U.)
Laser Irradiation to D-Loaded Pd

11:50-12:15  JCF20-15  I. Imoto et al. (Kyushu U.)
Anomalous Heat in New Binary Metal System under Hydrogen Stream (2)

Adjourn
Excess Energy Generation Experiments using a Nano-sized Multilayer Metal Composite and Hydrogen Gas

Shoichi Murakami¹, Takehiko Itoh¹,², Yasuhiro Iwamura², Mari Saito¹ and Jirohta Kasagi²

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Collaboration research between our company and Tohoku University have been carried out to develop a new energy source using anomalous heat generated by Condensed Matter Nuclear Reactions.

As for anomalous heat, it was reported that heat generations far beyond 10eV/H or D were observed during NEDO project [1]. The anomalous heat was obtained by heating nano-sized particles, such as CuNiZr-Oₓ or PdNiZrO-Oₓ, up to 200~300°C with D₂ or H₂ gas. On the other hand, transmutation reaction of Cs into Pr induced by D₂ gas permeation through nano-sized Pd and CaO multilayer composite was reported [2].

Based on these papers, we developed a new type of excess heat experiments using a nano-sized metal multilayer composite and hydrogen gas. Nano-size multilayer thin film samples consist of Ni and Cu were placed in the chamber. After evacuation of the chamber, samples were baked and cooled down. Next, H₂ gas was introduced into the chamber up to about 230 Pa at 250°C with a heater. Then, multilayer thin films started to absorb H₂ gas. After about 14h, H₂ gas was evacuated and simultaneously the samples were heated up by the ceramic heater up to 500~900°C. Anomalous heat generation was observed by the process of heating up samples. The excess heat was evaluated by the thermocouple embedded in the ceramic heater.

Up to now, maximum released excess energy reached 1.1MJ and average released energy per absorbed total hydrogen was 16 keV/H or 1.5 GJ/H-mol. It cannot be explained by any known chemical process. It was suggested that it was the consequence of the Condensed Matter Nuclear Reactions.

We have carried out pre-experiments for heat assessment taking account of heat radiation from multilayer thin films using a radiation thermometer. As a result, heat burst phenomena were simultaneously detected by a radiation thermometer and the thermocouple in the ceramic heater. It shows that heat measurement using the thermocouple correctly reflects the surface temperature detected by the radiation thermometer. We are planning to evaluate excess heat by combing the thermocouple with the radiation thermometer.

References

Acknowledgement
This work was supported by Research Center for Electron Photon Science of Tohoku University Electron Photon, Tanaka Kikinzoku Memorial Foundation and Thermal & Electric Energy Technology Foundation (TEET).
Elemental Analysis for Elucidation of the Anomalous Heat Generation Phenomena

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It has been reported that anomalous excess heat was observed using some metal composite absorbed hydrogen gas [1, 2]. Nano-sized metal multilayer composites on Ni substrate and hydrogen gas induced heat bursts and excess energy generation. However, the reaction mechanism and the conditions to cause these phenomena to have not been cleared. To obtain the key factors to understand what is happening in a nano-sized metal multilayer composite, we have been studying on reaction products using various analysis methods.

Surface of the multilayer thin films on a Ni substrate, prepared by Ar ion beam sputtering method, was used as the nano-sized metal composite. Multilayer samples were absorbed hydrogen gas and conducted to heat up to 500~900°C. To understand about the surface states, depth profiles and mass spectra for before and after experiment samples, we performed to analyze each sample using scanning electron microscope (SEM)/energy dispersive X-ray spectroscopy (EDX), transmission electron microscope (TEM) and time of flight secondary ion mass spectrometry (TOF-SIMS).

As a result, the surface states of the samples which were subjected to heating up to 500~900°C after hydrogen gas absorption were different from reference samples. The characteristic elements including C, O, Na, Mg, Al, Si, Zr, K, Ni and Cu were detected by SEM-EDX in a nano-sized metal multilayer composite sample that generated the anomalous heat. The sample was fabricated with Cu and Ni layers in 96 nm thickness. Also, the depth image of the sample with 96 nm layers of Cu and Ni indicated non uniform distribution for each element. We found that it did not remain the layer composition by the depth profile using TOF-SIMS.

On the other hand, nano-sized metal multilayer composites formed by Cu and Ni with CaO or Y₂O₃, which showed 10³ ~ 10⁴ eV order excess energies per hydrogen, indicated difference surface state. However, the behavior of Ca or Y derived from CaO or Y₂O₃ containing Cu and Ni multilayer by the depth profile using TOF-SIMS was similarly near the surface; they did not depend on each element. We guessed that any elements diffused through the similar process and interaction. To understand the relationships among anomalous heat generation, reaction products and elemental distribution, we will continue to examine using various analysis methods.

References

Takehiko Itoh\textsuperscript{1,2}, Yoshinobu Shibasaki\textsuperscript{1}, Jirohta Kasagi\textsuperscript{1}, Shouichi Murakami\textsuperscript{2}, Mari Saito\textsuperscript{2} and Yasuhiro Iwamura\textsuperscript{1}

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Since 2015, Tohoku University has established a division of Condensed Matter Nuclear Reaction, and has been conducting research on anomalous heat generation phenomena using hydrogen and nano-sized metal composite. As a result, we have succeeded in observing the anomalous heat generation phenomena that cannot be explained by the chemical reaction\textsuperscript{[1]}\textsuperscript{[2]}. At the same time, it should be noted in the experimental results that no γ-ray transitions from reaction-generated nuclei, which are supposed to be caused by the nuclear reaction, are observed at all.

However, there has been little search for electromagnetic radiation in lower energy than 50 keV. If the novel nuclear reaction occurs, a region with very high energy density would be formed locally in the condensed matter. Then, expected are emissions of low-energy photons associated with energy dissipation from the local high-energy-density spot. The unexplored low-energy region is very wide; from the X-ray region, where electron bremsstrahlung and characteristic X-rays of the host metal can be expected, to the infrared region where thermally radiated photons are expected mainly. Of particular interest is the spectrum of the visible light, because it may indicate thermal radiation from a local high-temperature region (hot spot).

In previous experiments, it was impossible to observe such low energy photons, since the heat-generating nano-sized metal composite is shielded by the stainless-steel container; even if the low energy photons are emitted, we cannot observe them. Therefore, we made a vacuum chamber that enables photon observation from the infrared region to the X-ray region, and started measuring photons accompanying the anomalous heat generation.

Our experimental process is as follows. First, we use samples in which Ni / Cu nano multilayer films are formed on Ni substrates by sputtering. These are fixed on both sides of a ceramic heater installed in the vacuum chamber. After baking, we made hydrogen absorbed (200-300Pa, 250°C, 15 hours) to the sample. After that, we heated the samples up while evacuating the chamber, and induced anomalous heat generation. We measured the sample temperature with a thermocouple installed inside the ceramic heater and evaluated the heat generation by comparing it with temperature of a reference sample (Ni substrate without a nanostructure).

The following two instruments were installed for the measurement of low-energy photons. (1) For low-energy soft-X-rays (1keV-100keV), we made it possible to detect with an SDD detector (XR-100SDD; AMPTEK Inc.) through a Be window installed in the chamber. (2) For visible light measurement, a spectroscope using amp array CMOS image sensor (C12666MA; Hamamatsu Photonics K.K.) was installed, and the visible light transmitted through the viewport was guided to the spectroscope using a fiber so that the spectrum (340-780nm) could be measured.

Details of the experiments and results will be reported.

References
Enhancement of Excess Thermal Power in Interaction of Nano-Metal and H(D)-Gas

Akito Takahashi\textsuperscript{1,2}, Toyoshi Yokose\textsuperscript{3}, Yutaka Mori\textsuperscript{3}, Akira Taniike\textsuperscript{3}, Yuichi Furuyama\textsuperscript{3}, Hiroyuki Ido\textsuperscript{2}, Atsushi Hattori\textsuperscript{2}, Reiko Seto\textsuperscript{2}, Joji Hachisuka\textsuperscript{2}
\textsuperscript{1}Prof. Emeritus Osaka University, \textsuperscript{2}Technova Inc., \textsuperscript{3}Kobe University

Latest results on anomalous heat effect (AHE) by interaction of binary nano-composite metal powders and H (or D) gas, after the NEDO-MHE project (2015-2017), were reported by our ICCF22 presentation and paper.

[See: https://www.researchgate.net/profile/Akito_Takahashi/research].

Re-calcination of PNZ (Pd\textsubscript{1}Ni\textsubscript{10}/zirconia) and CNZ (Cu\textsubscript{1}Ni\textsubscript{7}/zirconia) powders was found to be effective for the enhancement of weeks-sustaining excess thermal power Wex. We report further results by additional calcinations and baking treatments in this paper. In Table -1, summary results for PNZ10, PNZ10r and PNZ10rr samples with D (deuterium)-gas are given. Detail of data will be shown in the meeting. Study by CNZ7, CNZ7r and CNZ7rr samples with H (light hydrogen)-gas will also be reported.

Table-1: Summary of AHE data for PNZ10 (1kg), PNZ10r (0.45 kg) and PNZ10rr (0.438 kg) with D-gas, by elevated temperature runs (r: second calcination, rr: third calcination), (#2: after second baking, #3: after third baking)

<table>
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<th>Heater Input (W)</th>
<th>Wex (W/kg); PNZ10</th>
<th>Wex (W/kg); PNZ10r</th>
<th>Wex (W/kg); PNZ10rr</th>
<th>RC Av-Temp (°C); PNZ10</th>
<th>RC Av-Temp (°C); PNZ10r</th>
<th>RC Av-Temp (°C); PNZ10rr</th>
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</tbody>
</table>

sample weight: 1kg  sample weight: 0.45 kg  sample weight: 0.438 kg
Irregular oscillation pattern during heat generation from Ni-Zr based alloys and hydrogen gas.

Masanobu Uchimura¹, Masanori Nakamura¹, Etsuo Akiba², Rika Hayashi², Kouji Sakaki³
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²International Research Center for Hydrogen Energy, Kyushu University
³National Institute of Advanced Industrial Science and Technology

Kitamura et al. and Iwamura et al. reported they observed about several tens-watt anomalous heat generation and sudden rise of pressure and gas temperature using their original evaluation system when hydrogen isotope gas was introduced into the nano-metals [1, 2]. To apply this technology to industrial products such as automotive heaters, further output power enhancement is required. However, there is no distinct strategy for higher output since relation of output to experimental parameters is unclear. The conventional evaluation system is suitable for long time and large scale experiment (several months and a few hundred-gram of the material) and it is hard to change experimental conditions frequently.

In this work, we investigated the relation of output to temperature, pressure, interval of data acquisition and gas atmosphere using partially oxidized Pd-Ni-Zr alloys (PNZ) and non-oxidized NiₓZrₓ alloys with a differential scanning calorimeter (DSC) without exposure to air. In addition, sample structure and hydrogen storage property were analyzed using an in-situ X-ray diffraction (XRD) and pressure-composition isotherm measurement.

The DSC experiments revealed irregular oscillation pattern associated with heat generation was observed reproducibly using not only PNZ but also NiₓZrₓ alloys. Additionally, we found the width of oscillation was increased as temperature increase or hydrogen pressure decrease. When we changed the gas atmosphere from hydrogen to inactive gas, the oscillation was terminated immediately. This indicates hydrogen in the gas phase is necessary to occur the oscillation. The change of sample structure and P-C-T isotherm suggest the oscillation relates a phase transition of metal hydrides.

References
Start-up of Metal Crystal confinement Fusion Reactor
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The author announced that excess heat after heavy water electrolysis experiments would be generated by "Nuclear Fusion Mechanism in Metal Crystals[1]". In the mechanism, first, deuterium nuclei of intermetallic atoms are scattered by charged particles such as α-rays to generate high energy deuterons, which react with deuterium nuclei to generate 4He, react with the 4He nucleus to generate 6Li, furthermore, react with the 6Li nucleus and causes 2α reaction, and the α rays generated by the 2α reaction generate high energy deuterons again. These chain reactions create excess heat.

In fact, the production of 6Li had been confirmed from the Pd cathode used in the heavy water electrolysis experiment of Okamoto et al.[2], and 4He and α-rays had been confirmed in the experiment to deform the Pd sample in which deuterium is dissolved of Yamaguchi et al.[3] These suggests the validity of the mechanism.

The author presented the basic structure of “Metal crystal confinement Fusion Reactor” based on the mechanism at JCF-19. The author manufactured an experimental reactor with the structure. The reactor had a structure in which a Pd sample in which 6Li was doped and an α-rays souse were placed, and would be started by sending deuterium gas into the reactor.

And the author made a standard sample in which 6Li was doped and measured the standard sample by SIMS, and estimated the concentration of 6Li at the site where 6Li formation was prominent of the Pd cathode of Okamoto et al.[4] to 0.059 mol% from the ratio of secondary ions of 6Li and 106Pd.

When the author used a sample that was 99.95% pure Pd that had been annealed in air at 1300°C for 1h and had been doped with 180 keV of 5.1×10^{15}/cm² 6Li ions; the generation of γ-rays were confirmed for over an hour ,and also big neutron bursts were confirmed when deuterium gas was increased. However, when the author used a sample that was 99.95% pure Pd that had been baked in vacuum at 1200°C for 2.5h and had been doped same amount of 6Li ions; some consecutive small neutron bursts were confirmed, but no clear generation of γ-rays was confirmed.

References
Microstructure of Pd rod electrode during repeated cathodic and anodic electrolysis in glycerin-phosphoric acid: Hydrogen electrode: 
\[ \text{H/Pd} > \beta_{\text{min}} \text{ with vacancy} \]

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Introduction

Long-term electrolysis for well annealed thick Pd rods (9.0 and 21 mm diameters) in 0.1 M LiOD was performed. Microscopic observation of post-electrolysis Pd electrode showed the surface morphology of surface voids, two long faults without any crack [1]. For a precise understanding of surface and cross-sectional morphology of deuterated Pd, in situ measurements of the electrode potential, dilation, and resistance are of interest under well-controlled H absorption [2]. In this study we adopted the electrolysis method to in situ measurements in an aqueous system, because the experimental data of Pd-H system is considered to be identical to the Pd-D one.

Cold fusion experiments at ambient temperatures were conducted by electrolysis of heavy water on a Pd electrode. For in situ measurements of potential, dilation and resistance the absorption and desorption of H in Pd rod electrodes was performed by applying galvanostatic cathodic and anodic pulse currents, current densities, < 2 x 10^{-3} A/cm^2. The electrolyte was composed of glycerin and phosphoric acid (2:1 in volume ratio). The electrolytic cell and apparatus and detailed procedures are described elsewhere [3].

Results and discussion

Figure shows plots of the potential (Electrode potential) as a function of H/Pd ratio under the first C mode. The abscissa indicates logarithmic scale of H/Pd ratio offset by C(\(\beta_{\text{min}}\)), thus the potential shifts might respond to an activity increase with an increase of H concentration in single \(\beta\) phase. In single \(\alpha\) phase the potential obeys the Nernstian behavior: the potential exhibits a straight line whose slope is coincident with -62 mV/decade vs. the logarithmic scale of H/Pd. As shown in the figure the potential deviates from the dashed line calculated from Nernst's equation considerably. Alternatively at H/Pd > \(\beta_{\text{min}}\) the peak of the apparent molar volume accompanied with the potential shift is tentatively explained as follows: this peak is at the transition from the apparent volume recovery region to the next state (designated as \(\beta_{\text{tr}}\)). Applying thermodynamic analysis to vacancy-H interaction, according to Kirchheim's paper [4], we examined preliminary source data, i.e., equilibrium H pressure and \(n_V\) (vacancy concentration). As shown before hydrogen absorption in single \(\beta\) phase exhibits unusual and complicated behavior, which is attributed to heavily strained solid, vacancy formation accompanied with hydrogen insertion, macro voids etc. Hence we begin to investigate vacancy ordered Cu_{3}Au structure as single \(\beta\) phase (H/Pd > 0.6). We adopt fixed \(n_V\) as first approximation along with an increase of H/Pd over \(\beta_{\text{min}}\).

References
A theoretical study on the possible change of the phonon dispersion relation due to the nuclear reaction in two-dimensional lattice II

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Abstract

It is well known that phonon dispersion relations of crystalline solids sensitively depend on the lattice structures. So, lattice defects or impurities change phonon dispersion relations from those of pure crystals. As we know, nuclear reactions in solids, we interested in, change the sizes of impurities and the number of impurities. This means that we can detect the nuclear reaction in solids by observing the states of phonon.

In our previous works, we showed the possibility of the change of phonon dispersion relations due to the nuclear reactions in solids by using one-dimensional [1,2] and two-dimensional [3] theoretical models. For example, we consider the following reaction. When some impurity ions get closer through the two-dimensional lattice gaps, they collide with some probability and the nuclear reactions occur. After that, the structures of the crystal will change and it makes changes of phonon dispersion relations. If they are uniform reactions in macroscopic lattice, they can be easily detected. However, it is not easy, because they are local reactions.

In this study, we show the method how to detect the local changes of lattice defects by observing the change of phonon dispersion relations.

References

1. K. Tsuchiya, ”A theoretical study on the possible change of the phonon dispersion relation due to the nuclear reaction in solid”, proceedings of JCF9, p80.
2. K. Tsuchiya, “A theoretical study on the possible change of the phonon dispersion relation due to the nuclear reaction in solid II”, proceedings of JCF10, p59
3. K. Tsuchiya, “A theoretical study on the possible change of the phonon dispersion relation due to the nuclear reaction in two-dimensional lattice”, proceedings of JCF19, p77
For occurring of nuclear fusion and/or nuclear transmutation in metal lattices, two or more nuclei (proton or deuteron) of H or D atoms have been condensed. These H or D atoms will cause some trigger reactions while they gather to generate high density cohesion state inside the metal surface during the diffusion process prior to the condensation of p or d. Then, we estimated the differences of total energy (transition energy), following to in Al metal lattice, when an H atom transited between T sites and O sites in Cu, Ag and Au metal lattice by computer simulation, which were carried out by using Nudged Elastic Band method on a computer simulation program Quantum ESPRESSO of the first principle molecular dynamics using a PC.

As a result, the total energy of T sites in Cu metal lattice became periodically higher about 0.3 ~0.5 eV than that of O sites because of the lattice periodicity even with the surface and volume relaxation in which the metal atoms of lattice points could move. However, the energy differences between T sites and O sites in Ag became smaller than in Cu, and were not clear in Au similar to the last Al metal lattice, which were observed without the surface and volume relaxation. This suggests that H atoms can easily stay on the O sites of Cu metal lattice (be occluded), but hardly stay on O/T sites of Al, Ag and Au metal lattices.

Recently, a paper was published that photons are produced in inelastic collisions of electrons moving in metals with an energy above 0.5 eV which appears in the noticeable fraction of energy distribution of electrons above average about 0.1 eV (a temperature of about 1000 ℃), and then they are unlikely to break up into a pair of electron neutrino-antineutrinos of very small mass believed not to exceed 0.28 eV which provably cause rearrangements of nucleons with electron absorption of low energy nuclear reactions (LENR) due to weak interactions. Applying this theory to Cu metal lattice, we can expect the mentioned LENR will more easily occur at room temperature (average energy about 0.03 eV) when some electrons around H atoms which transit the energy barrier of about 0.3～0.5 eV between T sites and O sites are excited to above about 0.1 eV. Furthermore, this will be related to lesser occurrence of LENR in Al, Ag and Au metal lattices of lower energy barrier between T sites and O sites.

The movement of H or D atoms in metal lattices is considered to be essential to LENR in electrolysis, gas permeation, gas occlusion, and so on, then we are investigating the occurrence of LENR under vibratory agitation of aqueous metal chloride solutions.

References
Verification of anomalous heat detected by differential scanning calorimetry from Palladium-Nickel-Zirconium alloy in hydrogen flow (2)

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It has been reported that anomalous heat different from hydrogen absorption heat is maintained for several weeks when hydrogen or deuterium gas is in contact with some metal alloys at a temperature of 200-300 °C. The total amount of the heat is so large that it cannot be explained by only chemical reaction [1]. However, not only the mechanism of such anomalous heat generation but also the heat generation condition has not been revealed. To clarify the condition, it is important to evaluate the anomalous heat from a small amount of metal alloy sample that has a uniform temperature distribution.

In this work, we examined the anomalous heat generation from a small amount of palladium-nickel-zirconium alloy (PNZ) by differential scanning calorimetry (DSC) under various conditions, which is aimed to clarify abnormality of the heat generation phenomenon.

The DSC (Linseis PT1600H) measurements were conducted as follows. 100 mg of PNZ sample (provided by Nissan Motor Corp.) was heated at 5 °C/min to a predetermined temperature and then kept at the temperature for more than 4 h, during which the heat behavior of the sample was measured by DSC. In that manner, a sample was measured in both hydrogen and inert gas (helium, argon), and the difference of the heat flow between them was determined as the heat generation derived from hydrogen.

First, nickel or zirconia sample which is a component of PNZ showed no difference in the heat flow between hydrogen and helium. On the other hand, in case of PNZ, the heat flow in hydrogen was significantly larger than that in helium, and the difference was maintained for more than 4 h. The heat flow difference is often due to changes in the specific heat of the sample, so we examined the heat flow difference when the temperature was changed periodically at 400±5 °C. If the specific heat of the sample varies with hydrogen and helium, the heat flow difference must be constant even if the temperature changes periodically. However, it was observed that the heat flow difference changed periodically and that the change was out of phase with the temperature change. These results indicate that PNZ continuously emitted the heat derived from hydrogen at a constant temperature.

Such heat generation was observed at above 250 °C and the maximum heat flow was obtained around 400 °C, while hydrogen absorption was observed below 250 °C. Despite the maximum exotherm at 400 °C, the hydrogen desorption from PNZ was observed at 400 °C by a temperature-programmed-reduction (TPR) method. These results clearly show that the heat generation from PNZ in hydrogen is not due to hydrogen absorption. Next we examined the phase transition in PNZ by powder X-ray diffraction (XRD) method, since metal alloys can undergo phase transitions at high temperatures, often generating heat. As a result, it was suggested that no phase transition occurred in PNZ below 450 °C in hydrogen and inert gas. Therefore, it was clarified that the continuous heat generation from PNZ in hydrogen above 250 °C was not caused by hydrogen absorption heat and phase transition heat.

Reference
Calibration of Sendai mass-flow-calorimeter and heat measurements of recycled samples

Masahiro Takeya¹, Takehiko Itoh¹², Jirohta Kasagi¹ and Yasuhiro Iwamura¹
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²CLEAN PLANET Inc., 105-0022 Japan

The mass-flow-calorimeter [1] developed by the Kobe University—Technova group has enabled accurate heat flow measurement in heat generation experiments in the composite metal nanoparticle-hydrogen (deuterium) gas system up to a temperature of about 300°C by using oil coolant. In the NEDO project, we made basically the same calorimeter, conducted several experiments using the same sample as the Kobe-Technova group, and demonstrated the reproducibility of the anomalous excess heat generation phenomenon [2].

Now, we investigate further improvements in the thermal measurement in a search for X-ray radiations as well as reaction residual gases associated with excess heat generation. The following has been carried out. (1) Perform a calorimeter calibration by adding / changing measurement parameters without changing the current status, and (2) Calibrate the calorimeter by directly taking in thermal radiation emitted from the reaction chamber in addition to the measured quantity so far.

In (1), it was found that the discrepancies between calibrations performed at different times were reduced by changing the measurement position of the oil coolant outlet temperature.

In (2), measurement points of temperature were newly added on the side wall of the reaction chamber, and the loss of thermal radiation from the reaction chamber was estimated from these data. This makes the flow of heat-loss much clearer, but contributes little to improving the measurement accuracy.

We will report the results of these experiments in detail, and also report the results of the calorimetric measurements of the PdNiZr and CuNiZr samples (recycled samples).

References
Deuterium Desorption Experiment Using Surface Coated Pd Foil with Fine-structured Interface

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Excess heat evolution has been observed in deuterium absorption/desorption process for nano-composite-particle of Pd-Ni-Zr sample [1]. In addition, Yamaguchi et al. has reported that excess heat and helium generation were observed during deuterium desorption from a surface-coated Pd foil with Au and MnO [2]. These phenomena are supposed to be related to a low-energy nuclear reaction in condensed matter. Although the mechanism has not been clarified yet, it is considered that the sample conditions such as nanostructured-metal and the complex composition might be the key for inducing the phenomena.

We have conducted the deuterium desorption experiment using the sample fabricated by depositing various metal membrane on a Pd foil, and investigated temperature of the sample and deuterium diffusion behavior. For the Pd-Ni sample or the Pd-Ni-Zr sample with fine-structured interface, we observed a peculiar temperature variation [3, 4]. It is suggested that the characteristic diffusion of deuterium might happen due to the specific conditions. Although significant excess heat has not been observed in the experiments, we may find out a trigger condition to induce the excess heat by investigating the deuterium diffusion behavior systematically with various type of samples.

Now, in order to clarify the conditions to induce the peculiar temperature behavior observed for a Pd-Ni with fine-structured interface, and to improve reproducibility of the phenomenon, we tested the Pd foil with a finer structure at the surface by laser processing.

In addition, we quantitatively evaluated the hydrogen diffusion coefficient of the multi-layered metal membranes and characterized the behavior.

Reference
Development of reaction system with small chamber for fundamental experiments measuring anomalous heat effect

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It is known that anomalous heat is generated when hydrogen or deuterium is absorbed by nickel or palladium \([1-4]\). In this work, we developed reaction system with small chamber and conducted fundamental experiment to validate anomalous heat effect occurring. Reaction system is shown in Fig. 1. About 2mL reaction chamber is made from SUS316 and SUS316L. To make measurement of temperature increase easy, small test tube is used as heat insulating material. Sample (PNZ10r, provided by Technova Inc.) is put in small test tube inside of reaction chamber. Sample temperature is measured by K-type thermo couple (HTK0227, made by Hakko electric co., ltd.). Scroll pump (ISP-250C, made by ANEST IWATA Corporation) is used for evacuation. Bead bath (MD-MINI, made by Major Science) is used to warm reaction chamber. Experimental conditions are shown in table 1. To confirm temperature increase, mixture of hydrogen and PNZ10r is used, whereas mixture of nitrogen and PNZ10r is also employed. Experimental result is shown in Fig. 2. When hydrogen gas is absorbed by PNZ10r, about 3K temperature rise is observed. This temperature increase is larger than that of control experiment using nitrogen.

Table 1. Experimental conditions

<table>
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<th>Experiment number</th>
<th>No. 1</th>
<th>No. 2</th>
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<tr>
<td>Kind of gas</td>
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<tr>
<td>Gas pressure</td>
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</tr>
<tr>
<td>Temperature of bead bath</td>
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</tr>
<tr>
<td>Sample mass</td>
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</tbody>
</table>

Fig. 1. Reaction system with small chamber

Fig. 2. Experimental result


Laser Irradiation to D-Loaded Pd

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A gas-phase experimental research in quest of condensed-matter fusion is underway by using multilayered deuterium-containing Pd plates. In our experiment, we in particular directly apply a bias voltage across the Pd sample to provide a current injection through Pd, to stimulate the nuclear reaction by Joule heating, also anticipating strong electrodiffusion or electromigration, in addition to the conventional deuterium diffusion induced by pressure/mass-concentration and thermal gradients.

The intensity and density of the triggering energy supplied to activate the nuclear fusion reaction are key factors to produce a smooth and reproducible initiation of the reaction. We previously proposed and numerically analyzed a scheme to provide high-density optical or electromagnetic energy to fusion-fuel materials by lasers and plasmonic field-enhancement effects, to significantly increase the reaction probability [1–3].

Our experimental setup is a stainless-steel-made, gas-phase, clustered reactor system including a deuterium-loading chamber, an electron-beam deposition chamber, and a reaction-analysis chamber. A high-resolution small-amu quadrupole mass spectrometer, two gas proportional neutron detectors, a Geiger-Mueller detector for $\alpha$, $\beta$, and $\gamma$ rays, and two liquid scintillators are equipped to the facility. In the present work, we have installed multiple kinds of lasers in the gas-phase D-Pd reaction system to irradiate the Pd samples, as an energetic stimulation support, potentially with a boosting plasmonic local field-enhancement effect. The lasers are 405-nm and 594-nm continuous-wave semiconductor lasers with powers of 50 and 30 mW, respectively, and a 1064-nm pulsed YAG laser with a peak power of 10 MW.

As the preliminary result, we have simultaneously observed a sudden temperature increase with an overshoot and a neutron signal. Significantly, we have observed a clear signal of substantial-amount $^4$He generation from the Pd samples as a shoulder peak on the $^2$H peak, and a gas species with a mass number of three, via in-situ mass spectroscopy. We have also observed a sudden burst of these gas species out of the Pd sample. Our results might indicate a certain anomalous nuclear-related reaction in the D-Pd system. The experimental conditions and further outcomes will be presented in detail at the meeting.

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In recent years, it has been reported that anomalous heat different from hydrogen absorption heat is detected when some metal alloys are heated to 200-300 °C in deuterium or hydrogen. Since the anomalous heat generation lasts over several weeks, the total calorific value becomes extremely large. Such anomalous heat has been observed in metal alloy samples such as Pd-Ni-Zr, Cu-Ni-Zr, and Cu-Ni-Si [1, 2]. In order to utilize such anomalous heat industrially, it is important to find a new material showing greater output of the anomalous heat.

In this work, for the purpose of finding a new metal system which causes the anomalous heat generation, we examined the exothermal / endothermal behavior of several systems composed of binary metals based on Ni, Al, Ti, Ca and so on by using a differential scanning calorimeter (DSC) under hydrogen stream.

In the experiment, two kinds of commercially available metal powders were physically mixed, and then set to DSC. For pretreatment in DSC, the mixed sample was pre-heated in helium flow to a temperature higher than the melting point of one of the mixed metals, and cooled to room temperature. During this pretreatment, some metal alloys were formed in the sample. For measurement, the sample was heated to a predetermined temperature (300 to 800 °C) at 5 °C/min and kept constant for 2 hours at that temperature, during which the heat behavior of the sample was measured by DSC. The mixed sample was measured both in hydrogen and helium, and the difference in the heat flow between them was regarded as exothermal / endothermal heat derived from hydrogen.

As a result, we found for the first time that some binary metal systems generates anomalous heat at 500-800 °C. The samples of Al-Ni binary system showed the exothermic heat flow over 300 mW/g which was very large compared with 20 mW/g obtained with the same DSC apparatus for Pd-Ni-Zr sample. Similarly, Al-Ti (40 mW/g at 700 °C) and Al-Ca (90 mW/g at 400 °C) binary systems exhibited large anomalous heat at a high temperature. These samples were repeatedly evaluated, resulted in high reproducibility with respect to anomalous heat generation.

The crystal phase of the samples was analyzed by XRD. It was found that the samples showing the anomalous heat for the DSC measurement always include two kinds of metal alloys.

Reference