

Cu-Ni-Mn alloy wires, with improved sub-micrometric surfaces, used as LENR device by new transparent, dissipation-type, calorimeter.

Francesco Celani⁽¹⁾, E. F. Marano⁽¹⁾, A. Spallone⁽¹⁾, A. Nuvoli⁽¹⁾, E. Purchi⁽²⁾, M. Nakamura⁽²⁾, B. Ortenzi⁽¹⁾, S. Pella⁽¹⁾, E. Righi⁽¹⁾, G. Trenta⁽¹⁾, S. Bartalucci⁽¹⁾, G. L. Zangari⁽²⁾, F. Micciulla⁽¹⁾, S. Bellucci⁽¹⁾.

(1) National Inst. Nucl. Physics, Frascati Nat. Laboratories, Via E. Fermi 40, 00044 Frascati (RM)-Italy.

(2) ISCMNS, Latium1 Group, Via Cavour 26, 03013 Ferentino (FR)-Italy.

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*** Metallurgical Company of NE-Italy.**

*** Kresenn Ltd, (Angelo Ovidi principal investigator); 1Bell Street, SL6 1BU, Maidenhead-UK.**

***Dennis Letts Laboratory, 12015 Ladrido Ln, Austin, TX 78727-USA.**

***National Instruments, 11500 North Mopac Expy, Austin, TX 78759-USA.**

***Francesco Santandrea, ISCMNS, Latium1 Group, Via Cavour 26, 03013 Ferentino (Fr)-Italy**

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Abstract and motivations

- Starting in February 2011, we studied (secretly) *new Nickel-based alloys* that could be able to absorb some proper amounts of gaseous Hydrogen (H_2) and/or Deuterium (D_2), at mild pressures ($<10\text{bar}$) and that have, in principle, some possibility to generate anomalous thermal effects at temperatures $>100^\circ\text{C}$.
- The deep interest in Ni comes, among others, because: a) there is the possibility to use H_2 instead of expensive D_2 ; b) the much lower cost of Ni in respect to the precious metal Pd; c) reports by F. Piantelli (since 1992), G. Miley (about 1995), M. Patterson, F. Celani (since 2010) and, overall, *claims by A. Rossi and (later on) by Defkalion Company*, could be further investigated. Moreover, *cross-comparison of results using H_2 instead of D_2 can be made and could help the understanding of the phenomena involved (nuclear origin?) because use of such both isotopes (pure and/or mixed).*

- BTW, we started using gaseous environments since 2003 (possibility of larger temperature in respect to electrolysis) and focused on nano-materials after the experimental results of Yoshiaki Arata (Osaka University-Japan) shown at ICCF12 Yokohama Conference (November 2005) on Pd₃₅%-ZrO₂₆₅% nanostructures (used pressurized Deuterium, 60bar, and temperatures up to 210°C).
- Some of our February 2011 targets were achieved and the material now produced, based on in-deep modifications of the low cost commercial material ISOTAN 44 (composition: Cu₅₅-Ni₄₄-Mn₁), seems to be reproducible both about preparation procedures and amount of thermal anomalies.
- The time need to absorb H₂ (initial amount is the largest), is quite short (10-20m). The absorption is temperature activated: between 125 to 150-160°C, depending on sample history. Once absorbed, the H₂ is kept inside the sub-micrometric “skin” (and/or bulk), at Room Temperature, even under free-air or dynamic vacuum conditions (1 week test).

- The amount of anomalous heat increased over time and seems related to its temperature (positive coefficient), over a proper thermal threshold.
- Since June 5, 2012 we were able to improve, largely, the production procedure (active material increased from 1-2% to about 30%) of the sample (wire, diameter 200 μ m, length 100cm): **we will report, in details, only the most recent results with the new wires.**
- At December 2011 we went to know that also the experimental group of **Akito Takahashi** (Osaka University) and **Akira Kitamura** (Kobe University), in an joint collaboration with Technova Laboratories (side of Toyota Company), studied (in secret) an alloy based on **nanoparticles (5-20nm)** of **(Ni₆-Cu₁)_{35%}**, dispersed in the **(ZrO₂)_{65%}matrix**.
 - ***The results obtained from both Takahashi-Kitamura group and ours were qualitatively similar.***

- As key results, we de-loaded the Hydrogen from the wire (called #1) 2 times. The same wire was reloaded, at the end, by D₂ and were observed some weak, but with statistical significance, emission of X and/or gamma ray (energy range 25-2000keV) during the warming up-period (100→160°C). The emission was burst-like. After about 10 days under D₂ the wire broke by itself (fused, T>1200°C, according to SEM observations).
- A second wire (called#2), prepared the same day of the #1 and kept inside a plastic bag was used. The behavior was similar to #1, although the triggering temperature for H₂ intake was higher (150 instead of 125°C). Thermal anomalies were lower in respect to wire#1, although clearly detectable. After a cycle of de-loading and reloading the overall performances improved: it was even possible to evaluate the H₂/Constantan ratio.
- Such wire is still inside the reactor used for a “live” demonstration both in USA (National Instruments Laboratory, Austin-TX) and, hopefully, at Daejeon (South-Korea) for ICCF17 Conference.

Latest Information

- The wire #2 “overcome” the severe conditions of shipping and long time (8 days) without H₂, at free air conditions. When we resumed all the electrical connections, at Austin-Texas-USA, we realized that the R/R₀ value of Active wire remained almost unchanged (about 0.81).
- We point out that, at Austin, **ALL the control and measuring electronics** were provided by **National Instruments** modules. In Italy, we used a mix of instrumentations, starting from hand-made instruments to Agilent, Larcet, Pulsar, National Instruments. The acquisition system, as long time our habit, was based on LabView system (from National Instruments). Anyway, the measured parameters looked not different from that obtained from the previous instrumentations.

- Moreover, we were surprised because the overall performance, like the value of excess power, look improved. In addition, the wire was able to withstand also the condition of *direct heating* with *good success*. In Italy, before shipping, the wire#2 was unable to operate such high-performance, but overall severe, operating condition. Speculatively, the H₂ kept inside the lattice continued to reduce the dimensionality of the material thanks to its intrinsic propriety of fracturing materials.
- The maximum excess power reached, after 3 days of operations (in public) at Convention center of Austin, *NIweek 2012*, was about 21W with indirect heating and about 25W with direct heating of Constantan wire. The input power, as usual, was 48W. They were the best values that we observed up to now. *We remark that, because we used the “old” value of calibration obtained in Italy with different experimental geometric set-up, the absolute value of excess power has to be fully controlled. Anyway, the peculiar trend to increase the excess power versus elapsed time was reconfirmed.*

AN OLD ALLOY USED FOR NEW PURPOSES

- Because theoretical considerations, and thank also to some sentences reported in a paper (on catalysis) not related to LENR studies (Ref.1: S. Romanowski et al.; Langmuir 1999, 15, 5773), we decided to explore the possibility to use the “large family” of **CONSTANTANS alloys** as starting material that could fit our purposes.
- One of the merit factor was, according to us, the ability to decompose H_2 . One of the Constantans (**Ni₃₇Cu₆₃**), among the materials studied in the Ref.1, has the highest value (i.e. **3.2eV**; in comparison, pure **Ni** and **Pd** have respectively values of **1.74** and **0.42eV**) of such decomposition. Moreover, *even with large changes (factor of about 2) in the relative atomic amounts of Ni in respect to Cu (i.e. from 0.37 to 0.62), the decomposition values remain almost constant (from 3.16 to 2.86eV).*

- We focused on a commercial (low cost) material, called **ISOTAN 44**, nominal atomic composition **Cu₅₅Ni₄₄Mn₁**, developed many years ago by Isabellenhutte Heusler, GmbH, KG-Germany. The ISOTAN 44 was selected according to the following, overall, considerations (as pointed out by us at the “*X International Workshop on Anomalies in Hydrogen-Metal Systems*”, Pontignano-Italy, April 10-14, 2012; Ref. 2):
 - a) Measurable **diffusion coefficients of Hydrogen**, in even the pure (not alloyed) elements, i.e. Cu and Ni, at high temperatures: Cu= $10^{-6}\text{cm}^2/\text{s}$ at 200°C, $10^{-4}\text{cm}^2/\text{s}$ at 700°C; Ni= $10^{-7}\text{cm}^2/\text{s}$ at 200°C, $10^{-6}\text{cm}^2/\text{s}$ at 350°C. In comparison, the (good) values for Pd are: $10^{-5}\text{cm}^2/\text{s}$ at 200°C, $10^{-4}\text{cm}^2/\text{s}$ at 420°C; at 600°C were reported values as large as $8 \cdot 10^{-3}\text{cm}^2/\text{s}$, but not reproducible.
 - b) Lower cost, overall, even considering the procedure to “build” nano-structure at the surface, in respect to Pd, a very expensive precious metal.

- c) Very good mechanical properties, especially in respect to aging effects due to cycles of both low→high→low temperatures and H₂ absorption-desorption: the sample of our “old” long time lasting experiment was working for over 7 months. Only after such long time of operations we observed serious damages rising-up.
- c1) Our results are, in some aspects, different from that obtained by A.W. Szafranski (J. of Alloys and Compounds 404-406, 2005, 195-199): he observed extreme brittleness in, as received, Cu-Ni alloy that was only cold rolled from 200μm to 20μm (the penetration depth of H in Ni is about 30μm) and then cycled between 77K and 300K under 1GPa pressure of H₂. We could think, only, that high temperatures (or the small amount, 1%, of Mn) have beneficial effects on reducing brittleness problems. Moreover, we never made experiments at 77K.

- d) Extremely large values of measured catalytic power (ΔE , in eV) in respect to the dissociation of H_2 (Ref. 1), as following:

$$\text{Ni}_{0.3750}\text{-Cu}_{0.6250} \implies +3.16\text{eV}$$

$$\text{Ni}_{0.6250}\text{-Cu}_{0.3750} \implies +2.86\text{eV}$$

$$\text{Ni}_{0.8125}\text{-Cu}_{0.1875} \implies +2.10\text{eV}$$

$$\text{Ni} \implies +1.74\text{eV}$$

$$\text{Ni}_{0.1825}\text{-Cu}_{0.8175} \implies +1.57\text{eV}$$

$$\text{Ag}_{0.8125}\text{-Pd}_{0.1875} \implies +0.57\text{eV}$$

$$\text{Ag}_{0.625}\text{-Pd}_{0.375} \implies +0.51\text{eV}$$

$$\text{Ag}_{0.1875}\text{-Pd}_{0.8125} \implies +0.51\text{eV}$$

$$\text{Pd} \implies +0.42\text{eV}$$

$$\text{Cu} \implies -1.11\text{eV}$$

$$\text{Ag} \implies -1.42\text{eV}$$

- e) The possibility, at least in principle, to produce *nano-micro structures* at the surface, or even deeper into the bulk, because *selective oxidation of Cu in such alloy* at high temperatures (**650-1050°C**). Both segregation of pure Ni among to CuO_x and cooling rate are key aspects of the preparation to be studied in deeper details.
- *Our studies, very exploratory, were devoted to finding simple, and reliable/reproducible procedures to get these kinds of structures. Experiments with the selected material were operated for time as long as possible: “strength” and aging tests.*
- We initially achieved only partial success and produced small amounts of material (only some %) of proper dimensions at nanometric sizes (<100nm) in the previous “old” experiment. Finally, apart the absolute values of dimensions, to be further optimized, we obtained frequently tri-dimensional shapes of geometry, called *Skeleton type*. Such tri-dimensional geometry has several intrinsic potentialities in respect to gas absorption: a paper focused on Skeleton geometries is under preparation by us.

- Starting at January 2012, we developed completely new procedures of preparations, which were tested in June 2012 with a new, transparent, dissipation type, “calorimeter”.
- We would like to point out that multilayer geometrical structure, at nano-metric size, was inspired to us (since about 2003, several papers on multi-element coating on Pd wires were published by us) from the original work performed by Yashuiro Iwamura (and Colleagues) at Mitsubishi Heavy Industry (Yokohama-Japan) since 2000. In their work, aimed to elucidate transmutation of some specific elements (Sr, Cs, Ba), the number of layers was 5 and were used Pd-Deuterium as main “environment”.
- The main difference, and improvements, in the recent construction procedure, is the fact that we used **ONLY one material** (Constantan alloy) and take advantage of its intrinsic, multi-elements, composition. The complexity of preparation and cost were kept at low level. Our process of fabrication, in principle, is similar to process of fabrication of **SELF-SUPPORTED catalyzer**, often used in oil refining processes.

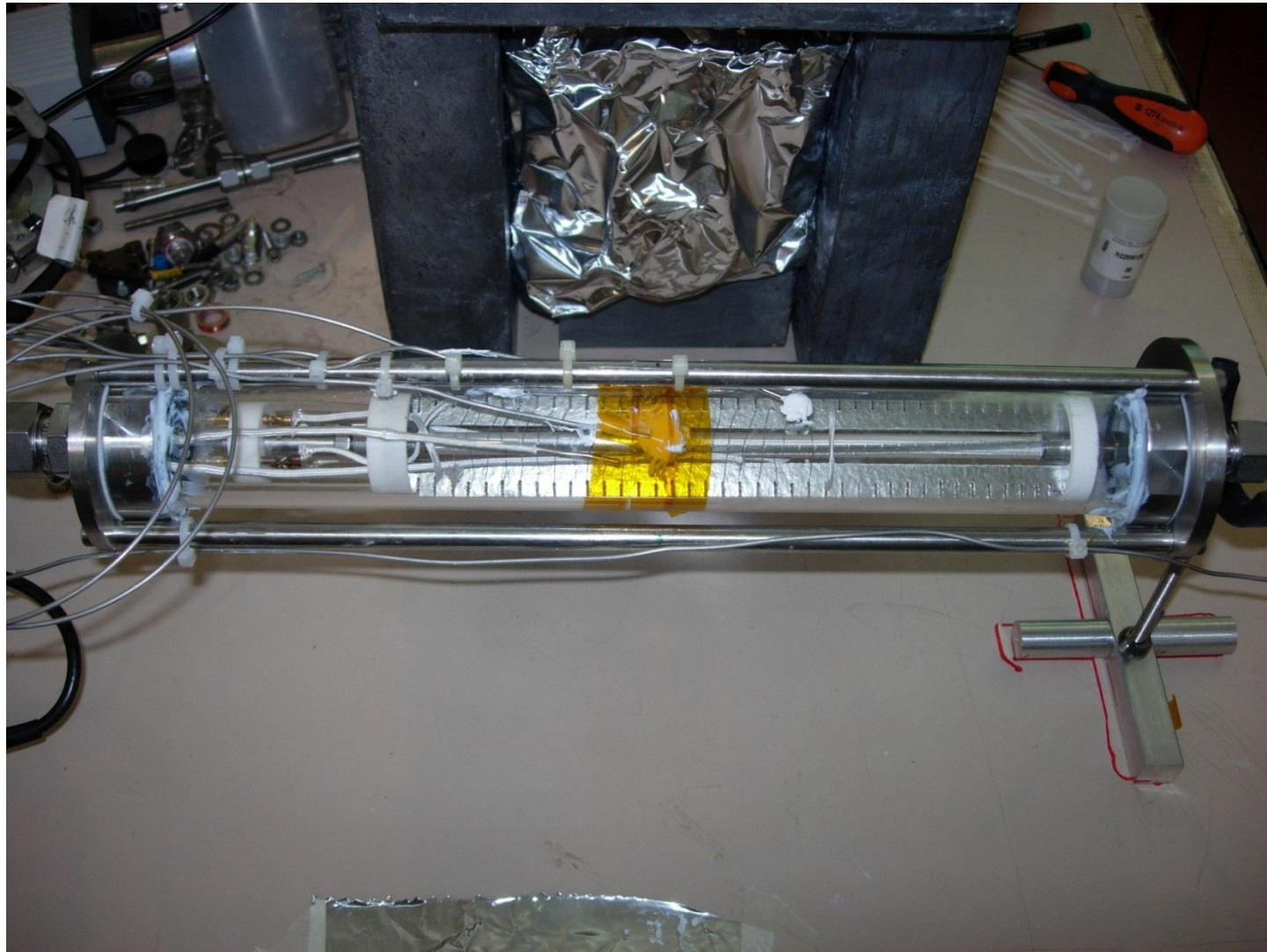


PHOTO OF THE SMALL, DISSIPATION TYPE, TRANSPARENT REACTOR OPERATING AT INFN-LNF.

THE 2 WIRES, REFERENCE AND ACTIVE, ARE ROUNDED ON A MICA SUPPORT. THE THERMOCOUPLES ARE TYPE K, SS SCREENED.

SAMPLE PREPARATION (PROCEDURES USED FOR EXPERIMENT UP TO MAY 2012).

SIMILAR MATERIAL, ABOUT COMPOSITION, DEVELOPED IN JAPAN.

- In our exploratory preparations/tests we used “standardized” wires: (“nuked”) $\Phi=200\mu\text{m}$, $l=105\text{cm}$. Initial values of weight (e.g. 307.4mg), diameter ($\pm 1\mu\text{m}$) and resistance (e.g. 17.16 Ohm) were carefully measured.
- We point out that, although very promising (expected) results with *pure Constantan*, in our explorative test (2-3 days of operations each, time span from February to June 2011) under Hydrogen atmosphere, we **NEVER** got *any type of anomalies* (like changing of resistance) using wires at temperatures up to 900°C, under the following status:
 - (1) as obtained from the Company (we call them ultra-virgin);
 - (2) with the surface cleaned-up from the plastic protection (plastic removed by burning up to 600°C in air, by “heat gun” or Joule heating, see later);
 - (3) acid etching of wire surface, after burning at 600°C.

* The wires, at the beginning (usually), were “cleaned-up” of the original plastic insulating layer (rayon type, as provided by Isabellenhutte) by Joule heating, in air, at current as large as 2000mA, time 5m. In such conditions the power dissipated was about 70W and the resistance ratio, in respect to the reference value (at 100mA of current injected) increased of only 1%, as expected for such kind of material (commercial name is *Constantan*, i.e. constant resistance).

* After first thermal treatment, the weight decreased of about 13mg, the resistance decreased from 17.16 to 17.02 Ohm.

- *We found that increasing both the current (up to 2500-3000mA) and the time at high power (5-1000s), decreasing the cooling speed (from 100s down to <1s) had dramatic effects on the growing of nano-microstructures and their dimensions. The role of O₂, because free air treatment, is quite important. The wire temperature, in some tests, was even larger than 1000°C (rough evaluation by colour temperature; the melting point of pure Cu is 1083°C).*

- The quality of wire produced by this method was evaluated by SEM observations. According to us, as smaller were the particles at the surface and larger the total fraction in respect to the whole wire, as better was the procedure of preparation.
- The “best material” that we were able to produce, at the end of July 2011, using thermal treatments were put in our (high resolution) flow calorimeter.
- As previously noted, such material was extensively studied, both in Hydrogen and Deuterium atmosphere using a very accurate flow calorimeter (indetermination $<2\%$). The time of experiments was really long (over 10 months) and only at the end the damages were so heavy to prevent further reliable interpretation of the experimental results. As quoted before, they were discussed, deeply, during the *X International Workshop on Anomalies...* on last April 2012.

- We were very happy, and surprised, to know that also Akito Takahashi and Akira Kitamura (and Colleagues), respectively from Osaka and Kobe University (Japan), studied in secret (like us), an alloy of Ni-Cu (at nanometric size) dispersed in an inert matrix of ZrO_2 . Such work was performed by them among a collaboration with the Research Group of Toyota Company (Technova). We got some information, by A. Takahashi and A. Kitamura, since January 2012, about promising results on a specific Ni-Cu- ZrO_2 alloy.
- *Such material is a further development/improving of the first historic, in LENR experiments, nano-material Pd_35%-ZrO₂_65% as developed by Yoshiaki Arata (Osaka University), in close collaboration with Tohoku University, since 2005. To get nanostructures they used the so-called “melt-spinning and quenching procedure”.*

- The “short information”, by Takahashi and Kitamura group, came because I was invited to give a “*Review Talk on Anomalous Effects in LENR Studies*”, at the WSEC2012 Conference (World Sustainable Energy Conference 2012) organized by the ISEO (International Sustainable Energy Organization). The ISEO is an ONG linked to several not-politic Organizations (UNESCO, WHO, ILO, WWF, ...) cross-connected to United Nations at Geneva. Obviously, I requested that everybody involved in LENR studies, worldwide, would communicate the most recent and interesting results to include in my talk.

- **The overall behaviour of our sub-micrometric Cu-Ni-Mn alloy, in respect to Hydrogen and Deuterium absorption, and the amount of anomalous heat detected, were, in several aspects, similar to results experienced by the Japanese Colleagues .**
- **Such kind of evidence reinforced our intention to develop a better quality material, keeping the Cu-Ni composition “constant” over time even cycling from low to high temperatures (and vice-versa). In other words, our efforts were devoted to improve the amount of active material at low dimensions ($\ll 100\text{nm}$) and, at the same time, avoid the adverse effect of “leakage” of the smallest particle from the surface.**

NEW TRANSPARENT, DISSIPATION-TYPE, “CALORIMETER”

- From the end of May 2012 we were able to produce sub-micrometric materials, with nominal overall performances several times better than the best material produced at the end of July 2011, with enough good reproducibility about preparation procedures.
- The new method, although started from the old one in some key aspects, was really revolutionary about the practical parameters of: mechanical stability (few “leakage” of the best material from the surface), percentage of material at small dimensions. Such last parameter increased from only 1-2% up to about 30% of the whole material.
- *Such big improvements were obtained because large economical (and man-power) help of an Italian Company that “believed” in our previous results. We were able to design, and achieve, specific electronics and mechanical set-up to produce such kind of sub-micrometric wires.*

- Moreover, because one of our goals was to see, by nuded eyes, if the wire was really stable about the leakage of “good” materials even after several cycles of low—>high—>low temperatures and Hydrogen loading (or even de-loading!), we build a *new transparent reactor* with, high quality, borosilicate glass (Schott DURAN) of large wall thickness to withstand enough large pressure drops (up to 8bar, at wall temperatures up to 250°C).
- For the calorimetric measurements, we adopted the simplified approach to measure the external glass wall temperature. Taking into consideration the temperature of interest, i.e. $T_{\text{wall}} > 100^{\circ}\text{C}$, the main channel of heat exchange to the environment is radiation of heat. In other words, it can be used the simple formula of Stefan-Boltzmann law:

$$P_{\text{out}} = 5.67 \cdot 10^{-8} \cdot (T_{\text{wall}}^4 - T_{\text{room}}^4) [\text{W/m}^2]. \quad (1)$$

In such formula the temperatures T are in K.

- **Calibrations were made using our usual procedure to add an “inert” wire (Ni-Cr), very close to the “active” one, and make several measurements. In the specific new set up, the wires were parallel, alternatively and helicoidally shaped, 22 turns. They were changed the input power, used different gases (He, Ar, Vacuum), fed the electric power alternatively to the inert and “active” wires. The inert wire was also used to allow controlled temperatures of the gas, and active wire, inside the reactor. Vice-versa, using the active wire as heater, its internal temperature, because the specific kind of material used, is impossible to be evaluated.**
- **Because in our real experimental set-up the geometrical dimension of the cell is constant (glass tube, external diameter 40mm, internal diameter 34mm, overall length of 280mm (central active length of 100mm), we can make a calibration curve just dividing the formula (1) by the input power.**

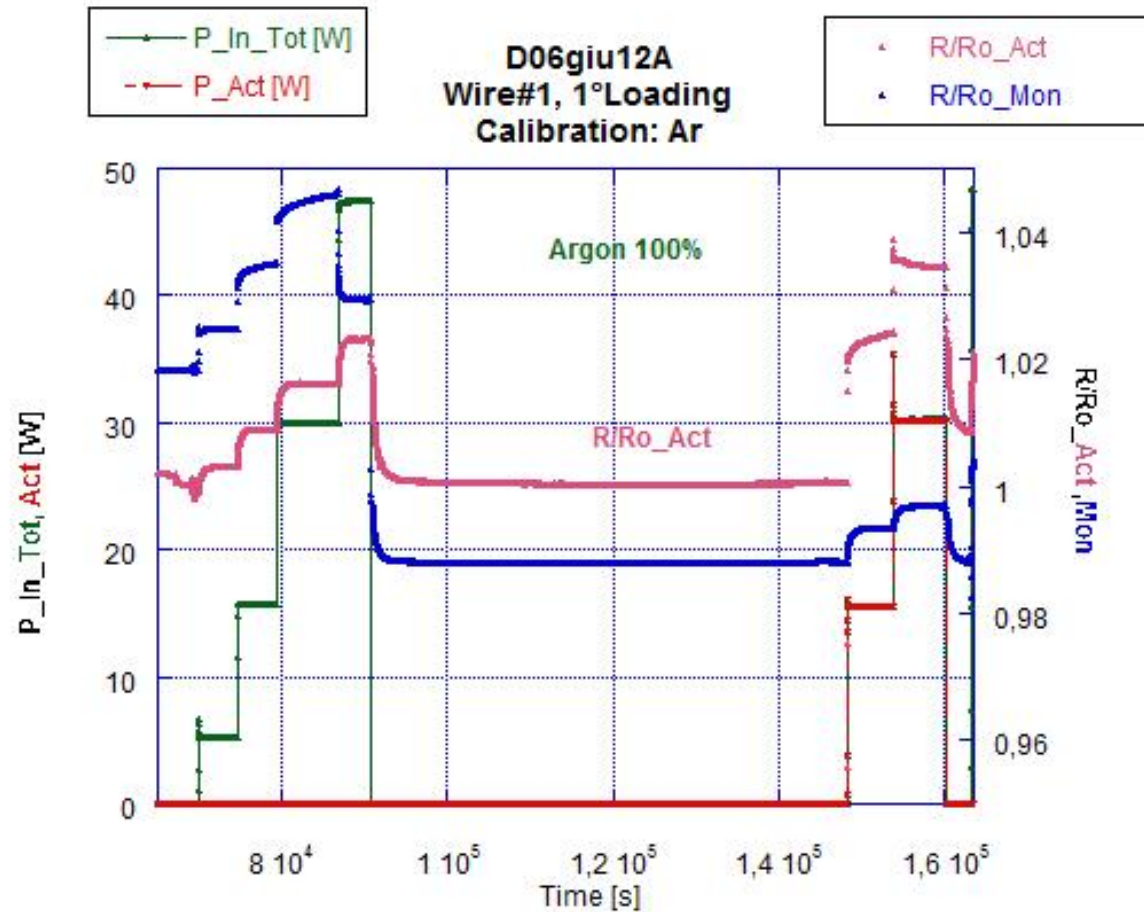
RESULTS WITH THE NEW WIRES

(AFTER JUNE 6, 2012)

- At the beginning of June, 2012 two wires were produced, both with the same nominal procedures.
- One was used few days later to the experiment, the second one was just put inside a HDPE envelop and kept closed at Room Temperature (RT). We named the experiments: [a] wire#1 (started 06 Jun, 2012); [b] wire#2 (started 10 July, 2012).
- The main improvements in respect to previous procedure of fabrication, according to SEM observations, were the multilayered structures **and total number of such layers extremely large: close to one thousand**. The thicknesses, of such multi-layers, were, mainly, in the range **of 20-100nm**. The mechanical stability, against leakage of sub-micrometric materials, was largely improved.

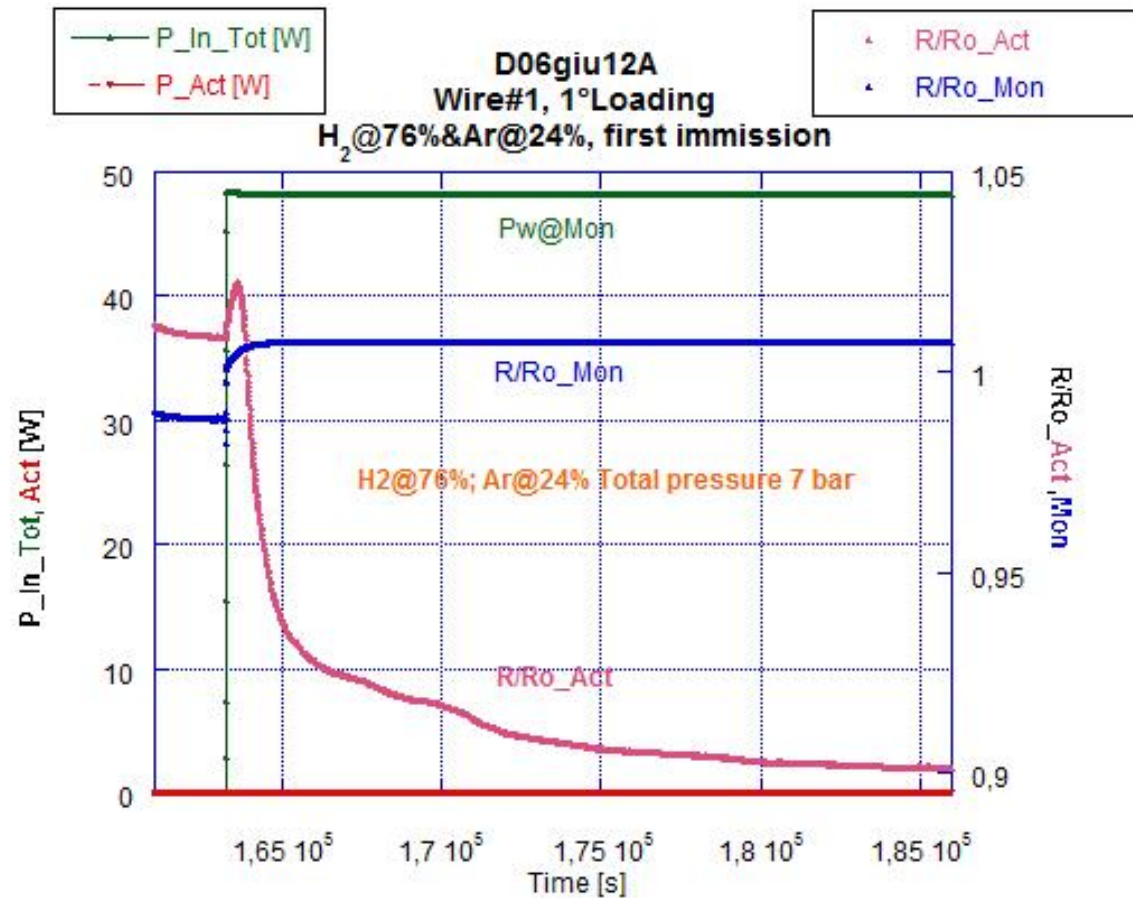
Main experimental procedures and results are listed as following:

- 1) In order to use simple parameters easy to be managed by calculations, we adopted the usual term of R/R_0 , very familiar to the LENR Scientist using Pd. R_0 is the initial value of resistance at RT, i.e. 23.5°C (in that calibration), in free air atmosphere, inside the reactor. With our wires we measured a value of resistance of 16.9684 Ohm and 57.4394 Ohm, respectively for active and Ni-Cr wires. The current injected, for resistance measurement at low power, were just 4mA: no self-heating of the wires.
- 2) First of all, were made calibrations by inert gases, with power of 5, 15, 30 and 48W applied to the inert wire. The maximum internal temperature of the chamber was of the order of 180-220°C, depending on the gas composition. Some tests, as cross reference, were made also on active wire. Using the values of temperatures measured outside the glass cell (and ambient temperature) it was possible to evaluate the power exchange constant of the small reactor.

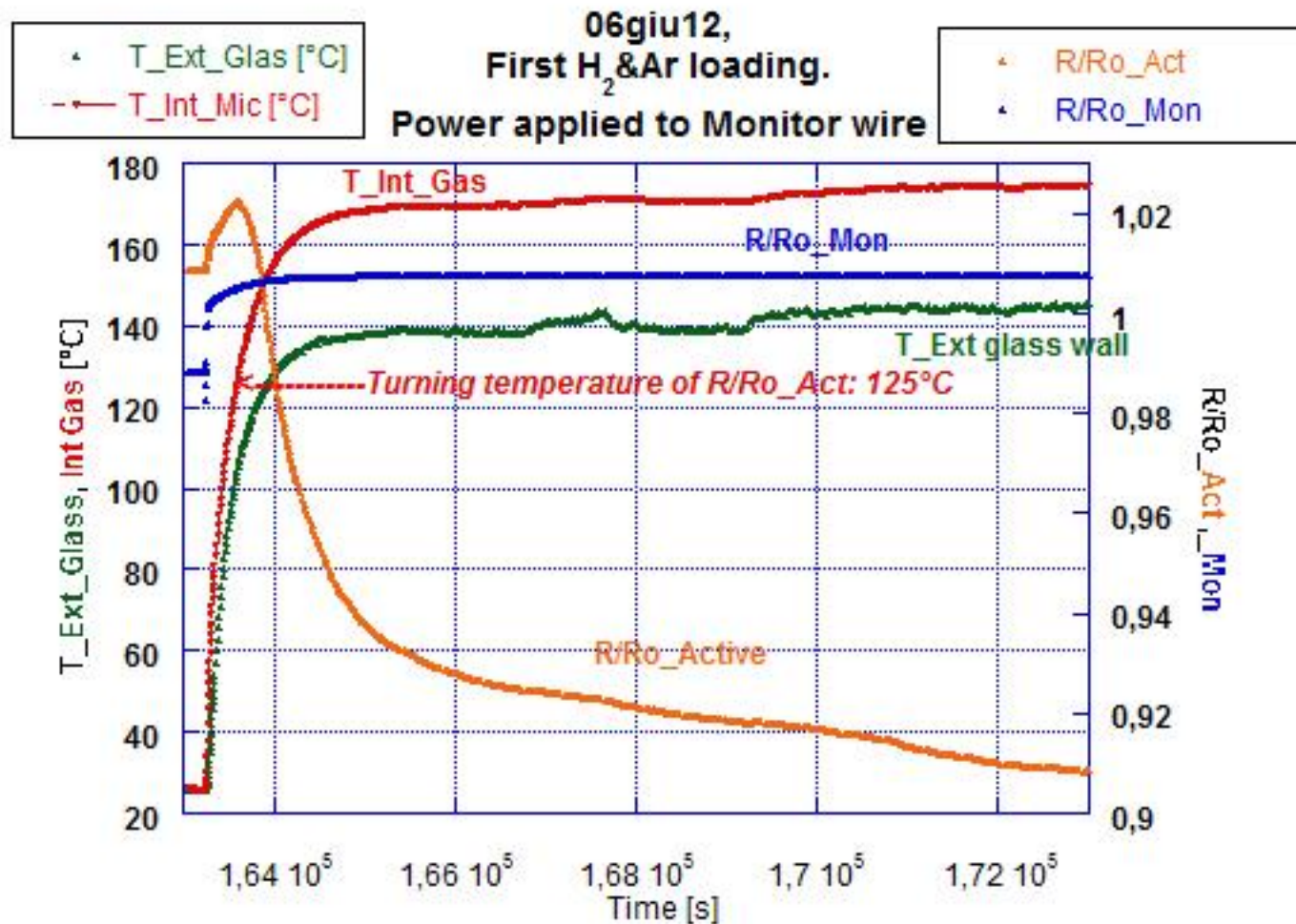


- Calibrations by Argon. Power, up to 48W, at Reference wire (Ni-Cr).
- Maximum power at sub-micrometric Constantan wire was limited to 30W: precaution against very deleterious sintering problems of nano-sized particles under pure noble gas.
- The R/Ro , of both wires, just slightly increased (as expected), increasing the temperatures.

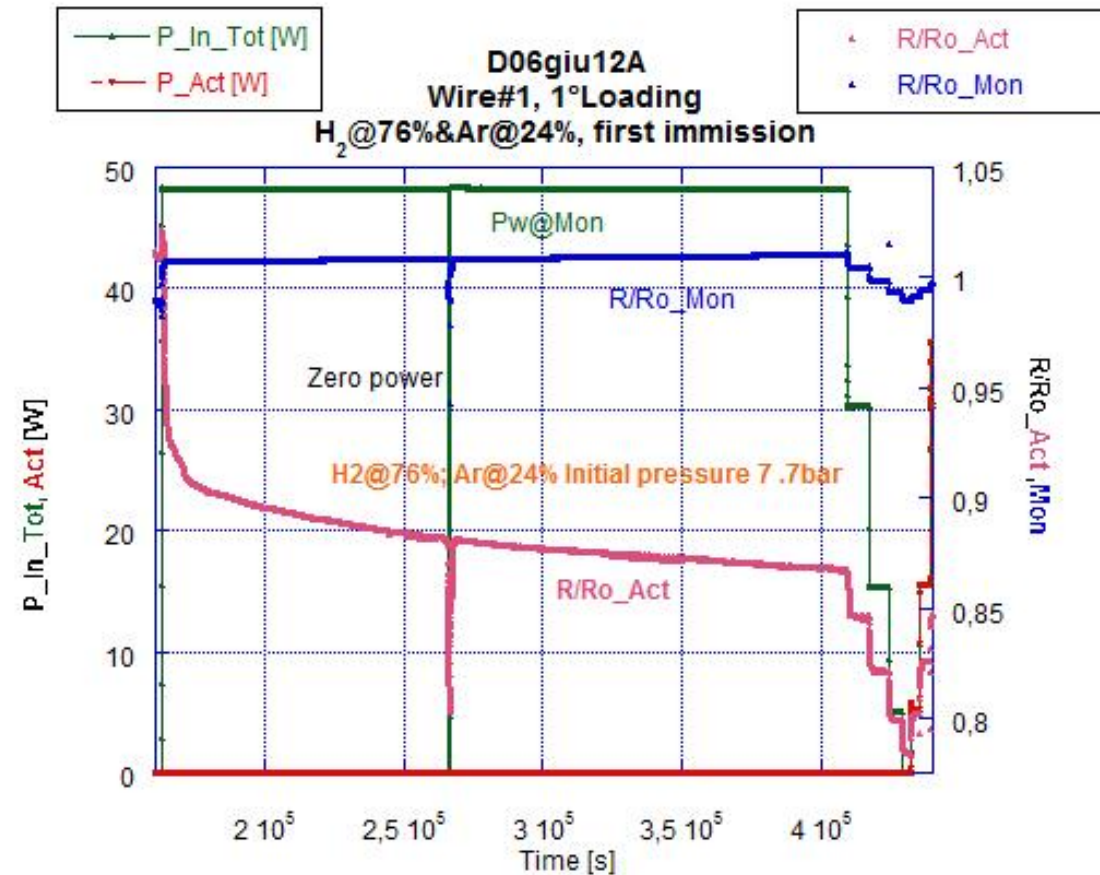
- 3) After adding a H_2/Ar mixture (about 75/25 ratio) at 7.7 bar of total pressure, and using as monitor parameter the R/R_o of both the active and inert wires, it was given power (48W) to the inert wire. It was found that when the temperature inside the reactor was larger than 125°C , the R/R_o value of active wire, after a very limited increase (to 1.02), dropped to 0.92 in 2500s. Later on, in about 100000 sec, decreased to 0.88.
- 4) *We observed a correlated increase of the “anomalous excess heat” (although quite unstable) with the R/R_o decreasing or better to say, “variation”.* The temperature inside the cell was about 180°C .



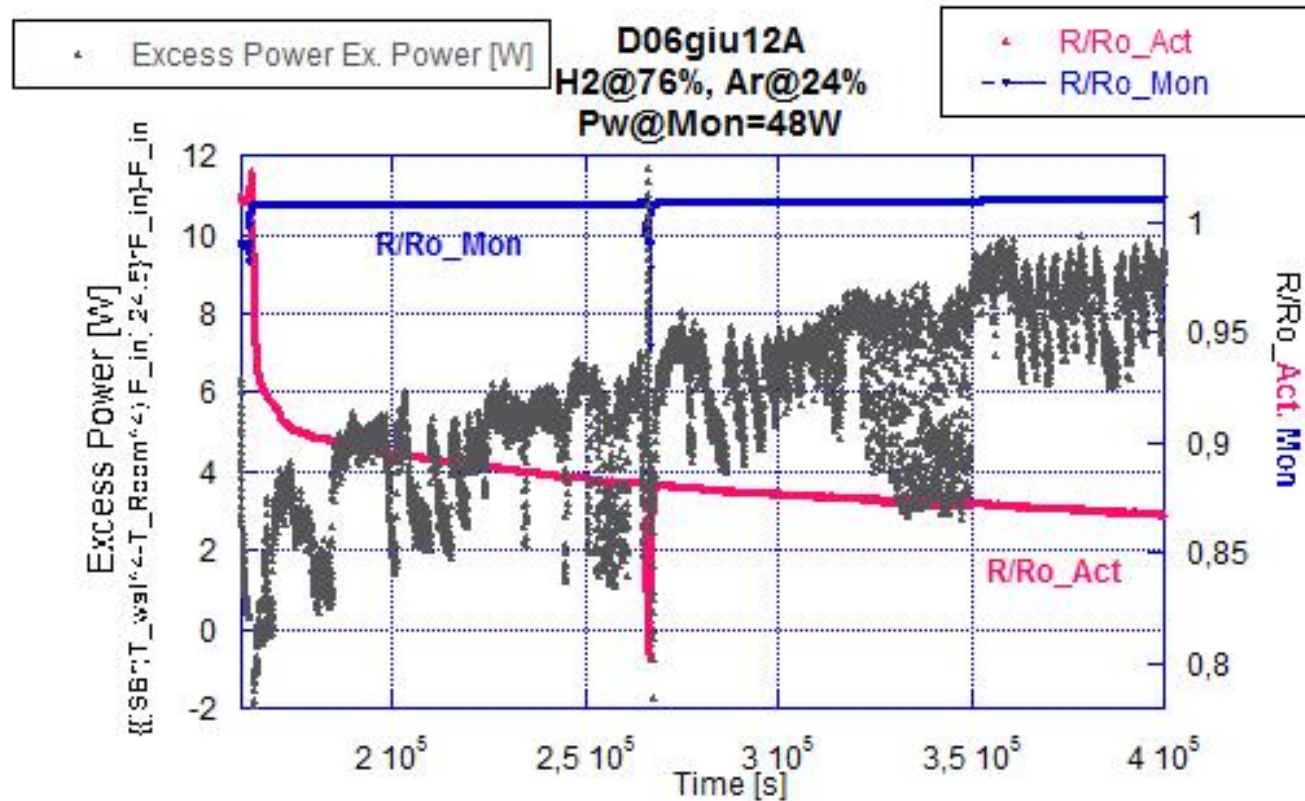
- First loading: H_2+Ar mixture, respectively at about 75% and 25% concentrations. Pressure 7.7 bar.
- Increasing gas temperature, the R/R_o ratio of Ni-Cr wire slightly increased, similarly to Ar experiments behaviour. The behaviour of **sub-micrometric Constantan** was fully **UNEXPECTED**: at the beginning increased (like Ar gas), later, **increasing the gas temperature (>125°C) and time**, **DECREASED** largely.



- Details of first loading by H₂-Ar mixture.
- The “**trigger**” **temperature**, to get the large resistance decrease of sub-micrometric Constantan wire, was about **125°C**. Temperature measured by a type K thermocouple (SS sealed) inside the gas cell.



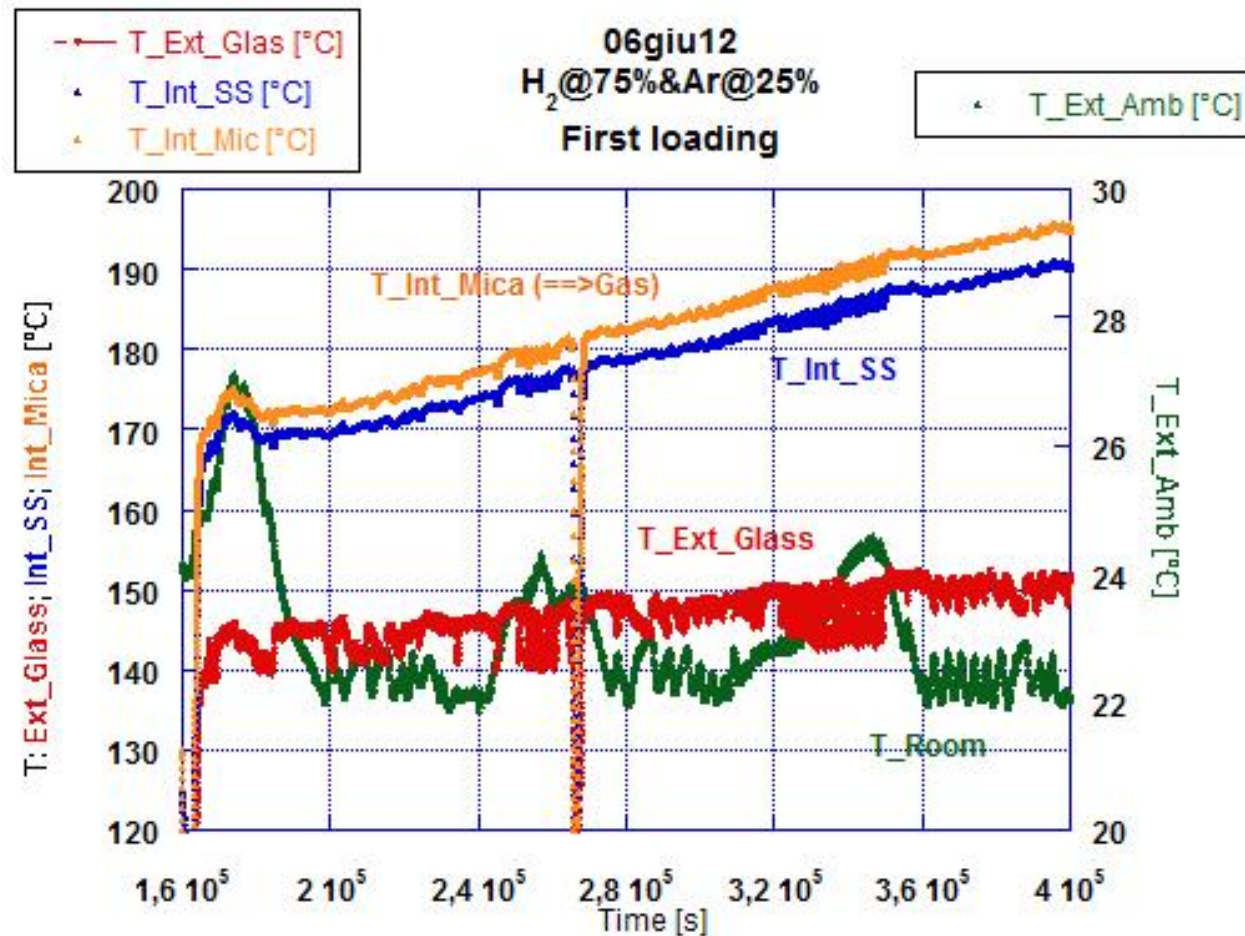
- Behaviour of R/Ro of both Mon. and Act. wires, up to times as long as 240ks from the beginning of first H_2 -Ar intake. The input power, obtained by Joule heating the Mon. wire, was kept almost constant (48W). After 240ks the R/Ro_{Act} reduced to **0.88** of its initial value. When the power was switched-off (i.e. $T_{gas}=25^\circ C$), at almost thermal equilibrium, the value of R/Ro_{Act} decreased to **0.784: it was still (slowly) decreasing over time.**



Behaviour of anomalous power generation, using indirect heating, i.e. power (48W) applied to Mon. wire .

Gas atmosphere was a mixture of H₂(75%) and Ar(25%) with a typical pressure of few bar (6-9). The anomalous power, quite irregular, showed a behaviour of increasing over time and SEEMED RELATED to the decreasing of R/Ro of sub-micro Constantan. It was computed using “self-calibration” of the apparatus: the “reference value” used was the value where the R/Ro was still decreasing and, anyway, the system had reached enough thermal equilibrium (as observed in previous experiments with Ar).

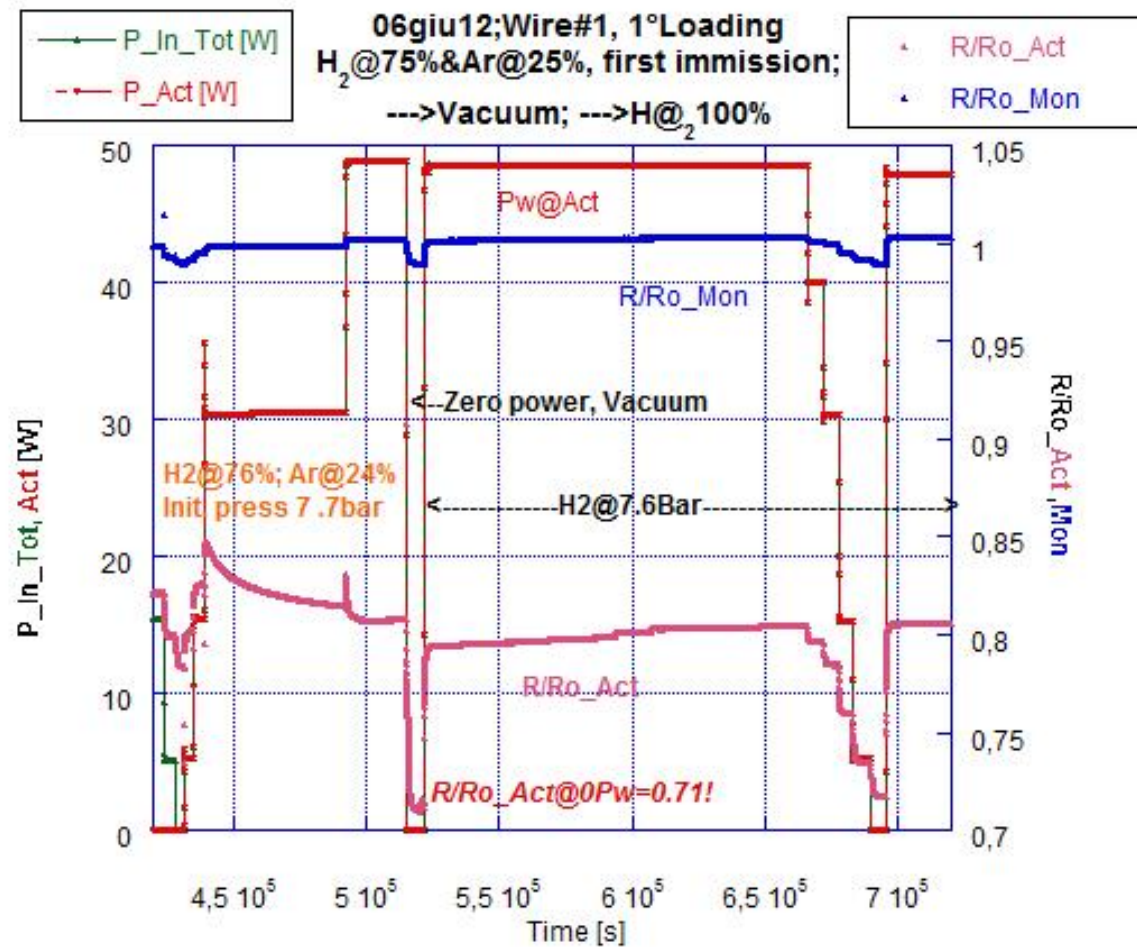
- 5) After 103ks from the beginning, we stopped the power to inert wire and allowed the reactor, and the wires, to cool to RT. The R/R_o value of active wire decreased to 0.80.
- 6) Just after that, we give the same previous power to inert wire and after others 150000s from the interruption we measured an R/R_o value of 0.867. The anomalous excess power increased further, in a way that, at a first observation, *depends mainly on the time lasted and not to the R/R_o value*. The instability of excess power, *if there weren't other uncontrolled parameters to fake it*, had values quite large and was correlated to the *small oscillations* (<1%) of R/R_o values.
- 7) We observed that the instabilities of room temperatures (usually 23-27°C) “helped”, in some aspects, the anomalous heat production, because, speculatively, introduced some non-equilibrium conditions. *In other words, in order to avoid misinterpretations of the results, after proper long times, the values of room temperature were the same as at the starting time while the anomalous heat increased over time.*



Behaviour of the temperatures of the reactor, at CONSTANT input power (48W at Mon. wire).

Flowing the time, **BOTH the external glass temperature (140→150°C) and the 2 internal (Gas, 170→195 °C; “Stainless Steel”, 168→190°C), increased. The effect was not directly related to room temperature variations, although such temperature variation helped to get not-stationary conditions.**

- 8) Among others, the positive effect, of long time lasting under Hydrogen gas at high temperature, was observed also by the A. Takahashi and A. Kitamura group and reported at the “X Pontignano Workshop” last April 2012. According to them, under their experimental conditions, constrains and materials, in 2 weeks of experiments the anomalous excess power slowly, and constantly, increased from 0 up to 3W.
- 9) We observed that the minimum cell temperature to stop the anomalous heat is around 120°C, i.e. similar to the first “loading” temperature (i.e. 125°C).
- 10) We observed a further increasing of anomalous power that, if there are no mistakes around, was about twice (i.e. absolute value of over 10W) of that detected when the power was applied to inert wire. The R/R₀ value, after initial increasing, stabilized to 0.808

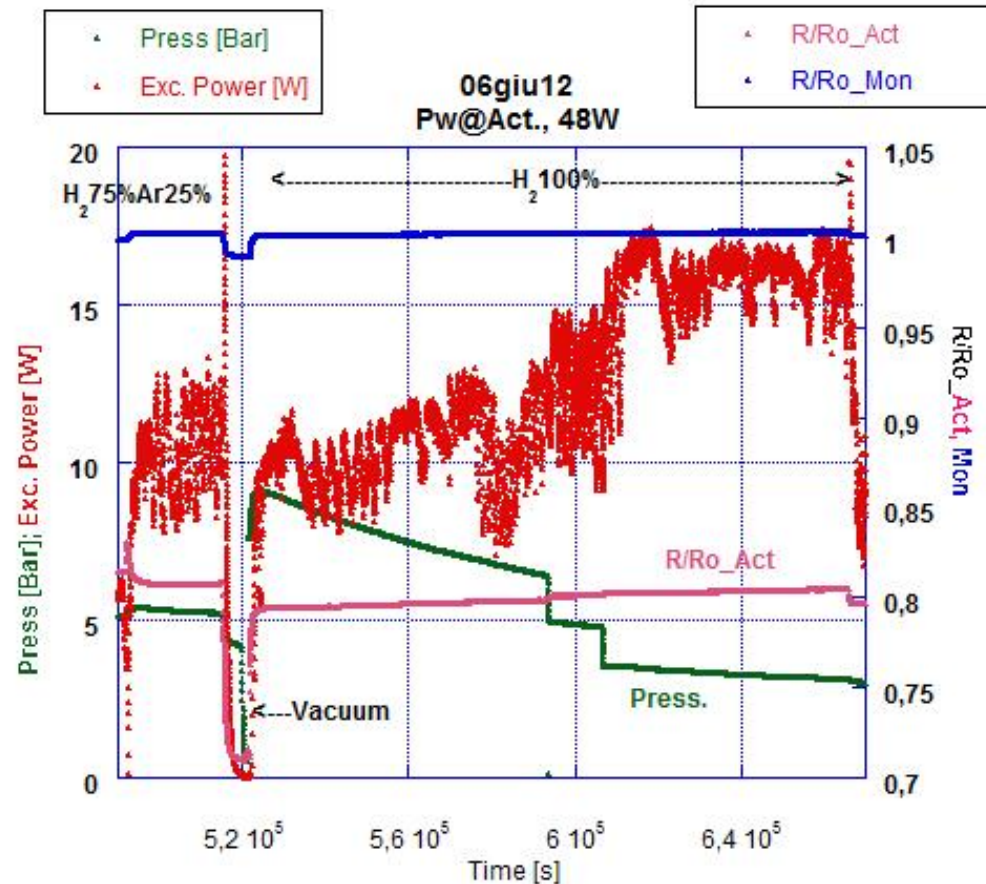


Behaviours of R/Ro changing: a) power applied from Mon. to Act. (time 420→520ks); b) Vacuum and no power (515→521ks); c) Gas changed from H₂&Ar to H₂ (at 100%) and power applied to Act.

After direct heating to Act., at the end of switch-off time (521ks), the R/Ro_Act dropped to only 0.71!

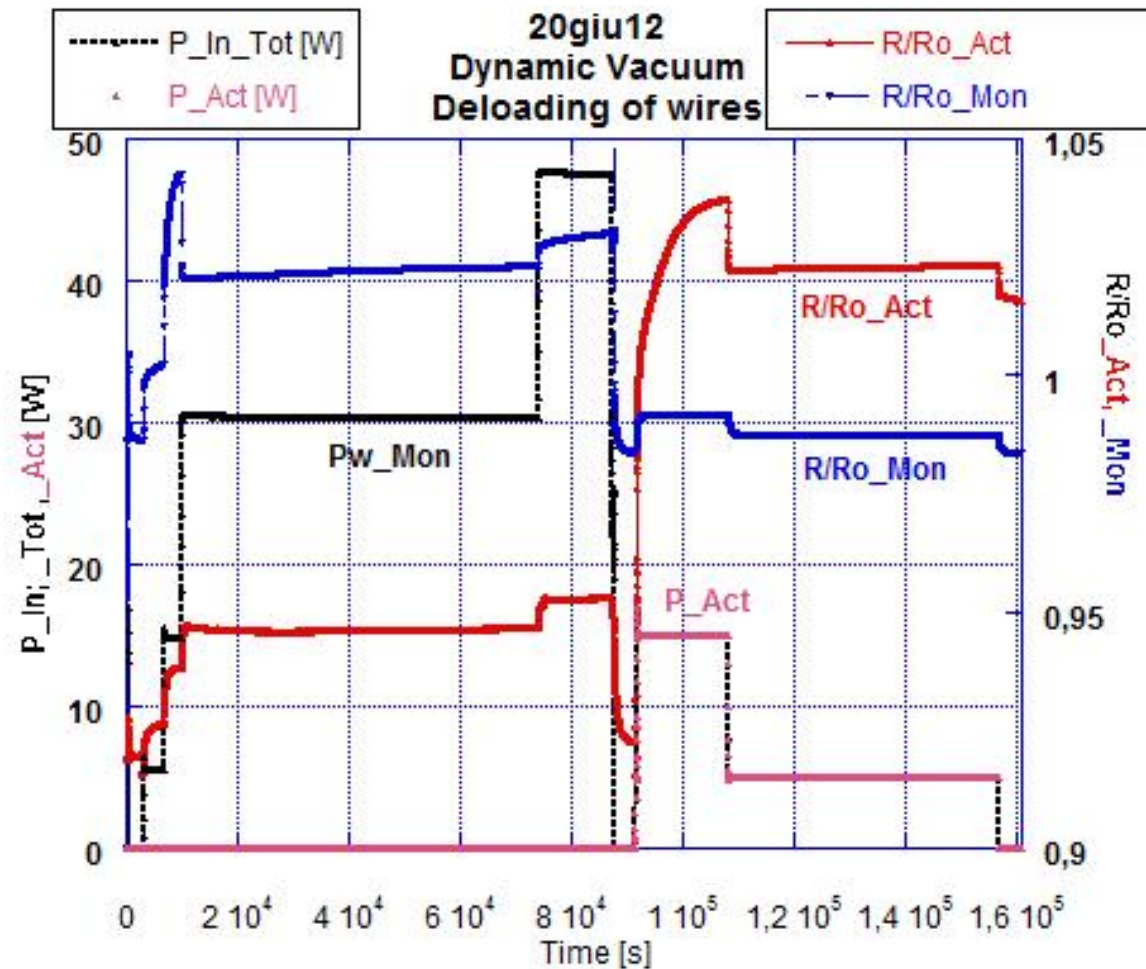
- 11) A possible explanation was that the local temperature of active wire, because Joule heating, was larger than that when the power was indirect. A very rough valuation of temperature is the range of 350-400°C, in respect to about 200°C with indirect heating.
- 12) If the consideration at point 11) is correct, we can think that the reaction, apart some temperature threshold, has a *positive feedback with increasing temperature*. A similar effect was found by: our self (with the old wire, and experiment, up to May 2012); A. Takahashi and A. Kitamura group with Ni-Cu-ZrO₂ powders. Among our previous experimental activity, on 2010, with a pure (50micron diameter) Ni wire, surface nano-coated (50 times) with several different elements, we measured the best excess power (26W) at wire temperatures as large as 900°C (under H₂/Ar mixture).
- 13) *After 360000s from the H₂/Ar gas intake, the power was switched off: the R/R₀, at RT, drop to 0.71. In other words, the direct heating (and related electro-migration phenomena) improved largely the loading, and then the anomalous power.*

- 14)After 410000s from first H₂/Ar intake, we made vacuum and added H₂ at 100% concentration.**
- 15)The results were similar to H₂/Ar gas and even better about anomalous heat production.**
- 16)We can't discriminate if the further improvements of performances were due to effects of pure H₂ or just time lasted under active gas.**



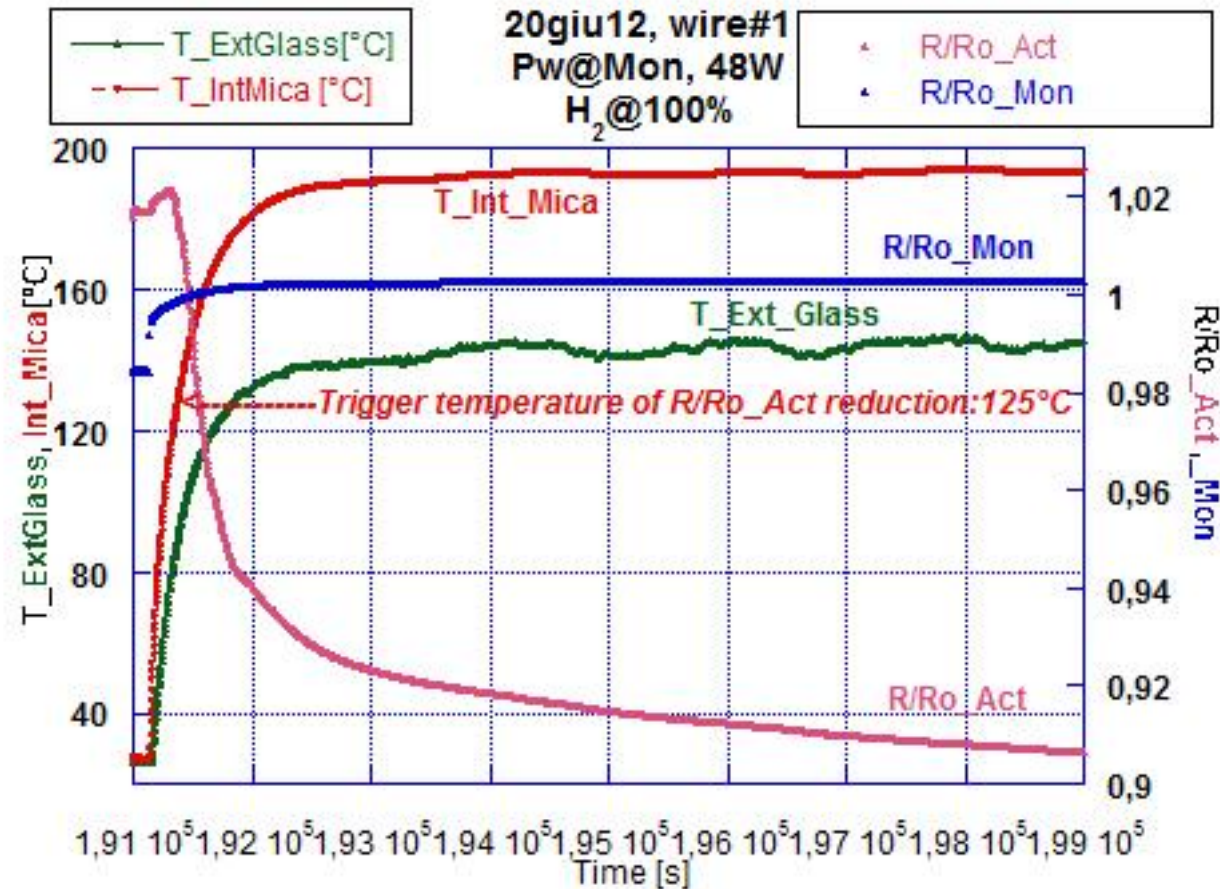
Experiment with power (48W) applied to Active wire. Up to time 518ks the gas mixture was H₂/Ar at 75/25 ratio. Later on, the power was reduced to zero and made vacuum: **R/Ro was as low as 0.71**. From time 522ks was added **pure H₂**: the excess power resumed the value before vacuum and, after controlled reduction of pressure, increased up to **16W**.

- 17) After another week of miscellaneous test, included the minimum pressure that the system can withstand (around 1 bar, to be further studied), we decided to de-load the wire from H_2 absorbed, to be sure that the resistance reduction observed was due to a real absorption and not to a variation of resistance due to the reduction of oxides (by H_2) at the nano-particles surfaces. To get de-loading we put the cell under dynamic vacuum and increased the temperatures.
- 18) After several hours, and not simple procedures, we get the original starting value of R/R_0 at 1: *the test was fully successful.* For a sake of comment, it was more easy, and fast (few minutes, later just wait) to load the H_2 inside the “good” nano-constantan than to remove it. Such evidence was founded by us since over five years, in gas, using thin *Pd wires covered with about 50 layers of multiple nano-materials*. Since that time, we named it “*diode effect*” because it was more easy to absorb D_2 than desorb. We recall that the “*diode effect*” was detected even in $Pd-D_2O$ *electrolytic experiments* (since 1998) and discussed the effect in details during several ICCF Conferences.



First de-loading of Act. wire #1. The full de-loading ($R/Ro=1.016$), under dynamic vacuum, was achieved fully only after quite large electro-migration current (about 920mA) at Act.

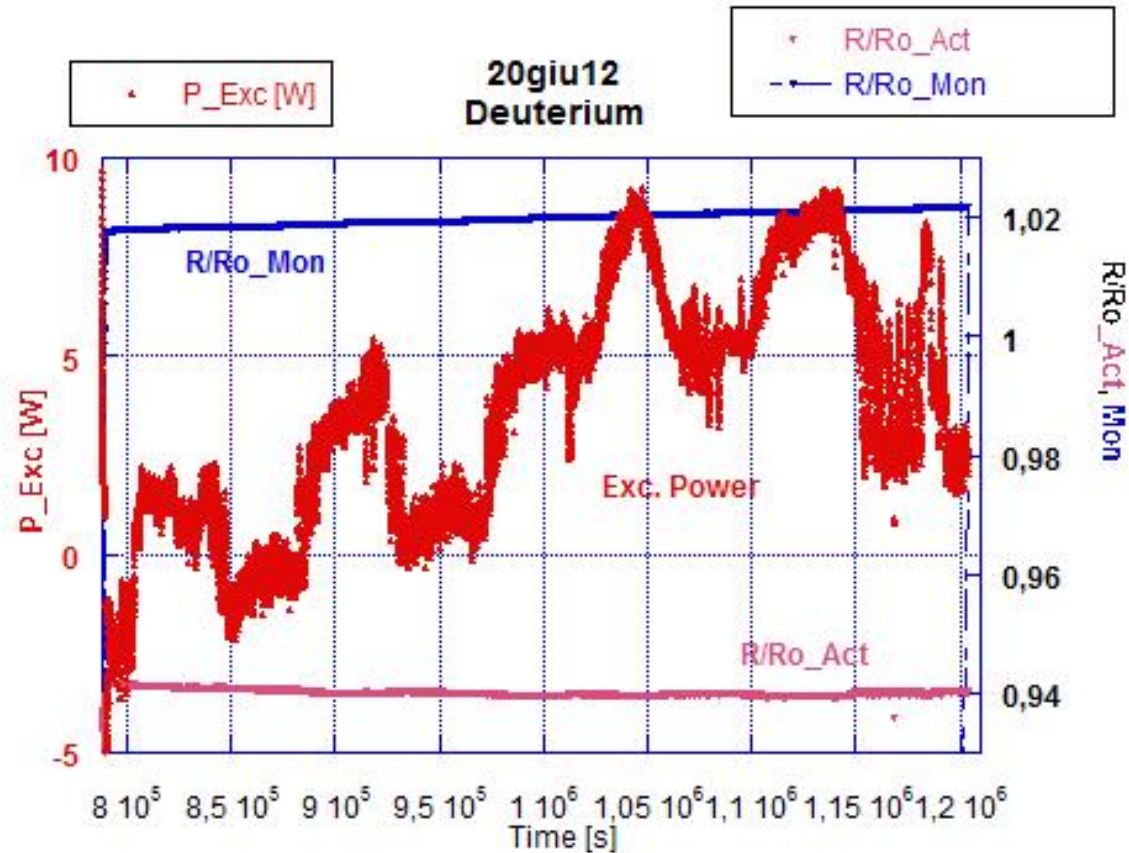
- 19) We reloaded again the wire and get behaviour of R/R_0 decreasing and anomalous heat not too different from the first cycle, although at lower intensity.**



Wire#1. First re-loading of H₂, after forced (electro-migration) de-loading under vacuum. The behaviour of re-loading was similar to first loading, even the trigger temperature (gas cell temperature) remained unchanged (i.e. 125°C).

- 20) Again we de-loaded the wire from H_2 to make experiments with Deuterium gas. This time the final value of R/R_0 was 0.93 and not 1, as expected. We supposed that some H_2 was stored, “in deep position” some-where in the lattice.
- 21) After D_2 intake, we increased, as usual, the temperature by power to the inert wire. The absorption was really of small amount.
- 22) We observed, for the first time in our experimentation with such kind of materials, some X (and/or gamma emission), coming-out from the reactor during the increasing of the temperature from about $100^\circ C$ to $160^\circ C$. We used a NaI(Tl) detector, energy range 25-2000keV used as counter (safety purposes), not as spectrometer. Total time of such emission was about 600s and clearly detectable, burst like.

- 23) About thermal anomalies, we observed, very surprising, that the response was endothermic, not eso-thermic. The second day the system crossed the zero line and later become clearly eso-thermic. Similar effects were reported also by A. Takahashi and A. Kitamura.**
- 24) After about 350000s from the beginning of D₂ intake the temperature abruptly increased and the wire was broken. We observed that the pressure decreased, because some problems to the reactor gas tight, but at times of 80000s before. The SEM observations showed fusion of a large piece of wire. The shape was like a ball. Further analyses are in progress.**



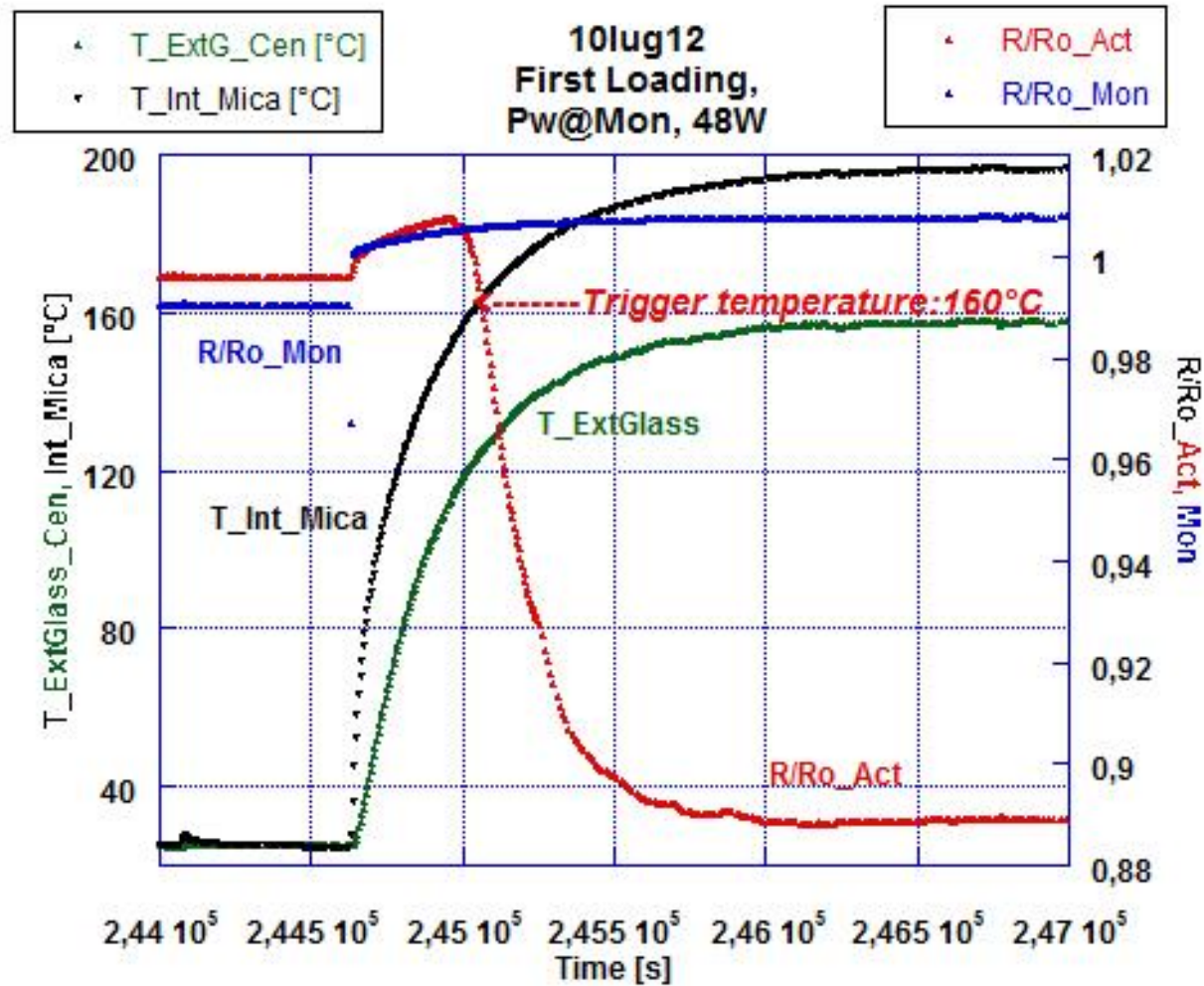
Measurement with **Deuterium** gas. At the beginning, up to 1 day long, the reaction was **ENDOTHERMIC**; later crossed the zero line and began to be eso-thermic, as usual with Hydrogen.

25) Starting from July 10, 2012, we used the second wire (#2), stored in a plastic bag.

26) In the meanwhile, we improved the overall detection of external temperatures and added 3 other thermometers for cross-control purposes. The main thermometer was moved from the original position, close to the end of the wire, to the center.

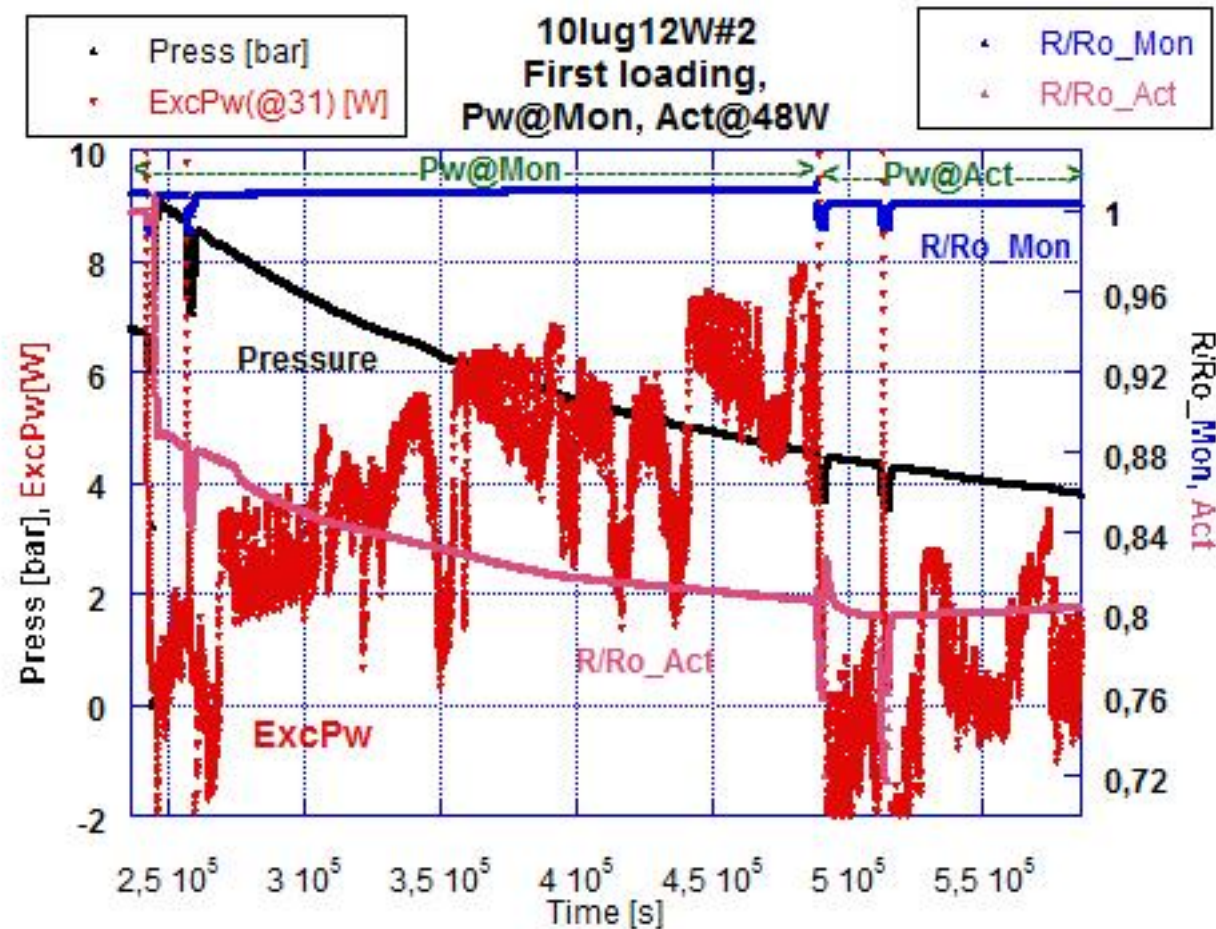
27) The results were qualitatively similar to the first wire, although at lower intensity.

The starting temperature of loading, from the value of 125°C of the wire #1, increased to about 160°C. In particular, the wire was not able to withstand direct heating conditions. We think that the surface was partially obstructed from something (HDPE plastic decomposed by catalytic surface?).

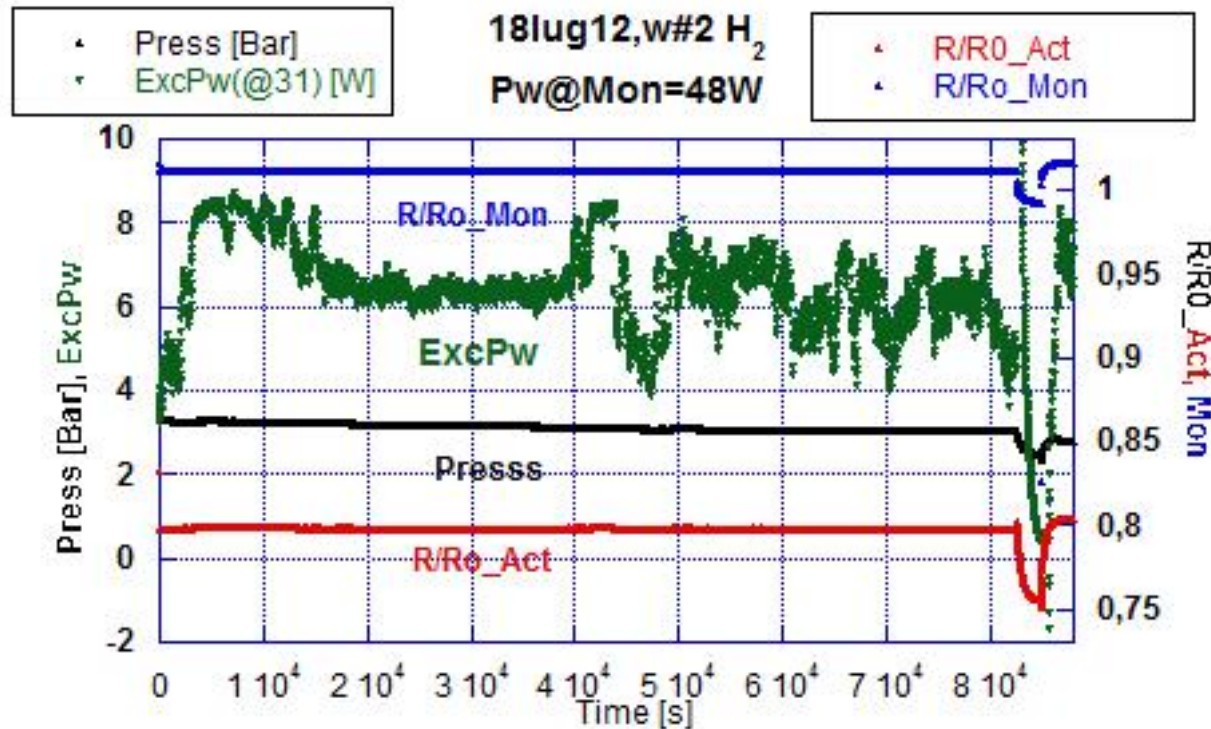


First loading of wire #2, prepared on some day of #1 and kept in plastic bag for 35 days.

The temperature of “triggering” for loading Act. was larger (160°C) in respect to #1 (125°C).

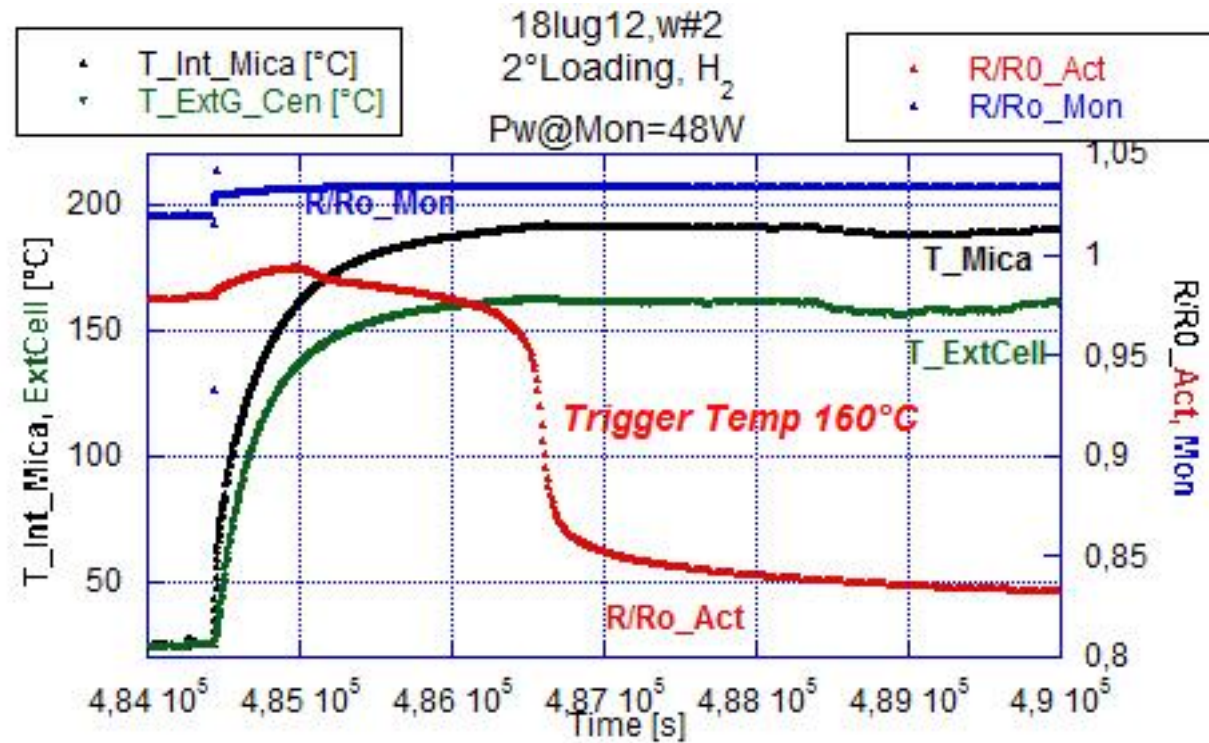


Behaviour of R/Ro_Act and Mon. About anomalous heat, the wire #2 had an overall behaviour similar, *but lower*, to #1 under Indirect heating condition (i.e. power at Mon.). Despite of wire#1, the anomalous power dropped close to zero with power was applied to Act. wire: surface damaged by plastic? *After 4 weeks the wire recovered the performances: anomalous power larger at Act. wire.*



Another example of anomalous heat behaviour on wire#2. This time, after a starting value “interesting”, i.e. about 7-8W, the trend was toward a reduction over time, opposite to the previous situations. It is important to note, for scientific understanding, the although the pressure gradually reduced, the reduction wasn’t enough to compensate for the excess power production. Moreover, the small **“dynamic” variation of R/Ro_Act, like oscillation, was strongly reduced**. Could be it the co-origin of anomalous power?

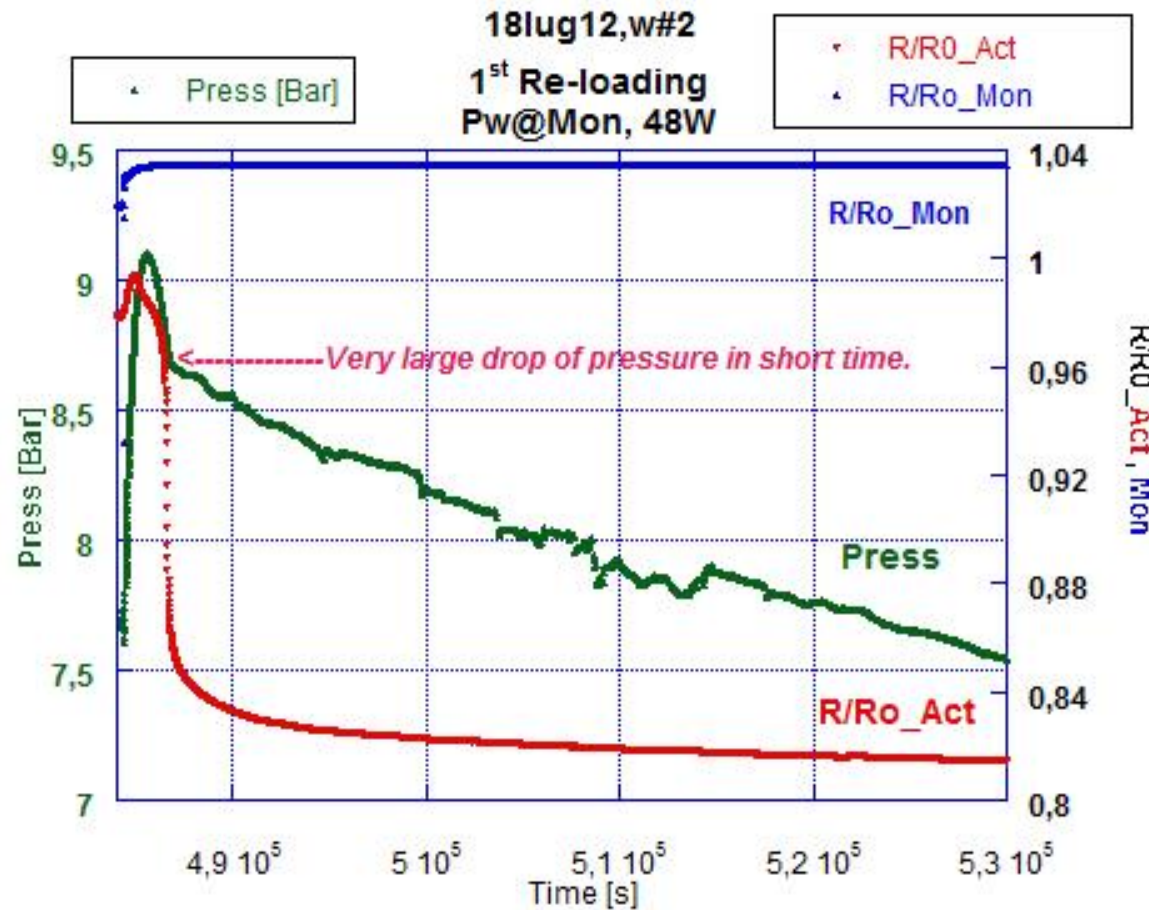
- 28) On July 23 we made de-loading by: dynamic vacuum conditions, 220°C internal reactor temperature, power at Ni-Cr, 50000s duration. On July 24 we made re-loading.
- 29) After reloading, the results seemed improved about: speed of loading (time of the drop of R/R_0 from 1 to 0.85 of only 2000s), time necessary to get anomalous heat (less than 6 hours). Anyway, the stability worsened.
- 30) Because the re-loading speed was extremely large, we were able to evaluate the **H/Constantan ratio, atom/atom**, by pressure drop inside the reactor. Results of calculations give a value >0.5 supposing all the whole wire involved and **>1** supposing only the skeleton section. The calculation was limited to the first 2000 seconds. Obviously the final values, at longer times, are larger but very difficult to evaluate.



Behaviour of first re-loading of wire#2.

The reloading speed was much faster in respect to first loading.

The “trigger” temperature of loading was almost the same as in the first loading, i.e. about 160°C.



Behaviour of pressure versus R/Ro_Act fast reduction. It was possible to evaluate the $H/Constantan$ ratio, considering just the first 2000s of the effect. The value was >0.5 if we consider the whole wire (300mg) and >1 if we consider only the volume of the “skeleton” skin.

- 31) After the re-loading the behaviour of anomalous power recovered but, overall, was quite unstable and with tendency to decreasing.
- 32) After dismantelling the apparatus (and wire still operating) for the expedition to USA (planned National Instruments public demo, Austin-Tx, 5-8 August 2012), the reactor wires were forced to over 1 week of free air condition. At the restart of the operations, we realised the *R/Ro_Act ratio remained almost unchanged* (about 0.8) and, for reasons to be deeply investigated, it *improved the performances about anomalous heat production*, even in the condition that previously was deleterious, i.e. direct heating to active wire.
- 33) We hope that also at the ICCF17 demo the overall behaviours will be satisfactory from the point of view of scientific understanding.

CONCLUSIONS

- 1) If there will be no errors in the measurements performed and procedure adopted, it seems that the commercial Constantan alloy, with the surface deeply modified about geometry (i.e. skeleton type) and dimensionality of 20-100nm, multy-layers, is a good candidate for anomalous heat prodution because: a) intrinsic low cost of raw materials; b) simple procedures (i.e. low-cost) of nano-structures growing, as recently developed by our group at INFN-LNF-Italy (in close collaboration with private Company); c) use of Hydrogen.
- 2) The numbers of open questions are still very large, among others the “strange” behaviour using Deuterium gas.

3) Several of the results found were similar to what detected by the Japan group (A. Takahashi, A. Kitamura) in collaboration with Technova (side of Toyota Company), using Ni-Cu alloy dispersed in Zirconia matrix.

4) The next step will be the use of **quartz tube** instead of borosilicate, at the moment in use. The quartz will allow to studies temperatures over 300°C, at the moment not allowed by borosilicate (1st softening temperature of borosilicate glass is about 270°C).

5) We observed (experiment ended at May 2012) that such materials have a behaviour of “positive feedback” of anomalous power in respect to temperature increasing.

6) If point 5) will be reconfirmed with the wire made by new procedures, it could be possible to reach “regions” of operations were even the **self-sustaining** regime could be observed.

- 7) *More, and systematic work, is necessary to elucidate the several open questions, first of all the stability over time of the anomalous heat generation and its reproducibility-safety.*
- 8) *The collaboration of all the Community involved in the LENR studies is welcomed.*
- 9) *As large advantage, we realised that most of the phenomenology previously observed in Pd-Deuterium system is present also in sub-microstructured Costantan-Hydrogen.*