

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

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Abstract-- Starting in February 2011, we studied the feasibility of new Nickel based alloys that are able to absorb proper amounts of Hydrogen (H₂) and/or Deuterium (D₂) and that have, in principle, some possibility to generate anomalous thermal effects at temperatures >100°C.

The interest in Ni comes in part because there is the possibility to use also H₂ instead of expensive D₂. Moreover, cross-comparison of results using H₂ instead of D₂ can be made and could help the understanding of the phenomena involved (nuclear origin?) because use of such isotopes.

Index Terms-- Calorimeter, LENR, Nickel based alloys, sub-micrometric surfaces.

I. 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 [1], 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 factors was, according to use, the ability to dissociate H₂. 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 dissociation. 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 dissociation values remain almost constant (from 3.16 to 2.86eV).

We focused on a commercial (low cost) material, called ISOTAN44, nominal atomic composition Cu₅₅Ni₄₄Mn₁, developed several years ago by Isabellenhutte Heusler, GmbH, KG-Germany.

The ISOTAN 44 was selected according to the following, overall, considerations, as pointed out by us since April 2012, Ref. [2].

A. Measurable diffusion coefficient of Hydrogen, in even the pure (not alloyed) elements, i.e. Cu and Ni, at high temperatures: Cu=10⁻⁶cm²/s at 200°C, 10⁻⁴cm²/s at 700°C; Ni= 10⁻⁷cm²/s at 200°C, 10⁻⁶cm²/s at 350°C. In comparison, the (good) values for Pd are: 10⁻⁵cm²/s at 200°C, 10⁻⁴cm²/s at 420°C; at 600°C were reported values as large as 8*10⁻³cm²/s, but not reproducible. We

think that the “flux” of H₂ or D₂, inside lattices is one of the is one of key factor to generate anomalous effects.

B. Lower cost, overall, even considering the procedure to “build” nano-structure at the surface, in respect to Pd, very expensive precious metal.

C. Very good mechanical properties in respect to aging effects due to cycles of both low->high->low temperatures and H₂ absorption-desorption: the sample of our (“generation one”) long time lasting experiment was working for over 7 months; only after such long time of operations, we observed serious damages rising-up. Our results are, in some aspects, different from that obtained by A.W. Szafranski [Ref. 3]: he observed extreme brittleness in, as received, Cu-Ni alloy that was only cold rolled from 200µm to 20µm (the penetration depth of H into Ni is about 30µm) and then cycled between 77K and 300K under 1GPa pressure of H₂. We could think, only, that high temperatures and/or Mn (at 1%) addition have beneficial effects on reducing brittleness problems. Moreover, we never made experiments at 77K.

D. Extremely large values of (computed) catalytic power (ΔE) in respect to the dissociation of H₂, Ref. [1], as following:

Ni _{0.3750} -Cu _{0.6250}	==> +3.16eV
Ni _{0.6250} -Cu _{0.3750}	==> +2.86eV
Ni _{0.8125} -Cu _{0.1875}	==> +2.10eV
Ni	==> +1.74eV
Ni _{0.1825} -Cu _{0.8175}	==> +1.57eV
Ag _{0.8125} -Pd _{0.1875}	==> +0.57eV
Ag _{0.625} -Pd _{0.375}	==> +0.51eV
Ag _{0.1875} -Pd _{0.8125}	==> +0.51eV
Pd	==> +0.42eV
Cu	==> -1.11eV
Ag	==> -1.42eV

E. The possibility, at least in principle, to produce nano-micro structures (and obviously voids) both at the surface and 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 CuOx and cooling rate are key aspects of the preparation to be

studied in deeper details, although we spent a lot of time (and money) to investigate such key aspects.

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.

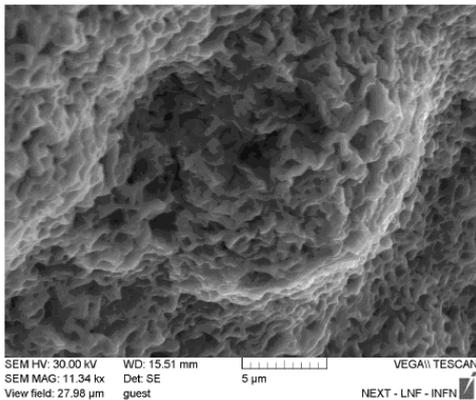
II. SAMPLES PREPARATION (PROCEDURES USED FOR THE EXPERIMENT UP TO MAY 2012, “GENERATION ONE”). SIMILAR COMPOSITION MATERIALS, TRUE NANOMETRIC, DEVELOPED INDEPENDENTLY IN JAPAN

In our exploratory preparations/tests we used “standardized” wires: (“naked”) $\Phi=200\mu\text{m}$, $l=105\text{cm}$. 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 H_2 atmosphere, we NEVER got any type of anomalies (like changing of resistance) on wires with applied temperatures as large as 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 enamel protection (enamel completely removed by burning up to 600°C in air) and stress released;
3. Acid etching of wire after burning at 600°C .

The wires, point 2), at the beginning, were just “cleaned-up” of the original “solderable” (type V) enamel insulating layer (as provided by Isabellenhutte) by Joule heating, in air, at current as large as 2000mA, time 5m.



SEM. Wire's surface after heat treatments at $I=2500\text{mA}$, 5m.

Figure 1. Wire surface after enhanced heat treatments, *generation one* experiments.

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 $<1\text{s}$) had dramatic effects on the growing of nano-microstructures and their dimensions (see Fig. 1, as example). The role of O_2 , 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 points of pure Cu is 1083°C , of constantan about 1200°C in inert gas).

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 (i.e. the core), 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 H_2 and D_2 atmosphere using a very accurate flow calorimeter (indetermination $<2\%$). The total times of experiments were really long (over 10 months) and only at the end the damages were so heavy to prevent further reliable interpretation of the experimental results. They were discussed, very deeply, during the “X International Workshop on Anomalies...”, on last April 2012 [Ref. 2].

We were very happy 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 true nanometric size, i.e. 5-20nm) dispersed in an inert matrix of ZrO_2 . Such work was performed among collaboration with the Research Group of a Toyota Company (Technova). We got some information, by A. Takahashi and A. Kitamura, since January 2012, about promising results by a specific $(\text{Ni}_{85}\text{-Cu}_{15})_{35\%}\text{-(ZrO}_2)_{65\%}$ alloy [Ref. 4].

We recall that such material is a further development of the nanomaterial $\text{Pd}_{35\%}\text{-ZrO}_2_{65\%}$ developed by Yoshiaki Arata (Osaka Univ., Japan) since 2005.

The “short information” about Ni-Cu- ZrO_2 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,...) connected to United Nations at Geneva. Obviously, I requested that everybody involved in LENR studies, worldwide, communicate the most recent and interesting results to include in my Review. A similar talk, with even more technical/scientific details, was given even at CERN (Geneva) on March 22, 2012 in the framework of the (prestigious) CERN Colloquium. Under my specific request, it was added also a talk (by Y. Srivastava, Univ. Perugia, Italy) related to overview of theories in LENR.

The overall behaviours of Ni-Cu alloys although at different ratios of two main elements, in respect to H_2 and D_2 absorption, and the amount of anomalous heat

detected, were, in several aspects, similar in the experiments performed both in Japan and Italy.

Such kind of evidence reinforced our intention to develop a better material (from the point of view of nano-dimensionality), keeping the starting Ni-Cu composition “constant”. In other words, our efforts were devoted to improve the amount of active material at low dimensions (<100nm) and, at the same time, avoid the adverse effect of “leakage” of the smallest particle from the surface, e.g. under dynamic vacuum conditions.

III NEW TRANSPARENT, DISSIPATION-TYPE, “CALORIMETER”

By 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 (almost no leakage of the “best” material from the surface), fraction of material at small dimensions. Such last parameter increased from previous 1-2% (*generation one*) up to about 30% of the whole material (*generation two*).

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 build, specific electronics and mechanical set-up to produce such kind of sub-micrometric wires. Systematic (but very boring and expensive) experimental work was the key factor for the success.

Moreover, because one of our goals was to see, to the naked eye, if the wire was really stable about the leakage of “good” materials even after several cycles of low→high→low temperatures and H₂ loading (or even de-loading!), we build a new transparent reactor with borosilicate glass (Schott DURAN) of large (3mm) wall thickness to withstand enough large pressure drops (up to 8 bar), at internal wall temperatures up to 280°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_{wall}>140°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:

$$P_{out} = 5.67 * 10^{-8} (T_{wall}^4 - T_{room}^4) \left[\frac{W}{m^2} \right] \quad (1)$$

In such formula the temperatures T are in K. Calibrations were made using our usual procedure to add an inert wire, very close to the “active” one, and make several measurements with inert gases. 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.

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 sort of simplified calibration curve just dividing the formula (1) by the input power. Obviously, we neglected the contribution to heat dissipations of free air convection (usual values are 5-25W*m²*K, in “normal environments” are 12-15W). Finally, we just recall that in the temperature range of our interest (internal cell 250-350°C), the thick borosilicate glass behaves like a black-body for the wavelengths of interest (>2.5µm). Moreover, the effects of pressure variations *inside* the reactor chamber, with related temperatures variation due to different convection values (i.e. the internal temperature increases versus pressure decreasing), have values of temperature changing that can be neglected at the external wall, for the purposes of our experimentations. Anyway, specific test were performed, in He, varying the pressure between 6.5 and 2.5bar. Such tests were made at the beginning of the experiment (wire new, reactor glass wall “clean”) and after some months (wire used, glass wall lightly “dirty”).

IV RESULTS WITH THE NEW WIRES (*generation 2*).

At the end of May, 2012 two wires were produced, both with the same new procedures (*generation two*).

The first 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 called 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: of the order of 700. The thicknesses, of each multi-layers, were in the range of 20-100nm. The mechanical stability, against leakage of sub-micrometric materials, was largely improved.

The primary 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_o. R_o 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, in situ (I-V methods), a value of resistance of 16.9684 Ohm and 57.4394 Ohm, respectively for sub-micro_Costantan and Ni-Cr (supposed inert) wires. The measuring currents were just 4mA, to avoid 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 180-250°C, depending on the gas composition and its pressure. 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 by (1).

3.) After adding a H₂/Ar mixture (75/25 ratio) at 7 bar of total pressure, and using as monitor parameter the resistance of both the active and inert wires, it was given power (48W) to the inert wire. It was found (Fig.2) that when the temperature inside the reactor was larger than 125°C, the resistance ratio of active wire, after a very limited increase (to 1.02), dropped to 0.92 in 2500s. Later on, in about 100000 sec, the R/Ro decreased to 0.88. We observed a correlated increase of the “anomalous excess heat” (although quite unstable) with the R/Ro decreasing. The temperature inside cell was about 180°C.

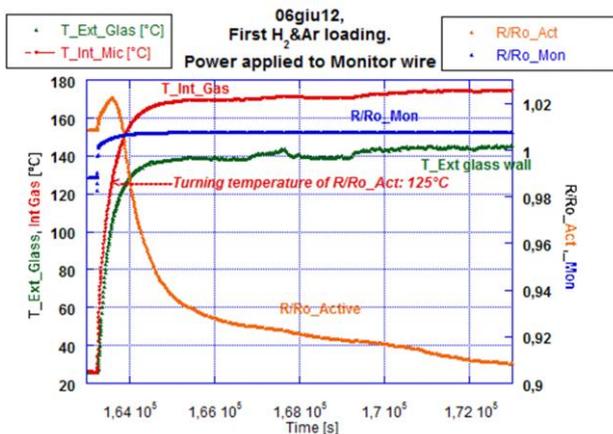


Figure 2. Details of first loading by H₂-Ar mixture

4.) After 103000 sec from the beginning (Fig. 3), we stopped the power to the inert wire and allowed the reactor, and the wires, to cool to RT. The R/Ro value of active wire decreased to 0.80.

5.) Just after that, we give the same previous power to inert wire and after others 150000s from the interruption we measured an R/Ro value of 0.867. The anomalous excess power increased further (Fig. 3), in a way that, at a first observation, depends mainly on the time lasted and not to the R/Ro value (low decrease). 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/Ro values.

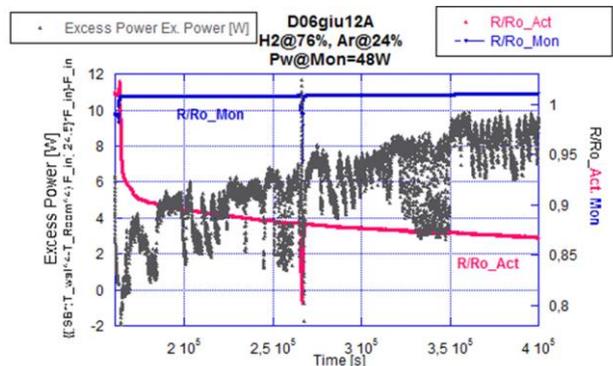


Figure 3. Behaviour of anomalous power generation, using indirect heating, i.e. power (48W) applied to Mon. wire.

6.) We observed that even 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 at the starting while the anomalous heat increased over time.

7.) Among others, the positive effect, of long time lasting under H₂ gas, was observed also by the A. Takahashi and A. Kitamura group (reported both at the X Pontignano Workshop and ICCF17). According to them, under their experimental conditions, constrains and materials, in 2 weeks of experiments the anomalous excess power slowly increased from 0 up to 3W.

8.) 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).

9.) After 330000 sec from the first H₂/Ar intake, the power was given to active wire (Fig. 4).

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/Ro value, after initial increasing, stabilized to 0.808.

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. Similar effects were 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.

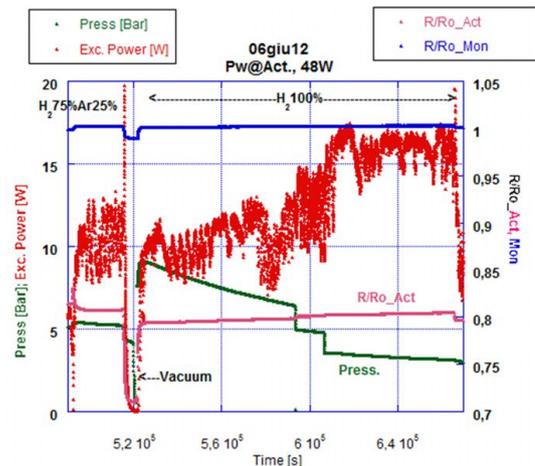


Figure 4. Experiment with power (48W) applied to Active wire. Up to time 518ks the gas mixture was H₂/Ar at 75/25 ratio. Later on, the power was reduced to zero and made vacuum: R/Ro was as low as 0.71. From time 522ks was added pure H₂: the excess power resumed the value before vacuum and, after controlled reduction of pressure, increased up to 16W.

13.) After 360000s from the H₂/Ar gas intake (Fig. 4), i.e. time 515ks, the power was switched off: the R/Ro, at

RT, dropped to 0.71. In other words, the direct heating (electro-migration phenomena and/or large temperatures) 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 (increased embrittlement).

17.) After another week of miscellaneous test, we decided to de-load the wire from H₂ 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 H₂) at the nano-particles surfaces.

18.) To get de-loading we put the cell under dynamic vacuum and increased the temperatures.

19.) After several hours, we get the original starting value of R/Ro at 1: the test was fully successful.

20.) We reloaded again the wire and get behaviour of R/Ro decreasing and anomalous heat not too different from the first cycle.

21.) Again we de-loaded the wire from H₂ to make experiments with D₂ gas (Fig. 5). This time the final value of R/Ro was 0.93, not 1 as expected. We supposed that some H₂ was stored some-where in the lattice.

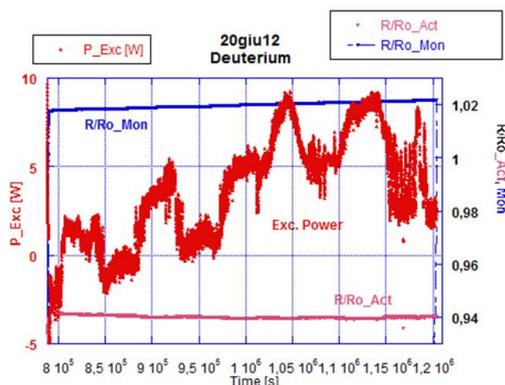


Figure 5. Experiments under D₂ gas. At the beginning, up to 1 day long, the reaction was ENDOTHERMIC; later crossed the zero line and began to be exothermic, as usual with H₂...

22.) After D₂ intake, we increased, as usual, the temperature by power to the inert wire. The absorption was really of small amount.

23.) We observed, for the first time in our experimentation with such kind of materials, some X (and/or gamma) emissions, coming-out from the reactor during the increasing of the temperature from about 100°C to 160°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.

24.) About thermal anomalies, we observed, very surprising, that the response was endothermic, not exothermic. The second day the system crossed the zero line and later become clearly exothermic. Similar effects were reported also by A. Takahashi and A. Kitamura.

25.) 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.

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

27.) In the meanwhile, we improved the overall detection of external temperatures and added 3 other thermometers. The main thermometer was moved from the original position, little bit close to one end of the wire, to exactly at the centre of the area of glass tube where are located (in the inner) the wires.

28.) 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?).

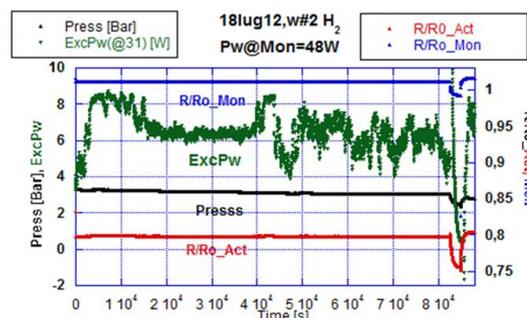


Figure 6. An example of anomalous heat on wire#2

29.) On July 23 we made de-loading and on July 24 we made loading again. The sequences were: a) dynamic vacuum conditions, 220°C internal reactor temperature, power at Ni-Cr, 50000s duration; b) H₂ filling.

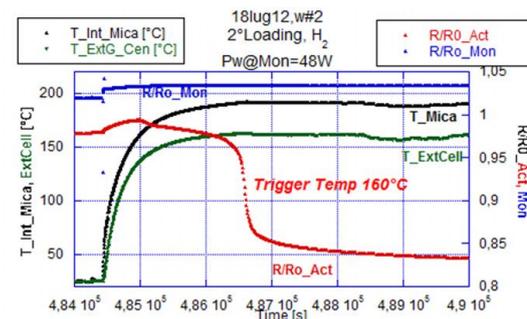


Figure 7. Behaviour of first re-loading of wire#2

30.) The results, Fig.7, seemed largely improved about: speed of loading (time of the drop of R/Ro from 1 to 0.85 of only 2000s), time necessary to get measurable anomalous heat (less than 6 hours).

31.) The experiment had been stopped on July 28 to package and "shipping" the reactor to USA (National Instruments Meeting at Austin-Texas) and later-on to Korea (ICCF17 Conference at Daejeon).

32.) The wire #2 “overcomes” 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 (USA), we realized that the R/Ro value of Active wire remained almost unchanged (about 0.81). At Austin, ALL the control and measuring electronics, and new specific software, were provided by National Instruments modules and Researchers.



Figure 8. Dr. Celani and his reactor at Austin (USA)

33.) The maximum excess power reached, after 3 days of operations (in public) at Convention center of Austin (Fig.8), NIWeek 2012, was about 21W with indirect heating and about 25W with direct heating of sub-micrometric 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.

V. CONCLUSIONS

It appears that the commercial Constantan alloy, with the surface deeply modified about geometry (i.e. skeleton type) and dimensionality of 20-100nm, multi-layers, is a good candidate for anomalous heat production due to:

- 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;
- c) use of Hydrogen.

We observed that such materials have a behaviour of “positive feedback” of anomalous power in respect to temperature increasing.

The experiment showed to be reproducible as experienced both during the Austin (USA) NI Week and ICCF17 Conference (Korea). 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. Anyway more and systematic work is necessary to elucidate the several open questions, first of all the stability over time of the anomalous heat generation, safety and a confirmation about reproducibility, not mentioning the “strange” behaviour using Deuterium gas.

Collaboration of the Community involved in LENR

studies is welcomed and a series of attempts to replicate the experiment is currently performed by different organizations and laboratories worldwide.

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 around 280°C).

If positive results will be reconfirmed with the wire made by new procedures (i.e. “second generation” of preparation), it could be possible to reach “regions” of operation were even the self-sustaining regime could be observed.

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