



Research Article

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 significant amounts of hydrogen (H_2) and/or deuterium (D_2) and might, in principle, possibly generate anomalous thermal effects at temperatures $>100^\circ C$. The interest in Ni alloys comes in part because there is the possibility to use H_2 instead of expensive D_2 . Moreover, a cross-comparison of results using H_2 instead of D_2 can be made and could help the understanding of the phenomena involved (and the possible nuclear origin).

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1. An Old Alloy Used For New Purposes

Based on some theoretical considerations, and thanks also to some sentences in a paper on catalysis not related to LENR studies [1], we decided to explore the possibility of using Ni–Cu alloys (including Constantan) as starting material that could fit our purposes.

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One of the merits was, according to us, the ability to dissociate H_2 . One of the Ni–Cu alloys ($Ni_{37}Cu_{63}$), among the materials studied in [1], has the highest disassociation value (3.2 eV; in comparison, pure Ni and Pd have values of 1.74 and 0.42 eV, respectively). Moreover, even with large changes (a factor of about 2) in the relative atomic amounts of Ni with respect to Cu (i.e. from 0.37 to 0.62), the dissociation values remain at quite high levels (from 3.16 decreasing to 2.86 eV).

We focused on a commercial low-cost material, brand name ISOTAN®, CuNi44. The mass components in percent are $Cu_{55}Ni_{44}Mn_1$. This material was developed several years ago by Isabellenhutte Heusler, GmbH, KG-Germany. ISOTAN® was selected based on the following considerations, as we pointed out in April 2012, Ref. [2].

A. A measurable diffusion coefficient of hydrogen, even in the pure (not alloyed) elements, i.e. copper and nickel, at high temperatures: Cu = $10^{-6} cm^2/s$ at 200°C, $10^{-4} cm^2/s$ at 700°C; Ni = $10^{-7} cm^2/s$ at 200°C, $10^{-6} cm^2/s$ at 350°C. In comparison, good values for Pd are: $10^{-5} cm^2/s$ at 200°C, $10^{-4} cm^2/s$ at 420°C; at 600°C reported values are as large as $8 \times 10^{-3} cm^2/s$, but are not reproducible. We suppose that the “flux” of H_2 or D_2 inside the lattice and/or near surface (either longitudinal or transversal) is one of the key factors needed to generate anomalous effects. Our opinion is based on the experimental observation that the anomalous effects increase with the increasing of R/R_0 value oscillations, i.e. loading and de-loading of hydrogen or deuterium.

B. Lower cost, overall, even considering the procedure to “build” nano-structured at the surface, compared to palladium, which is a very expensive precious metal.

C. This material has good mechanical properties and resistance to aging effects caused by cycles of both low→high→low temperatures, and cycles of H_2 absorption–desorption. Our first sample, (“generation one”) was used in a long duration experiment lasting for over 7 months; only after such a long time did we observe the beginnings of serious damage. Our results are, in that respect, different from those obtained by Szafranski [3]. He observed extreme brittleness in as-received Cu–Ni alloy that was only cold rolled from 200 to 20 μm . (Note that the penetration depth of H into Ni is about 30 μm .) His material was then cycled between 77 and 300 K under 1 GPa pressure of H_2 . We can only suppose that that high temperatures and/or adding manganese at 1% has the beneficial effect of reducing brittleness. We have never done experiments at 77 K, so it is difficult for us to judge.

D. Extremely large values of (computed) catalytic power (ΔE) in respect to the dissociation of H_2 [1], as shown in Table 1.

E. The possibility, at least in principle, of producing nano-micro structures (and voids) both at the surface and deeper into the bulk, with selective oxidation of Cu in such alloys at high temperatures (650–1050°C). Both segregation of pure Ni among to CuOx and the cooling rate are key aspects of the preparation need to be studied in deeper detail, even though we spent a lot of time and money investigating them.

Our exploratory studies were devoted to finding simple and reliable/reproducible procedures to get these kinds of structures. Experiments with the selected material were operated to last as long as possible: including “strength” and aging tests.

2. Sample Preparation (Procedures Used for the Experiment up to May 2012, “Generation One”).

Similar Composition Materials, All Nanometric, Developed Independently in Japan

In our exploratory preparations and tests, we used standardized wires: $\Phi = 200 \mu m$, $l = 105 cm$. Weight (307.4 mg enameled), Φ and resistance (17.16Ω) were carefully measured. The resistance measured in this batch was 5% higher

Table 1.

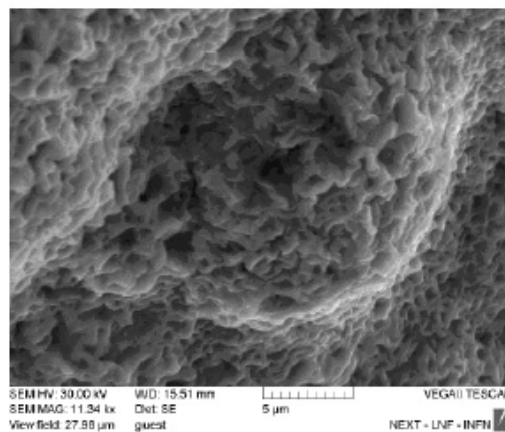
Alloy materials	Dissociation value (eV)
Ni0.3750–Cu0.6250	+3.16
Ni0.6250–Cu0.3750	+2.86
Ni0.8125–Cu0.1875	+2.10
Ni	+1.74
Ni0.1825–Cu0.8175	+1.57
Ag0.8125–Pd0.1875	+0.57
Ag0.625–Pd0.375	+0.51
Ag0.1875–Pd0.8125	+0.51
Pd	+0.42
Cu	–1.11
Ag	–1.42

than the nominal values reported by the manufacturer. We suppose the difference is related to iron impurities, which we detected by SEM-EDX analysis.

We point out that, although very promising results were obtained with pure Constantan, in our explorative test under H_2 gas (2–3 days of operations each, from February to June 2011), we *never* observed any type of anomaly, such as changing resistance, on wires with Joule heating up to 900°C under the following experimental conditions:

- (1) As received from the company (which we designate *ultra-virgin*); maximum temperature about 150°C .
- (2) With the enamel protection on the surface completely removed by burning up to 600°C in air, and stress released.
- (3) Acid etching of wire after treatment 2.

The wires in condition 2 at first were just “cleaned-up” with the original enamel insulating layer removed. This was done with Joule heating in air current up to 2000 mA, time 5 min.



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

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

In these conditions, the power dissipated was about 70 W and the resistance ratio, with respect to the reference value at 100 mA of current, increased only 1%. This is expected for this type of material (which has the commercial name Constantan, i.e. constant resistance). After the first thermal treatment, the weight decreased by about 13 mg, the resistance decreased from 17.16 to 17.02 Ω .

We found that increasing both the current (up to 2500–3000 mA) and the duration at high power (5–1000 s), decreasing the cooling speed (from 100 s down to <1 s) had dramatic effects on the growing of nano-microstructures and their dimensions (see e.g. Fig. 1). The role of O₂ and H₂O, because of open air treatments, is important. The wire temperature, in some tests, was even larger than 1000°C (rough evaluation by color temperature; the melting point of pure copper in inert gas is 1083°C, and the melting point of ISOTAN® is 1280°C).

The quality of the wire produced by this method was evaluated by SEM observations. We determined that the better methods of preparation resulted in smaller particles at the surface, and a larger mass of particles compared to the mass the whole wire (i.e. the core).

The best material, we were able to produce at the end of July 2011 using DC thermal treatments, was put in our flow calorimeter (which has uncertainty of only 2%).

As previously noted, this material was extensively studied both in H₂ and D₂ gas as well as in calibrations and tests in helium, argon, air and in vacuum. The total time of experiments was quite long (about 10 months) and only toward the end of the tests, after 7 months, the damage to the wire increased to such an un-controlled level that it prevented reliable interpretation of the experimental results. These results were discussed in detail during the “X International Workshop...”, April 2012 [2].

Key information: we were happy to learn that Akito Takahashi (Osaka University) and Akira Kitamura et al. (Kobe University) studied in secret (as we did) an alloy of Ni–Cu. In their case, most of the materials were at nanometric size, i.e. 5–20 nm dispersed in an inert matrix of ZrO₂. This work was performed in collaboration with Technova, a Toyota research group. We received brief information by them at the end of December 2011 about promising results from a specific alloy (Ni₈₅–Cu₁₅)_{35%}–(ZrO₂)_{65%} (for details see Ref. [4]).

We note that such material is a further development of the nanomaterial Pd_{35%}–ZrO₂_{65%} made by Yoshiaki Arata at Osaka University since 2005. This material is made with complex melt spinning and quenching (cooling rate > 10⁵°C/s) process.

Brief information about Ni–Cu–ZrO₂ came to my (FC) attention, for the first time, at the end of December 2011. I was invited to give a review talk on *Anomalous Effects in LENR Studies*, at the WSEC 2012 conference (January 10–12, 2012; Geneva) organized by the ISEO (Non-Governmental Organization cross-related to UN). I requested that everybody involved in LENR studies, worldwide, communicate the most recent and interesting results to include in the Review. Only Takahashi and Kitamura had performed experiments with Ni–Cu alloys and give permission to share their results, even though these results were qualitative and preliminary. A similar talk, with more technical and scientific details, was given at CERN (Geneva) on March 22, 2012 in the framework of the prestigious CERN Colloquium [5]. The overall behavior of Ni–Cu alloys, although at different ratios of the two main elements in response to H₂ and D₂ absorption, even with different geometrical shapes (powders used by the Japanese groups, wires from us) and amount of anomalous heat detected, were qualitatively similar.

Such evidence reinforced our intention to develop a better material from the point of view of nano-dimensionality, keeping the starting Ni–Cu composition “fixed”. Our efforts were devoted to increase the amount of active material at low dimensions (<100 nm) and, at the same time, reduce the adverse effect of “leakage” of the smallest particle from the surface due to: vacuum conditions, temperature cycling, loading, de-loading, etc.

3. New Transparent, Dissipation-Type Calorimeter

From the end of May 2012, we were able to produce sub-micrometric materials. Based on SEM observations, we expected performance several times better than the best material produced at the end of July 2011, with enough good reproducibility in the preparation procedures.

The new method, although similar to the old one in some key aspects, was really revolutionary in the practical parameters of mechanical stability (reduced leakage of the “good” material from the surface); and in the fraction of material at low dimensionality. The latter increased from 1 to 2% (*generation one*) up to about 30% of the whole material (*generation two*).

Such big improvements were obtained thanks to large financial help and man-power of an Italian company that “believed” in our previous results. We were able to design and build specific electronics and a mechanical set-up to produce sub-micrometric wires. Systematic, albeit very tedious and expensive experimental work was the key factor for success.

One of our goals was to see with the naked eye whether the wire was really stable in the rate of leakage of “good” materials even after several cycles of low→ high→ low temperatures and H₂ loading and de-loading. To this end, we build a new transparent reactor with borosilicate glass (Schott DURAN) of large (3 mm) wall thickness enough to withstand large pressure drops (up to 8 bar), at internal wall temperatures up to 280°C.

For the calorimetric measurements, we adopted the simplified approach of measuring the temperature on the outside of the glass wall. Taking into consideration the temperature of interest, i.e. $T_{\text{wall}} > 140^\circ\text{C}$, one of the main channels of heat exchange to the environment is heat radiation. In other words, we can use the Stefan–Boltzmann law:

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

In this formula the temperatures T are in K and ε (the emissivity) is about 0.9. Calibrations were made using our usual procedure to add an “inert” wire, very close to the “active” one, and to make several measurements with inert gases. In the specific new set-up, the wires were parallel, alternatively and helicoidally shaped, with 22 turns. They were stepped through a range of input power levels in different gases (helium, argon and in a vacuum). Electric power was applied alternatively to the inert Ni–Cr wire and then to the active Constantan wire.

Because in our experimental set-up the geometrical dimension of the cell is constant (glass tube, external diameter 40 mm, internal diameter 34 mm, overall length of 280 mm and central active length of 100 mm), we can make a sort of simplified calibration curve just dividing formula (1) by the input power. Obviously, we neglected the contribution to heat dissipation by free air convection (which is 5–35 W m² K, in usual environments, at 12–15 W). We note 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 (>2.5 μm). Moreover, the effects of pressure variations *inside* the reactor chamber, with related temperature variation due to different convection values (i.e. the internal temperature increases versus pressure decreasing), can be neglected at the external wall. Tests were performed in helium by varying the pressure between 6.5 and 2.5 bar. These tests were made at the beginning of the experiment, when the wire was new and the reactor glass wall “clean,” and again after a few months, when the wire was used and the glass wall lightly “dirty.”

4. Results with the New Wires (*generation 2*)

At the end of May 2012 two wires were produced: an old batch (with iron) with new procedures (*generation two*).

The first one was used a few days later to the experiment, the second one was just put inside a HDPE envelop and kept closed at Room Temperature (RT). We designated the experiments:

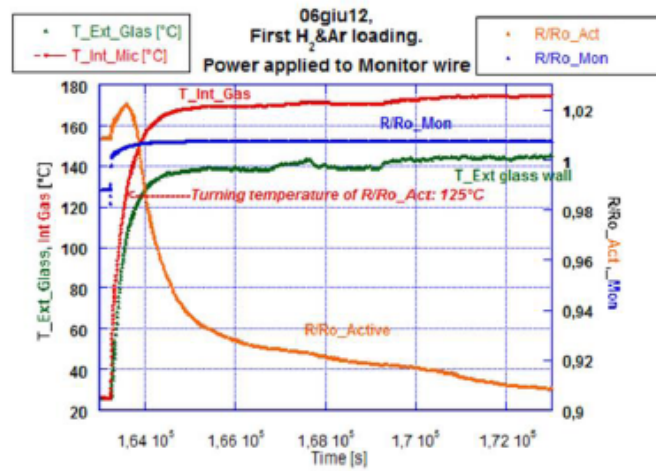


Figure 2. Details of first loading by H₂–Ar mixture.

[a] wire#1 (started in 06 June 2012);

[b] wire#2 (started in 10 July 2012).

The main improvements with respect to the previous procedure of fabrication, according to SEM observations, were multilayered structures, and total number of such layers, “which was extremely” large: “on” 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 improved.

The primary experimental procedures and results are as follows:

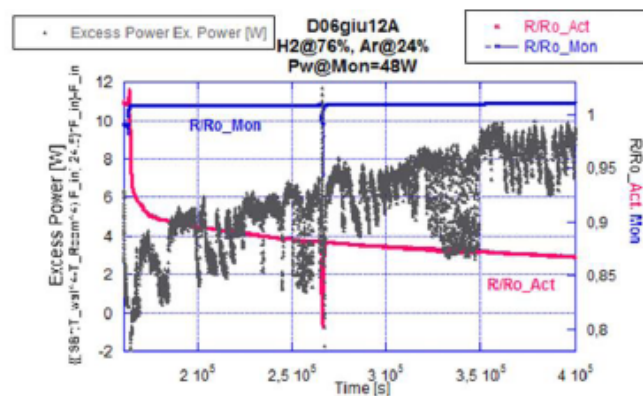


Figure 3. Behavior of anomalous power generation, using indirect heating, i.e. power (48 W) applied to Monitor wire.

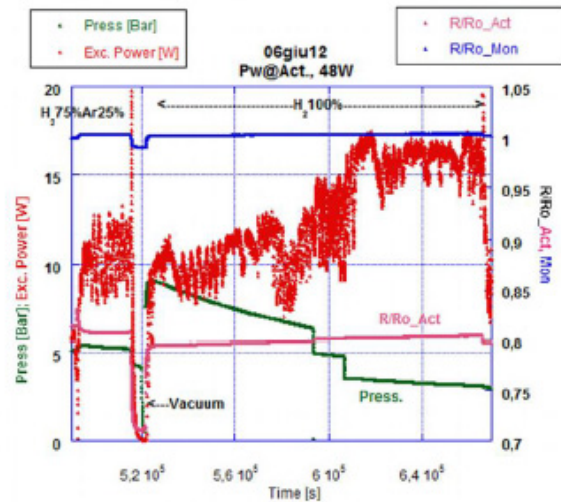


Figure 4. Experiment with 48 W applied to the active wire. Up to time 518 ks the gas mixture was H_2/Ar at a 75/25 ratio. Later, the power was reduced to zero and the cell was evacuated. The R/R_0 ratio was as low as 0.71. At time 522 ks pure H_2 was added: the excess power resumed, at the same level it was before the cell was evacuated. After a controlled reduction of pressure, excess power increased up to 16 W.

- (1) In order to use simple parameters easy to be managed by calculations, we adopted the usual term of R/R_0 . R_0 is the initial value of resistance at room temperature, i.e. at $23.5^\circ C$ (in that calibration), in a free air atmosphere, inside the reactor. With our wires ($l = 100$ cm) we measured, in situ ($I-V$ methods), a value of resistance of 16.9684 and 57.4394 Ω , respectively, for sub-micro_Costantan and Ni-Cr (presumably inert) wires. The

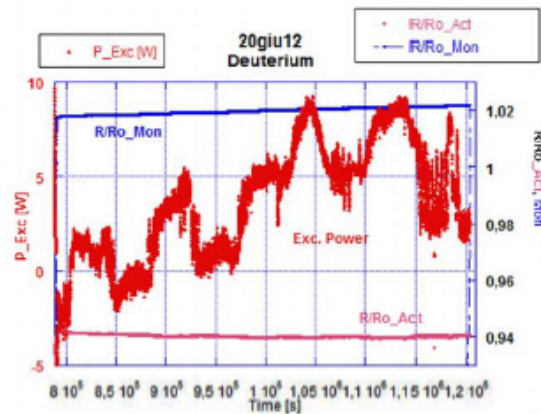


Figure 5. Experiments under D_2 gas. From the beginning, continuing for about a day, the reaction was endothermic; it later crossed the zero line and began to be exothermic, as it usually is with H_2 .

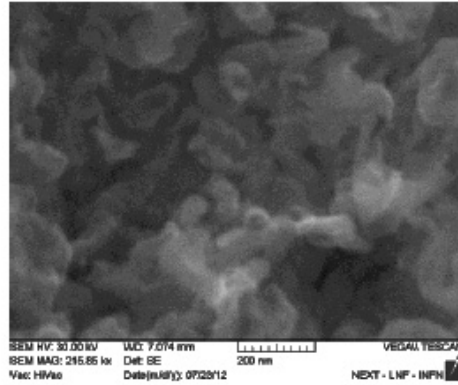


Figure 6. SEM after long H_2 interaction at HT. Experiment #1.

measuring currents were just 4 mA, to avoid self-heating of the wires.

- (2) We began the tests by calibrating with inert gases at the power levels of 5, 15, 30 and 48 W 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 references, were made with the active wire. Using the values of temperatures measured outside the glass cell and ambient it was possible to evaluate the “power exchange constant” ($^{\circ}C/W$) of the small reactor by (1).
- (3) After adding a H_2/Ar mixture (75/25 ratio) at 7 bar of total pressure, while monitoring with the resistance ratio R/R_0 of both the active and inert wires, 48 W of power was applied 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 the active wire, after a very limited increase to 1.02, dropped to 0.92 at 2500 s. Later on, at about 100,000 s, the R/R_0 decreased to 0.88. We observed a correlated increase of anomalous excess heat, which was quite unstable with the R/R_0 decreasing. The temperature inside the cell was about 180°C.

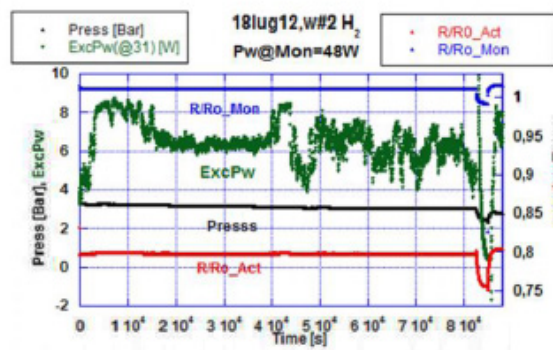


Figure 7. An example of anomalous heat from wire #2.

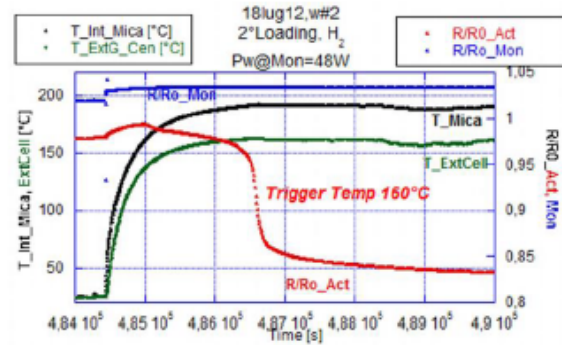


Figure 8. Behaviour of first re-loading of wire #2.

- (4) After 103,000 s (29 h) from the beginning (Fig. 3), we stopped the power to the inert wire and allowed the reactor and the wires to cool to room temperature. The R/R_0 value of the active wire decreased to 0.80.
- (5) Just after that, we powered the inert wire at the same power level and another 150,000 s (42 h) after the interruption we measured an R/R_0 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/R_0 value (low decrease). The instability of excess power, assuming there were no other uncontrolled parameters distorting it, had values quite large and was correlated to the small oscillations ($<1\%$) of R/R_0 values.
- (6) We observed that even the instabilities of room temperature (usually $23\text{--}27^\circ\text{C}$) helped anomalous heat production in some ways. We speculate this was because these instabilities introduced non-equilibrium conditions. To avoid errors in the interpretation of results, after a sufficiently long time, we note that the values of room temperature were the same as at the start of the experiment, while the anomalous heat increased over time.
- (7) The long-lasting positive effect with H_2 gas was also observed by the Takahashi and Kitamura groups (reports at X Pontignano Workshop and [4]). Depending on their conditions, constraints and materials, during 2-week

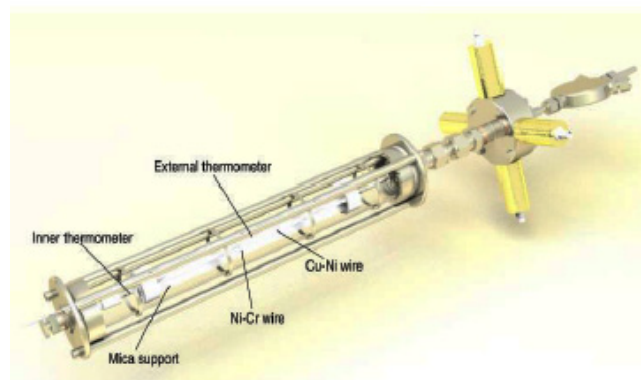


Figure 9. Schematic of the cell assembly.

long experiments, the anomalous excess power slowly increased from 0 up to 3 W.

- (8) We observed that the minimum cell temperature at which anomalous heat is observed is around 120°C, i.e. close to the first “loading” temperature of 125°C.
- (9) Around 330,000 s (4 days) after the first H₂/Ar intake, the active wire was powered (Fig. 4).
- (10) We observed a further increase of anomalous power that, assuming no errors were made, was about twice that detected when the power was applied to inert wire; that is, of more than 10 W. The R/R_0 value, after initially increasing, stabilized at 0.808.
- (11) A possible explanation was that the active wire’s temperature with Joule heating was larger than that when the power was indirect. The active wire temperature was 350–400°C, 200°C with indirect heating.
- (12) If the explanation in 11 is correct, we see that the reaction, above from some temperature threshold, has a *positive feedback* with increasing temperature. Similar effects were found by our group with first generation wire/experiment, up to May 2012; and by the Takahashi and Kitamura group with Ni–Cu–ZrO₂ powders.
- (13) After 360,000 s (4 days) from the H₂/Ar gas intake (Fig. 4), i.e. at time 515 ks, the power was switched off: the R/R_0 , at room temperature, dropped to 0.71. In other words, the direct heating (electro migration phenomena and/or larger temperatures) improved the loading, and then improved the anomalous power.
- (14) After 410,000 s (5 days) from the first H₂/Ar intake, we evacuated the cell and then added H₂ at 100% concentration.
- (15) The results were similar to H₂/Ar gas. The cell produced even more anomalous heat.
- (16) We cannot discriminate whether the improvements in performance were due to effects of pure H₂, or due to more time under active gas, leading to increased embrittlement.
- (17) After another week of miscellaneous tests, we decided to de-load the H₂ absorbed by the wire, to be sure that the resistance reduction observed was due to real absorption and not to a variation of resistance caused by the reduction of oxides (by H₂) at the nano-particles surfaces.
- (18) To de-load we put the cell under a dynamic vacuum and increased the temperature.
- (19) After several hours, we returned to the original starting value of R/R_0 at 1; the test was fully successful.
- (20) We reloaded the wire again and observed the R/R_0 decrease and anomalous heat, similar to what we saw in the first cycle.
- (21) Again we de-loaded H₂ from the wire, to do experiments with D₂ gas (Fig. 5). This time the final value of R/R_0 was 0.93, not 1.0 as expected. We suppose that some H₂ was stored somewhere in the lattice.
- (22) After deloading the wire, we increased the temperature by powering the inert wire, as we usually do. Only a small amount of deuterium was absorbed.
- (23) We observed, for the first time since 2011, some X and/or gamma ray emissions, coming from the reactor while the temperature increased from about 100°C to 160°C. We used an NaI(Tl) survey meter with an energy range 25–2000 keV, which was operated for safety purposes, not as a spectrometer. The total duration of this emission was about 600 s. The emission was burst-like and clearly detectable.
- (24) We observed a very surprising thermal anomaly: the reaction was endothermic at first, not exothermic. On the second day the system crossed the zero line and later on become clearly exothermic. Similar effects were reported also by Takahashi and Kitamura.
- (25) After about 350,000 s (4 days) from the beginning of D₂ intake the temperature abruptly increased and the wire broke. We noted that the pressure decreased, because there were problems with the seal, and the reactor was not perfectly gas tight, but this occurred at 80,000 s, *before the wire broke*. SEM observations showed fusion of a large piece of wire: ball shape. An SEM analysis after H₂ and/or D₂ interaction shows reduced size (Fig. 6).
- (26) Starting on July 10, 2012, we used the second wire (#2) that had been stored in a plastic bag.
- (27) In the meanwhile, we improved the overall detection of external temperatures by adding 3 more thermometers. The main thermometer was moved from the original position, which was not central over the wire length, to

exactly at the center of the area of the glass tube.

- (28) The results were qualitatively similar to the first wire, although at lower intensity (Fig. 7). The starting temperature of loading increased 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 by something (perhaps HDPE plastic).
- (29) On July 23 we de-loaded the wire, and on July 24 we loaded it again. The sequences were: (a) dynamic vacuum conditions, 220°C internal reactor temperature, power at Ni–Cr, 50,000 s duration; (b) H₂ filling.
- (30) The results in Fig. 8 showed improvements in the speed of loading. It took only 2000 s for the R/R_0 ratio to fall from 1 to 0.85. The time necessary to get measurable anomalous heat was also reduced to less than 6 hours.
- (31) The experiment with the cell shown in Fig. 9 was stopped on July 28, in order to package and ship the reactor to the National Instruments NI Week meeting at Austin, Texas in the US and later to the ICCF17 conference at Daejeon, South Korea.
- (32) The wire #2 “overcame” the severe effects of shipping, especially the long time (8 days) without H₂, when it was exposed to free air. When we reconnected all the electrical connections at the NI Week conference, we realized that the R/R_0 value of the active wire remained almost unchanged (about 0.81). At the NI Week conference, all of the control and measurement electronics and software were provided by NI. In the previous experiments in Italy we used DAQ (6.5 digit) and MUX by Agilent; specific hardware and software were homemade.
- (33) The maximum excess power reached after 3 days of operation in public at NI Week 2012 (Fig. 8) was about 21 W with indirect heating and about 25 W with direct heating of the sub-micrometric Constantan wire. The input power, as usual, was 48 W. These are the best values that we have observed up to now. We note that because we used the “old” value of calibration obtained in Italy, but a different experimental geometric set-up and instrumentation, the absolute value of excess power has to be scrutinized. In any case, the trend of increasing excess power versus elapsed time was reconfirmed.

5. Conclusions

It appears that the commercial Constantan alloy, with the surface geometry deeply modified (i.e. skeletonized) and size reduced to <100 nm, with multiple layers, is a good candidate for anomalous heat production due to:

- (1) Intrinsic low cost of raw materials;
- (2) Simple, low-cost procedures for growing nano-structures, as recently developed by our group at INFN-LNF, Italy;
- (3) Use of Hydrogen.

We observed that such materials exhibit “positive feedback” of anomalous power with increasing temperature.

The experiment has been shown to be reproducible as experienced both during the NI Week and the ICCF17 conferences. Several of the results we obtained were similar to those obtained using Ni–Cu alloy dispersed in a zirconia matrix by the Takahashi and A. Kitamura groups. (Both of these groups are in a collaboration with Technova, a research organization of the Toyota Company.) More systematic work is necessary to elucidate several open questions, first of all, the stability over time of the anomalous heat generation, safety and overall, third party independent confirmation about reproducibility, not to mention the “strange” endothermic behavior using deuterium gas.

Collaboration with the community involved in LENR studies is welcomed. A series of attempts to replicate the experiment is currently being performed by different organizations and laboratories worldwide.

The next step will be to use a quartz tube instead of the borosilicate now in use. Quartz will allow studies of temperatures over 300°C. Borosilicate glass begins to soften at around 280°C.

If positive results are reconfirmed with the wires made with the new procedures (i.e. “second generation” wires), it may be possible to reach regions of operation where even self-sustaining operation occurs, using larger amounts of materials.

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