

Research Article

Celani's Wire Excess Heat Effect Replication*

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Abstract

This paper describes progress made by the Martin Fleischmann Memorial Project while attempting to replicate Celani's experiments. Celani claimed to see consistent and reproducible excess heat generation results coming from treated constantan wires using different protocols. The design of the cell is described in detail, with attention to the choice of materials, the design geometry and operating conditions. Differences between the original experiment and later replications that improved believability are explored. Results and interpretations are discussed.

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1. Introduction

During ICCF-17 in Daejeon, South Korea, Celani demonstrated an apparatus that used a treated constantan wire as the active material in a hydrogen atmosphere. This wire had been specially treated to enable activation for the sub-micrometric material. Before the end of the conference, the newly organized Martin Fleischmann Memorial Project (MFMP) decided as its first project to replicate the Celani experiment using wires provided by Celani.

Three different protocols similar to Celani's [1] have been used. The first one was identical to the one presented at ICCF-17 [2]. A second, slightly improved protocol similar to the one later used by Celani et al. [3] was then used. Finally, we compared the active cell to a dummy cell in order to make the results independent of the laboratory ambient temperature changes.

*This work is dedicated to the memory of the great Prof. Martin Fleischmann.

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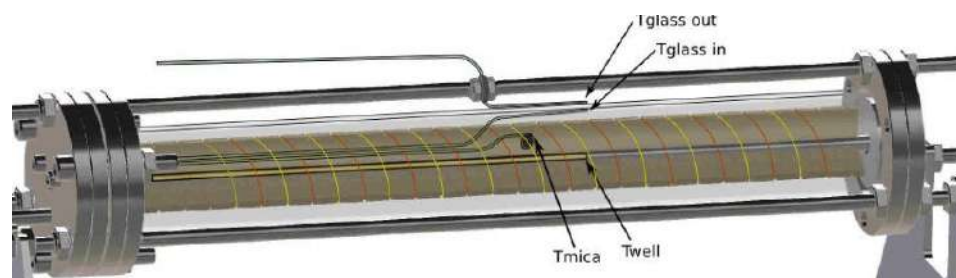


Figure 1. Design of the replica. In red the active wire wound in parallel with the heating wire in yellow.

2. Original Replication of the Experiment

The first experiments used a device similar to Celani's. The reaction chamber was a Schott Duran borosilicate tube 40 mm OD, 3.2 mm thick and 300 mm long equipped with Conflat flanges at each end. A third flange was used for thermocouples and power leads. The low transmittance at infrared wavelengths of this type of tube is important because it allowed Celani to assume that most of the heat loss was by radiation. Two wires were wound in parallel, the first one being the active wire prepared by Celani (200 μm), and the second one being a 150 μm nichrome wire (80% Ni, 20% Cr from Goodfellow). The wires were wound on mica supports. We used 0.5 mm thick mica whereas Celani used 3 mm ones. The mica was pre-baked in an oven for 30 min at 330°C prior to use in order to complete the polymerisation process of the silicone binder used during the mica manufacturing. The wires were not wound as closely together as Celani's device. One-meter long wires were wrapped on 28 cm length on the mica supports, whereas Celani wrapped 1 m of wire per 12 cm. This is the main difference from the original design. Another difference was related to the equipment used to monitor the experiment.

The system was placed 20 cm above an aluminum plate. Four thermocouples were used in this setup. The first one measured the ambient temperature 50 cm away from the experiment; the second one was placed on the mica support; the third one was positioned on the inner surface of the glass tube; and the last one was on the outer surface of the tube. An additional thermocouple was placed inside the central support, which is called the "well". Celani used Kapton tape to glue the thermocouple on the glass surface, but we did not, because the tape stores heat locally, and also because of the difficulty of reproducing this design correctly. We found that the elastic property of the stainless steel sheath was sufficient to allow an acceptable measurement of the glass tube in contact with the thermocouple. The end-tip of the thermocouple measuring the outer surface temperature of the tube was placed at half the height of the tube, half the length of the horizontal tube with a large length of its end sheath making an asymptotic curve meant to collect heat and prevent the sheath from playing the role of a heat sink. We operated in constant voltage mode. The acquisition system was based on a custom board made by Hunt Utilities Group with data acquisition at 50 samples/s and 16 bit resolution.

Experiments were performed in parallel in Europe and in the United States. In Europe the room temperature was regulated with a precision of $\pm 1.4^\circ\text{C}$. In the US the system was placed in a vent hood with continuous airflow having a stability of $\pm 1^\circ\text{C}$.

3. Method of Excess Energy Calculation Compared to the Celani's Experiment

The original experiment done by Celani used the Stefan–Boltzmann black body radiation law to calculate the heat loss of the system using the emissivity of the glass tube. The calculation of the heat loss using this law is only valid in a vacuum. In addition to the energy lost by radiation, it is necessary to take into account the heat loss by conduction and convection. In order to simplify the calculations, Celani calculated the effective emission coefficient of the cell that

empirically includes the other sources of heat loss. This method is not accurate enough. It is much better to calibrate the cell with an inactive wire for comparison.

To evaluate the influence of the different parameters, we looked at the variation in the thermal response of the system when calibrated under different gas mixtures and pressures inside the tube. The wire temperature is difficult to measure because the closest thermocouple is placed on the mica, and not on the wire. Moreover, exposure to highly thermally conductive hydrogen gas makes comparisons of wire temperatures close to impossible. Therefore only the external temperature of the glass tube was used to calculate the thermal energy (Fig. 1). The calibration curve was obtained by increasing the input power from 25 to 100 W in 5 W steps.

4. Calibrations and Results of the Original Attempt

Altogether 13 calibrations were made in Europe with an untreated 200 μm constantan wire. Three typical gas mixtures composed of Ar, He, and H_2 were used: 75% H_2 with 25% Ar, 100% He or 100% H_2 at 1 bar, 2 bar and 3.5 bar. Figure 2 shows an example, three calibrations at 1 bar.

For calibration we used a 200 μm diameter constantan wire coated with MnO on the surface (manufactured by BLOCK). It was supposed to be a good candidate for cell calibrations because it is advertised as the most stable constantan on the market in any operating conditions. We discovered that the manufacturer should say it is stable in any condition other than in a hydrogen atmosphere! Almost 2.8% variation of resistance was observed in this wire during calibrations. This variation raised serious questions about the possible “activation” of the BLOCK constantan.

However, compared to Celani’s wire, this material showed good stability at high temperature, the MnO surface having an oxide surface similar to the active wire. It also had resistivity and structural behaviour identical to Celani’s wire.

The two blue lines shown in Fig. 2 are the very first calibrations done with the BLOCK wire (*dark blue*) and the Celani wire (*light blue*). For the latter, it was not necessary to extend the temperature range higher as it is assumed that it would not deviate from the linearity of the curve. This was also not done because Celani advised us not to expose the wire to helium, because it would take the place of mono-atomic hydrogen in the “skeleton” of the nanomaterial, obstructing that path during later exposure to hydrogen. The light red line is the highest calibration baseline, where the BLOCK constantan wire was used in hydrogen.

The thermal gap between these two calibrations is very large. In terms of timeline, the dark blue curve was generated first, then almost all the others calibrations were close to the light red line. (They are not shown in this figure for the sake of simplicity.)

It is possible that hydrogen changes the optical transmittance/opacity characteristics of the borosilicate tube, thus modifying the thermal response of the system. Changing the tube would have reset this effect. It has also been postulated by Langmuir [5] that glass may have better properties to recombine hydrogen than other materials.

A series of test was performed to evaluate the leakage rate, the effect of the mixture on the system and on calibrations. This information can be extracted from the logbook^a. This was done to anticipate the behaviour of the wire after the first exposure to hydrogen, which is known to change it irreversibly.

A mistake made by the operator required to replace the tube, and clean the chamber and the mica before installing the active wire. Then power input was set to the previous value with an upper limitation of 45 W. In doing only the first range of power input that was used to generate the light blue calibration curve, we ensured the change of the tube and the modifications made on the cell did not impact the previous calibrations.

A final step was necessary before getting into the excess energy mode. This step is also known as the “loading phase”. It consists in exposing the wire to hydrogen for the first time, then increasing the electrical input power in

^a<http://goo.gl/i19B2f>

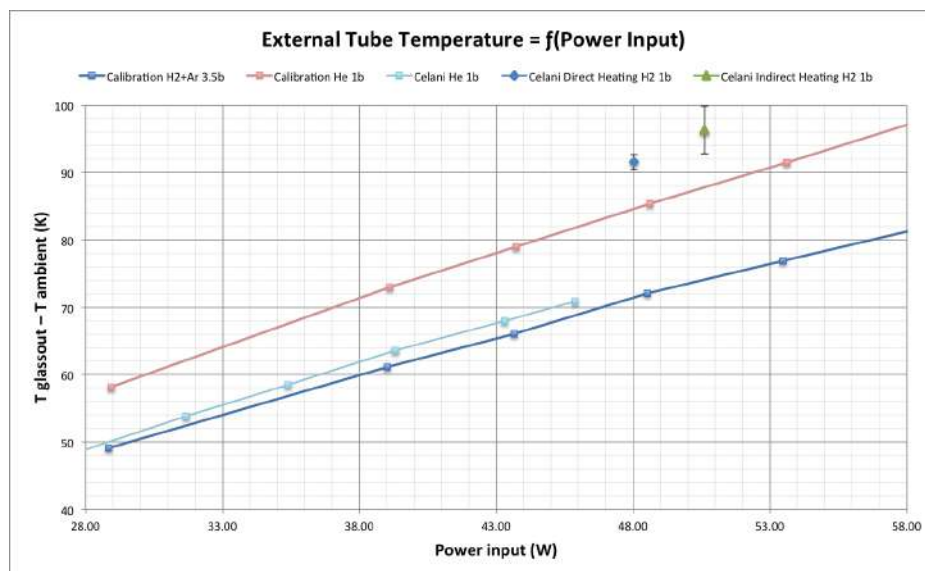


Figure 2. Results compared to the calibrations. The calibration in red reached the highest temperatures of all calibrations done.

order to achieve a dramatic reduction of the wire resistance. Further details are given in Section 7. The resistance was reduced by 16% of its original value from 19.4 Ω down to 16.3 Ω .

Soon after the resistance stopped dropping, the wire was set in the replication condition, with an input of 51 W of direct current in the nichrome wire (indirect heating), in an atmosphere of pure di-hydrogen. In Fig. 2, the measurement shows excess energy compared to the calibrated thermal response of 6 W in averaged value of the darker red plot, if we compare to the light red calibration line the graph shows an estimation of 20 W excess energy. This was run for 24 h before the power was switched to the Celani wire.

Whereas the input power is slightly less (48 W), the energy provided by the wire is similar. After this, power is generated in the same range as before, 6 W for two days. Then the cell was prepared for shipping to Switzerland where it produced similar results.

Due to many improper conditions, and a lack of confidence in doing this type of experiment for the first time, it was decided by the project members to move forward with a better protocol.

In this new experiment a “dummy” cell gave a permanent baseline to the active cell. Due to the unstable nature of this cell design, a permanent baseline is an asset to understanding the system. All the work done before remained archived in a comprehensive folder accessible by everyone^b on the internet to verify the measurements.

While many people remember results reported during ICCF 17 and NI Week 2012 of 30% excess energy, we were surprised to be informed by Celani that results in this range were no longer attainable. These results were a mistake in the measurements, induced by the power supply, that underestimated power input, adding to the apparent excess energy.

^b<http://goo.gl/2VRb0k>

5. Description of the New Protocol Done in Dynamic Vacuum

Celani did a test in December 2012 that involved a two-layer wire and a three-hundred-layer wire installed in the cell he used previously. The variations that characterised this test are related to the pressure induced within the chamber of the cell. Instead of having positive relative pressure, the chamber is put under constant vacuum in order to artificially insulate the wire within the reaction chamber and increase its thermal dissipation. With higher temperature involving less power input, the signal/noise ratio is increased. This technique also addresses some legitimate concerns about the validity of measurement made between H₂ and He, and the pressure variation of the gas, which would change the heat dissipation. It also improved the thermal stability. Finally, it enhanced infrared radiation over conduction and convection.

This protocol is available in two forms, as a table^c or as a text^d. To summarize briefly, two locations ran this protocol almost synchronously, in the USA and in Europe. The protocol is again split in two major steps, the calibrations and the experimental run.

Each set of two cells is run simultaneously with similar power input and conditions to create a valid baseline for comparison, in a calibration. This baseline is established under conditions identical to the live experimental run that follows. The only major distinction between the calibration and the live run is that the loading process is not done before the calibration. When the thermal response is established for each specific input power level, a third order polynomial approximation is made with a 95% confidence level as a minimum. Finally, this polynomial equation is inserted into the software in order to compare the current temperature reading with the baseline. By simple subtraction it gives the excess heat in real time for the Live Open Science broadcast.

This protocol allows us to set conditions for the wire to be loaded and unloaded back and forth. This is an interesting test because it tends to stress the material, induce a flux of protons, and speed up the aging process in comparison to the static pressure test.

6. Description of the Second Iteration Apparatus

The second apparatus is a modification of the first attempt with the aim to fix the wounding density and to be as close as possible to the original cell presented in ICCF-17. It uses a modified version of the first attempt, where the wrapping is more concentrated at the center using 12 cm of the central part of the tube instead of using almost the entire length of 30 cm. The mica supports are made of MACOR instead of steel. This is identical to Celani's apparatus, and it improves the insulation on the sides of the system.

The most noticeable change in this experiment is that two cells are mounted in the same place. One cell contains treated constantan wires made by Celani and the other one contains nichrome wires that are considered not reactive at such low temperatures with hydrogen.

The data acquisition as well as the power supply is duplicated to provide the additional means corresponding to the mirroring of the initial cell. The nichrome is re-designed to get as close as possible to the median value of the resistance reached by the wire during and after loading. A diameter of 0.3 mm is hence used in place of the 0.15 mm to fit this constraint.

Three thermocouples are used to measure the surface temperature of the tube on each cell. This was a good way to measure the value of the thermal gradient withstood by the tube. In Europe the end-tip of each thermocouple was warped in an SEM-type carbon sticky tape to allow better and stronger thermal coupling between the two parts. In America, a copper ring was added and wrapped on the surface of the tube to make the coupling.

^c<http://goo.gl/VY5MMB>

^d<http://goo.gl/z42Eqv>

The loading process however is done differently between the two cells. Because the “passive cell” has a non-activated wire, this cell is never exposed to hydrogen. Only the “active cell” is loaded with hydrogen until the resistance drop reaches a constant value. Then the vacuum is pulled again in both cells before starting electrical power input.

The vacuum level is identical in both cells during the experiment phase and constantly measured. We decided to use a vacuum of 2 mbar in both laboratories because that is the highest vacuum the European lab was able to achieve. We soon realized that this level of vacuum was high enough to deload the wires almost completely and quite quickly, even though it was not high enough to thermally insulate the wire from its environment.

Whereas the thermal stability of the room was improved in Europe, the US team decided to use insulating materials forming a box around the cells and flow constant temperature air over the cells to achieve a more stable environment.

7. Analysis of the Resistance Drop

The resistance drop is an effect that was assumed upfront by Celani to be related to the level of hydrogen the material stores in its metallurgical lattice [1]. This assumption is derived from the work done by McKubre et al. on how deuterium absorption relates to the increase of resistance. While McKubre presented his work on the H/Pd or the D/Pd system, with a description of a Baranovsky curve of the system studied [6]; no similar study has been done so far of the H/Ni system. Hence it is an assumption that must be taken with caution. However Ni was already a candidate for excess heat effect study, due to the close chemical properties of Pd and Ni.

Figure 3 is plot showing how the resistance of the wire changes over time. The temperature of the wire is unknown; only the temperature of the mica holding the wire is known, and it is equal to 200°C. The power was held constant for six hours, and then turned off for one hour. The aim was to monitor regularly how the resistance changes over time.

As we can see the resistance is reduced as time goes on. This might be due to reduction of the surface oxides present on the surface of the wire up to a point. However, resistance drops down to 30% of its original value cannot be explained with oxide reduction only, another phenomenon is taking place in the sample.

Because the value of resistance before and after the one-hour power down period is identical, the effect of the cooling is negligible. But one unexpected effect on the understanding of the resistance variation using this power input pattern was observed: the variation of resistance is a nonlinear effect. This is proved by the increase of the amplitude

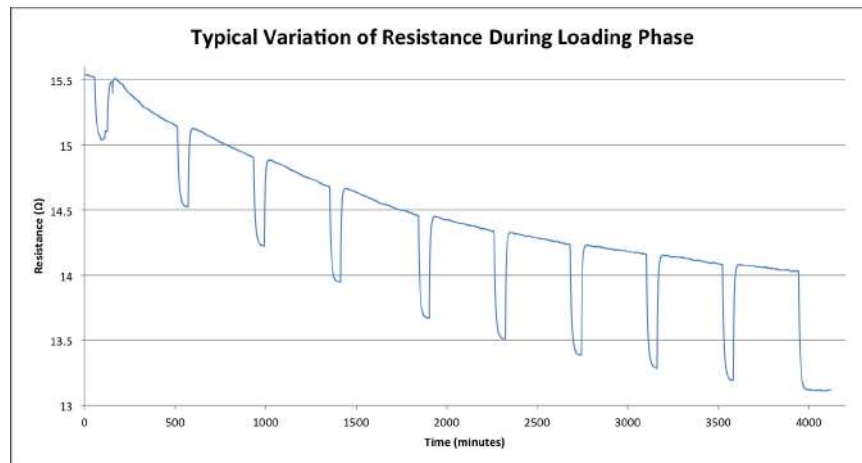


Figure 3. Example of resistance drop while the wire is set at proper temperature under hydrogenated atmosphere. Power is set on for 6 h then turned down for 1 h, 10 times.

of the drop each time it happens. We can then assume that if the resistance drop is related to hydrogen absorption, then the effect is tempering while the ratio Ni/H is still increasing.

The most compelling understanding MFMP has is that resistance drop requires a specific minimal temperature to start appearing. While temperature is increasing, the resistance is also increasing too. Once this value is passed, the resistance drops by 10% up to 30% and sometimes more.

8. Analysis of the Latest Results

Even though some limitations are induced by the setup, interesting results were obtained, demonstrating the capabilities of Celani's technology. While we compared the cells to one another, we carefully calibrated them under identical condition without any exposure to hydrogen. Helium or vacuum were used for calibrations. Vacuum was only used in the identical comparative configuration.

One other variation of temperature between the passive cell and the calibration is due to the nature of the gas used. Although He is a much smaller atom than the hydrogen molecule, the vibrational mode of this gas allows it to expel more heat than helium does, hence lowering the thermal response consequently. Calibration against Ar would have been a good option since the heat transfer is similar to di-hydrogen.

Figure 4 shows the comparison between calibrations and the run for each cell, in an identical environment. As explained above, helium creates a different thermal response of the cells. We expected to see the calibration show a hotter temperature than the hydrogen run. This is seen with the dummy apparatus. However, the cell using the active component from Celani runs significantly hotter than the dummy cell. A maximum of 5°C is seen for 25 W of power input as shown in Fig. 5.

The main issue with this protocol is that hydrogen exposure of the borosilicate tube is not even between the two cells. While the activated wire in one cell is loading, the second Celani wire – the one not yet activated – is still exposed

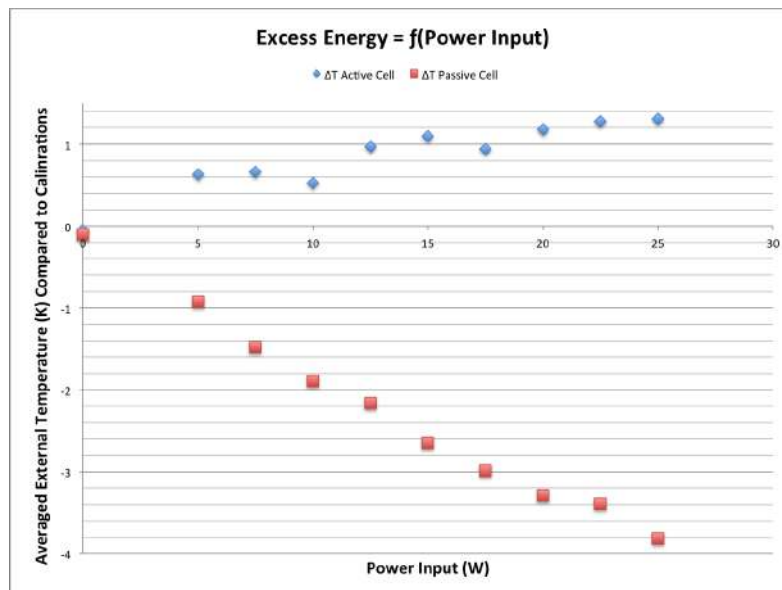


Figure 4. Results of the differential test subtracted to the helium calibrations. Active cell shows consistently higher temperature compared to calibration whereas passive cell is cooler.

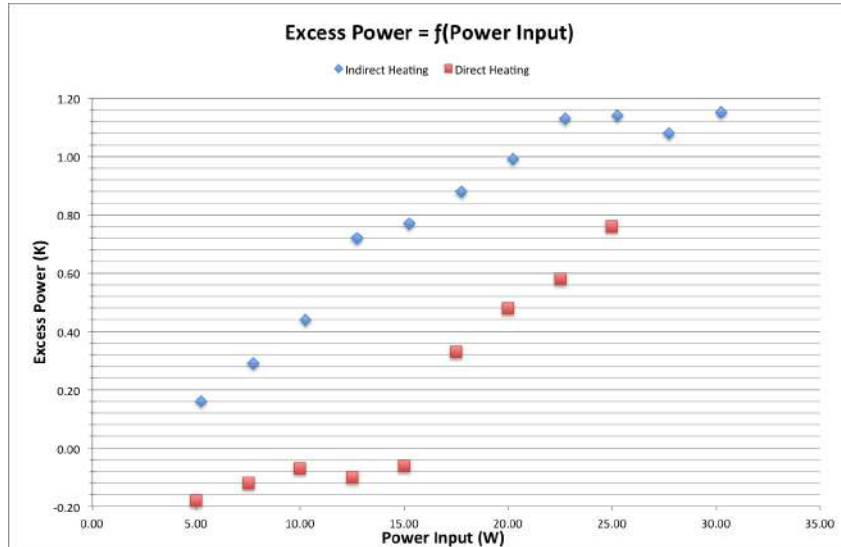


Figure 5. Excess power as a function of power input on a set of differential apparatus under dynamic vacuum condition.

to vacuum. Since we did not have the optical instruments to evaluate the changes of the glass characteristics, we cannot characterize this effect. This will eventually be the subject of further investigations.

Another effect that takes place on the surface of the wires is related to the cycling of loading and unloading. This

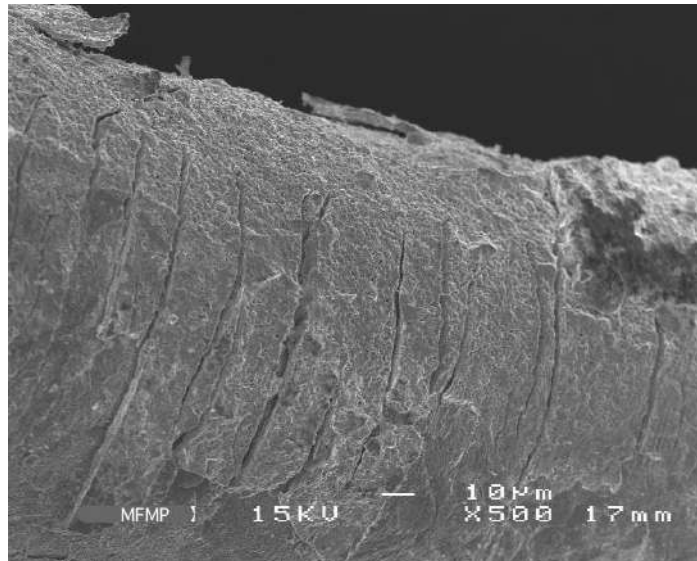


Figure 6. SEM image of the surface of the wire used under the repetitive dynamic vacuum protocol. Hydrogen embrittlement as well as vacuum conditions have forced the hydrogen to leave the wire, inducing cracks on the surface of the material.

is shown by the protocol used. As soon as the cells are put under active vacuum, the resistance of the wire rapidly increases. This releases hydrogen from the wire, which produces apparent excess heat.

The yield of energy released by the system is lower and lower over time and shows a fatigue effect. This is probably due to expansion of surface cracks that takes place. This would also be triggered by enlargement of cavities volumes in sub-surface microstructure. Finally, repetitive reduction and oxidation of the metal structure tends to disrupt the structure, as shown in Fig. 6.

9. Conclusions

The results produced by the original experiments done by the Martin Fleischmann Memorial Project show very similar orders of magnitude to what Celani has shown in his papers. Because Celani discarded his results during ICCF-17 and NI-Week 2012 due to an acquisition issue that under-estimated measurement of the input power, we also discard these results, and do not include them in our comparisons.

The range of coefficient of performance provides a solid answer to questions made regarding the level of excess energy showed by the apparatus built by Celani. With an optimum yield of excess heat measured for 15 W of power input, the system is very interesting. During the first test 12% excess heat was demonstrated, and 5% in the second test.

However, the calorimetric method used in that case is very much a concern and required a lot of effort to master, which should not be necessary. Two main criticisms are relevant to this calorimetric method. First, during the experiment, the infrared emission is changed a little because of the reduction of the oxide layer originally present on the surface of the tested treated wire during calibration. Secondly, the sensitivity of the system to environmental changes is pronounced, making it difficult to implement, measure, and achieve confidence in this method.

There is also a possibility that the tube changes its transparency while pressurized with hydrogen. This is one of the many possibilities that could change the measurements over time.

Finally, the experimental run under vacuum showed the same trend without replicating the amplitude of the results. Between a lack of confidence due the inexperienced operator in the first attempt, and the small signal during the last attempt, conclusions about the nature and the amplitude of the effect are still to be verified. Mass-flow calorimetry has been widely requested in crowd-sourced comments and by the scientific community. This will soon be implemented.

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