*Improved stability and performance of surf*ace-modified Constantan wires, by chemical additions and unconventional geometrical structures.

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OUTLINE

- **Motivation.**
- **Description of experimental set up: key details.**
- **Details on wire preparation with knots.**
- **Sub-micrometric surface modification of Constant wires by High Power Pulse (LNF-Frascati procedure) and repeated addition of Fe, K-Mn liquid solution: SEM images of the wire after chemical and thermo-electrical preparation.**
- **Main results.**
- **Highlights and Final Remarks.**
- **Conclusions.**

Motivation

Main motivations were:

a) **Improve the reproducibility of results: we realized (since 2014) that some Fe impurities into the main material, and K at low concentration, were crucial to get positive results.**

b) **Increase the amount of Anomalous Heat Effect (*AHE*) previously (since 2011) detected by our group using a Constantan alloy ($Cu_{55}Ni_{44}Mn_1$, Cst; made by Isabellenhutte-D).**

- ***AHE* mainly due to the interaction of Hydrogen (H_2) or Deuterium (D_2) gas absorbed and/or adsorbed by the Cst itself and/or on proper host sites, by enhancing effect of nanostructures, under external *non-equilibrium conditions*, even local, of any type: electric; thermal; H, or D, gradient concentration; magnetic; laser (Cravens&Letts -USA; Holmlid and Olafsson-Se; others); radiation; ultrasound; etc.**

We remark that the AHE origin/process, with Cst, is the same as using Pd. Cst has the advantages of lower cost and is more robust: months of experiments cycling H<->L temperatures. Drawback is the "easiness" to sintering nanoparticles in vacuum/inert gas.
Cst selected because has the highest values of catalytic values (about 3 eV) for H_2 (or D_2) dissociation from molecular to atomic state (H_2->2H).

Cst were used in the shape of long (l=100 cm) and thin (Φ=0.2mm) wires, weight=278 mg. Surface made sub-micrometric (some particles have dimension even less than 100 nm, see SEM) by proper, high peak power, electric-thermal treatments (typical energy, given several hundred times by pulses with typical 50 ms duration, was 600-1000 J/g of CNM).
In previous, *successful experiments* we observed that, after prolonged cycling at high-low temperatures, under H_2 gas, the SEM of the wires demonstrate that there are fracturing phenomena due to Hydrogen embrittlement: *the typical dimensionality of particles was reduced in respect to the beginning of the experiment*.

Because such type of powerful, fast rise/fall times (<1μs) and short overall duration pulses, the wires get red color (about 800-900°C), and oxidized, in some ms of time and fast quenched at the end of pulse. The oxidation conditions, in free air, end at about 600°C.

The procedure developed by us remembers the, highly sophisticated, *melt-spinning and quenching procedure* adopted/optimized by Prof. Yoshiaki Arata (Osaka University) and Collaborators (Tohoku University) to produce nano-powder of Pd and Pd-Zr in the field of *Solid State Fusion* *(the name given by Prof. Arata to LENR)*, starting since 1995. We point out that we were inspired by such procedure and properly modified it for the use with wires.

Description of experimental set-up: some key details

- **The experimental set-up is basically similar to that developed and published since 2011.**
- **Furthermore, were made *several changing, specially about wire geometry (i.e. several knots) and new type of chemical impregnation of borosilicate glassy sheaths*.**
- **We will explain only the set-up of latest experiment (started on 20/05/2016; still now operative).**
- **In the experiment described we used only D_2, not H_2, even for the, home-made, preparation/mixing of chemical reagents.**
- **Practically, there is some residual quantity of Hydrogen.**
- **From isotopic point of view, will be present, at different concentrations: H_2, HD, D_2.**

The overall system is based on:

a) Tick wall (3mm) borosilicate glass tube, l=25cm, Φ=32-40mm. Gas pressure conditions: vacuum, pressurized. The value of maximum pressure depends on glass temperature: usually <3bar at the glass tube wall temperature of 300 °C (safety reasons);

b) Pt wire (usually l=100 cm; Φ=100 μm). In this experiment l=102.5 cm. Pt has multi-purposes use: a) power calibration of the system; b) to heat, indirectly, the Cst wires; c) evaluation of mean wire temperature (i.e. Pt used as thermometer).

c) 2Cst wires. Φ =200 μm. One with 41 knots, another with 71 knots nominal. The initial length (without knots) were 113 and 127 cm. After preparation of knots the apparent length reduced to 102.5 and 108.5 cm. During assembly, the lengths were equalized to 102.5 cm: knots were reduced from 71 to 65 (because previous convention in our logbook,, we kept the name identification as Cst71).

d) Usually one wire is polarized and the other is left unconnected and used to measure the so-called "spontaneous voltage" (by a high resolution multimeter Fluke 187) or current under 2 Ohm load of the same multimeter (mA range). The current is limited only by the internal resistance of the wire (about 17-21 Ohm). The resistance decreases when Deuterium is absorbed, similarly to previous experiment with Hydrogen.

e) Each wire is inserted inside a *borosilicate glass multi-filamentary flexible sheath (made by SIGI&Favier-I&F)* with Φ=1mm. Each filament has a diameter of 5μm. The weight of 1mm diameter sheath, after burning the oil used for texture of fibers by SIGI, is 1.9135 g/m. Each fiber is immersed into $Sr(NO_3)_2$ solution (home-made: starting from decomposition of $SrCO_3$ by 65% HNO_3 and diluted by D_2O). After drying/heating (to 400°C) and. partial dissociation of SrNO3 to SrO (a low working function material for electron emission, similar to CaO of Iwamura), the weight increase of typically 44 mg/meter. *We think that the glassy sheaths could have a key-role in the whole reaction.*

f) *NEW PROCEDURE*. After inserting the 3 wires inside the fibers, they are immersed in a solution of $Fe(NO_3)_2$+$KMnO_4$ (*be careful!!!*). Both are home-made using D_2O. Atomic ratio of Fe:K-Mn is 10:1. The K is used as "promoter" of activation for the absorption of H(or D) inside Fe lattice at high temperatures (>500°C). *The Mn is used to reduce the "evaporation" of K, keeping the reactivity of Fe-K quite constant over time.*

g) In separated tests we measured an increase of 10-20 mg/m of weight of sheaths due to $Fe_{10}K_1Mn_1O_x$ deposited at fiber surface, after drying and partial decomposition and oxidation at 500°C. The glassy sheaths are mechanically stable up to about 650 °C.

h) After drying/heating at 500 °C, the glassy sheath is inserted inside into another Al_2O_3 sheath (Φ=2mm) able to withstand, without self-damaging, up to 1200 °C in continuous operations: avoid dispersion of glassy materials, FeKMn, Cst; electrical insulation.

i) The 3 sheaths were braided and inserted inside another, 4mm diameter, glassy sheath for mechanical stability and further thermal homogeneity.

j) At the end, they are “rolled” around the SS (Φ=4-2mm) tube central support. The SS tube, to reduce the problem of Sulfur content of AISI 304 was, several times, conditioned at 700°C and cleaned by acid etching and ultrasound.

k) K-type, SS covered, thermocouple is inserted inside the SS tube to measure the inner temperature of the reactor (Tss nomenclature in the experimental results section).

l) The 3 wires are interconnected to external ambient using a MACOR cylinder (Φ=7--24 mm), relatively heavy (about 10 g) inside the reactor. The MACOR cylinder isn’t in direct contact with main glass tube wall (internal diameter is 34mm).

m) We guess that such MACOR cylinder, among others effects, could act also as location for the recombination of $D+D \rightarrow D_2$ gas. *In deep discussions on such specific aspect and thermal effect are still now in progress.*

Details of wire preparation with knots

[Fig.1, 2, 3. Preparation steps 1—9]

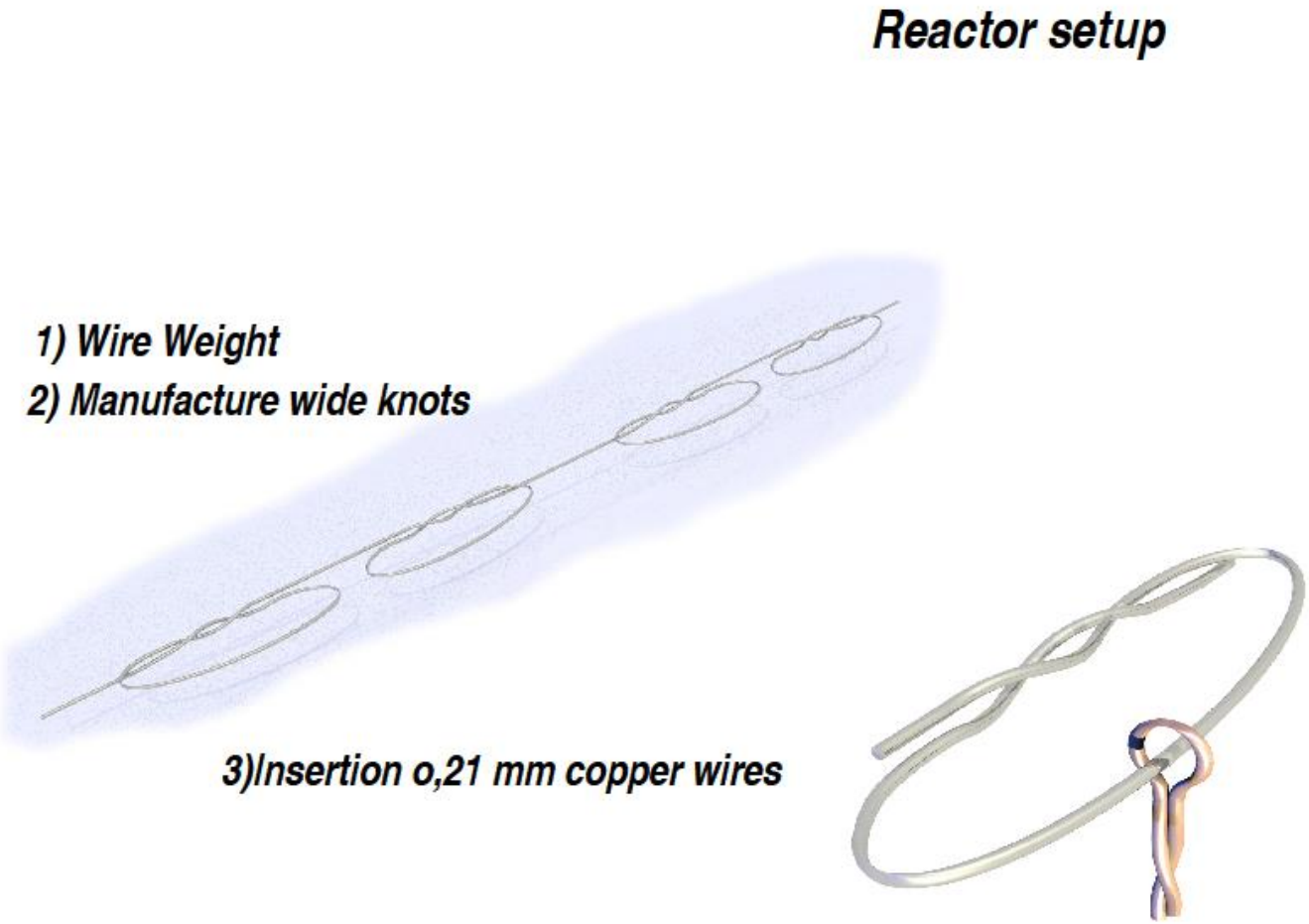


Fig.1. Wire preparation. The Φ=0.2mm Cu wire, step 3, is used to keep the knots hole limited at 0.2mm. To avoid to break the wire, the "fixing" is made every 4 knots: mechanical stresses reduced.

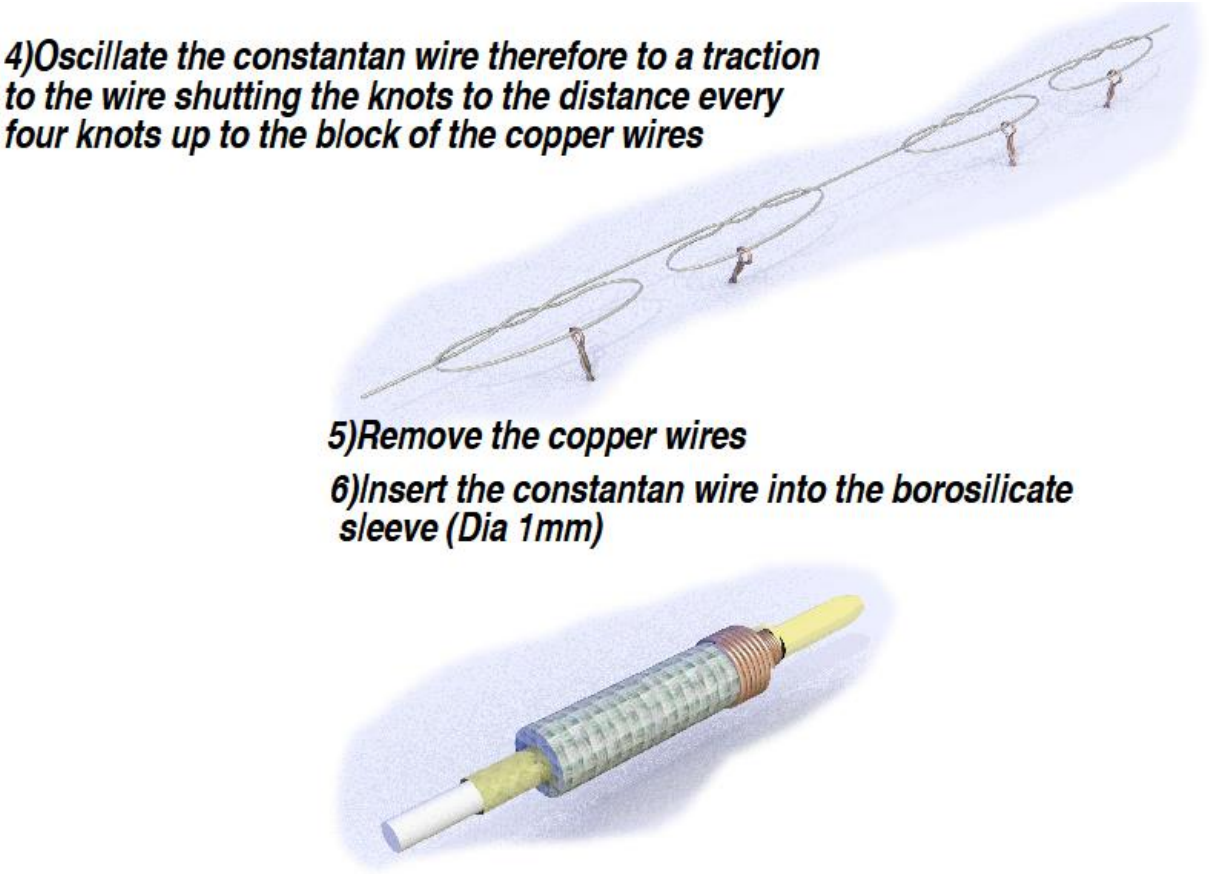


Fig.2. Between the step 5 and 6 the wire underwent the pulsing procedure to produce sub-micrometric materials at the surface. In addition, the holes are filled by the FeKMn solution, several times, and just later-on get oxidation pulsing: multilayers geometry, *tentatively similar to the Y. Iwamura plates used for transmutations experiments.* *Total increase of wire weight was 9-11 mg/m of Cst wire.*

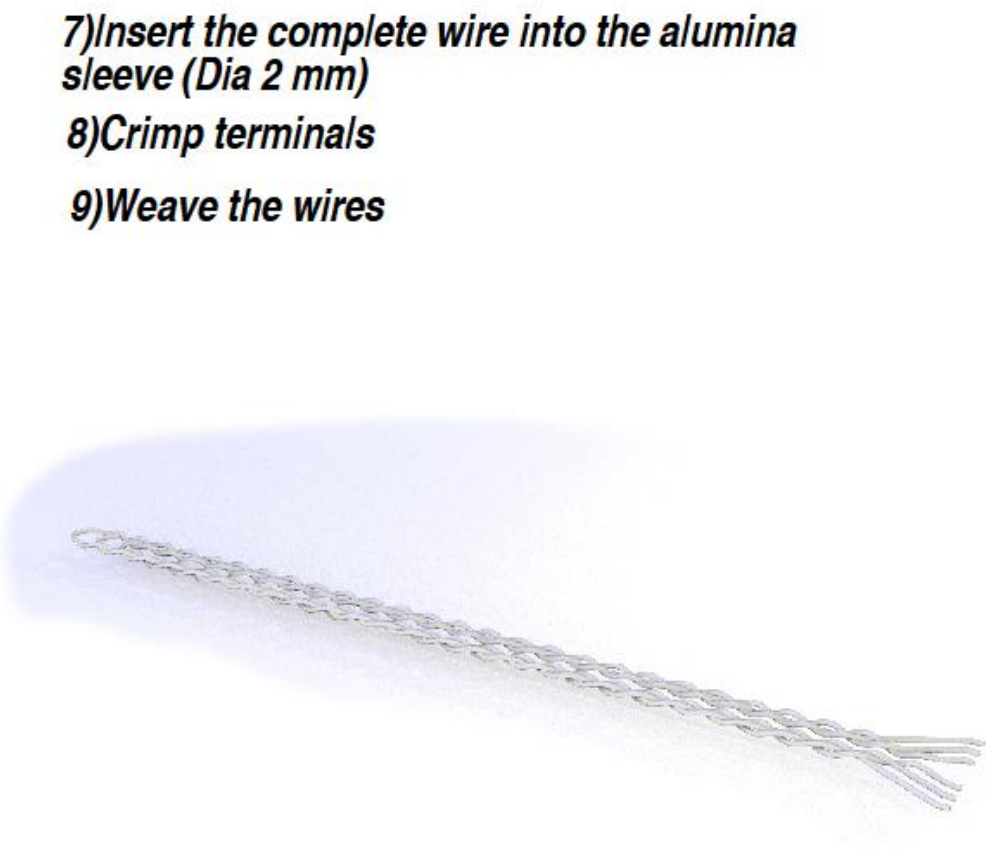


Fig.3 of wire preparation. The final step is the braiding of the 3 wires and further covering by a 4mm diameter glassy sheath (not shown).

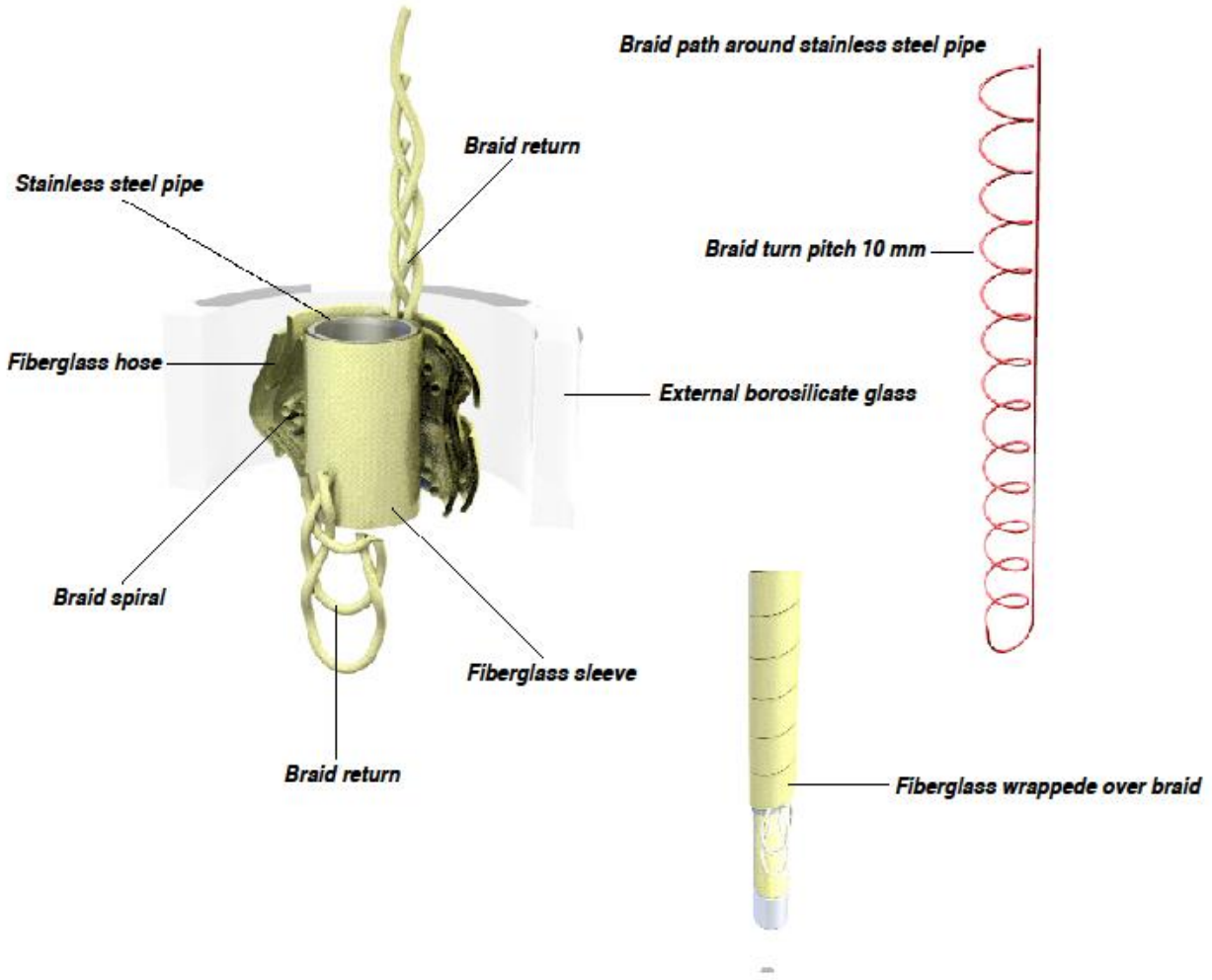
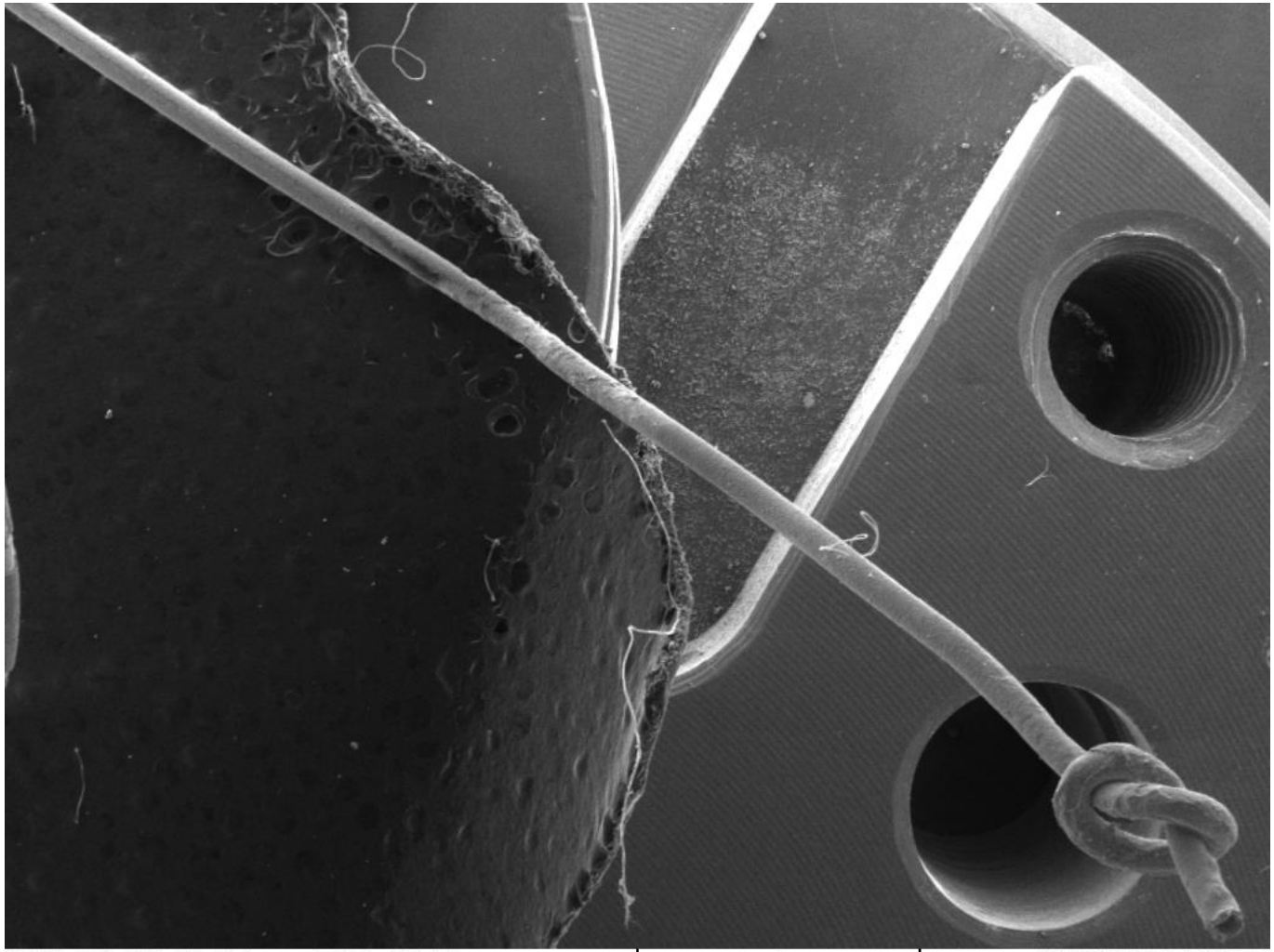


Fig.4. Wire assembling. Further details about the multilayer assembly of glassy sheaths.

SEM images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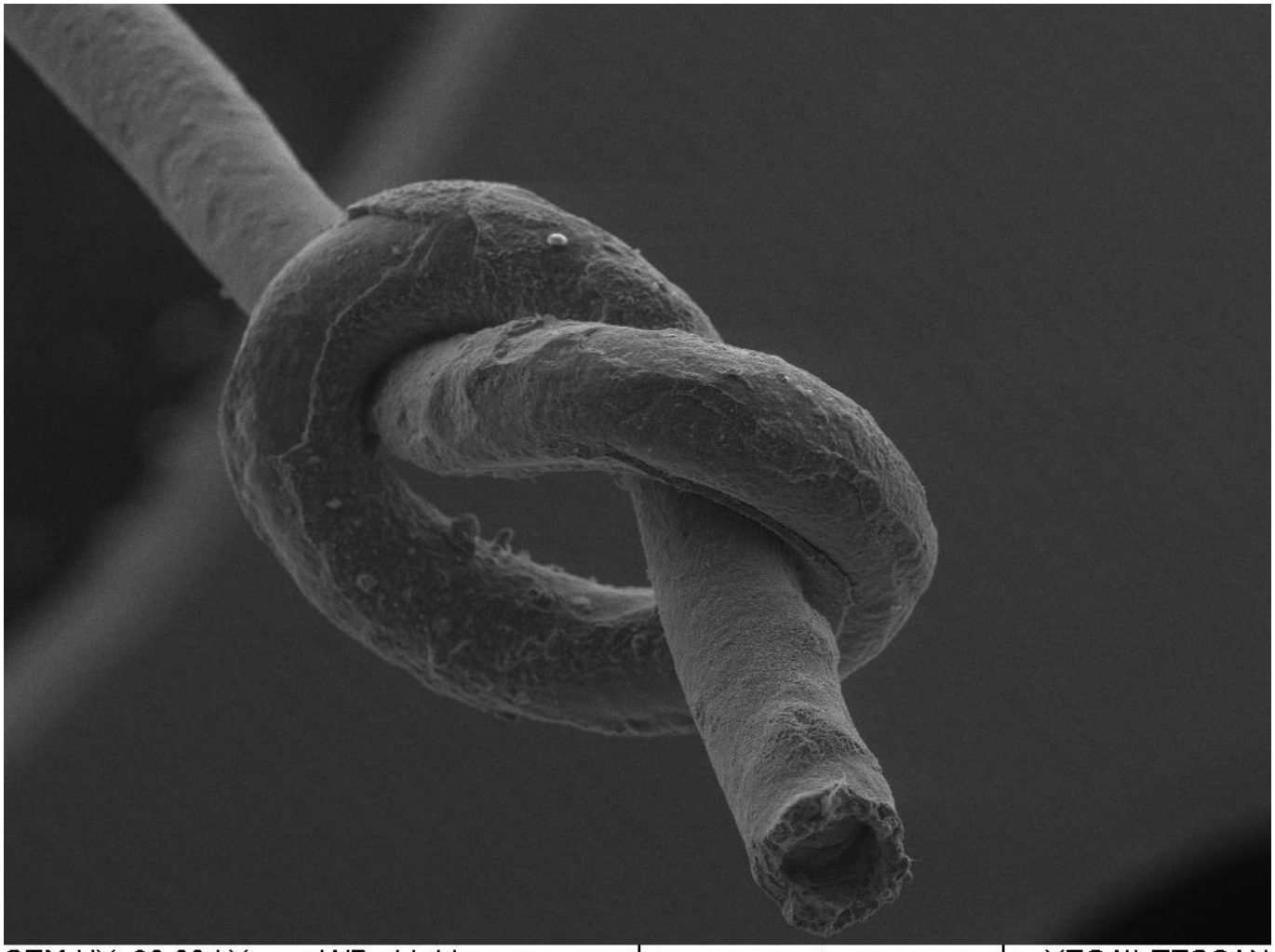
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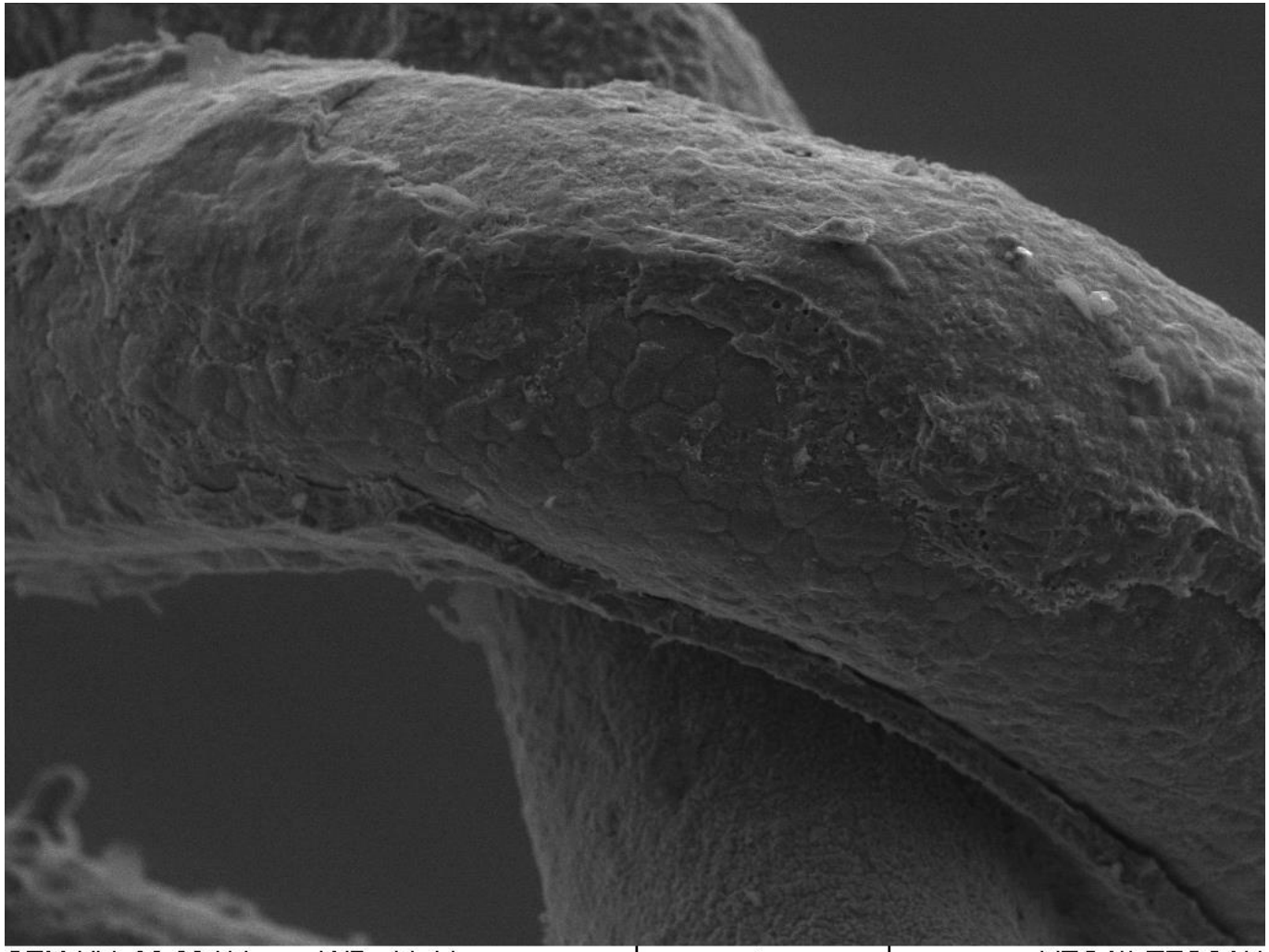
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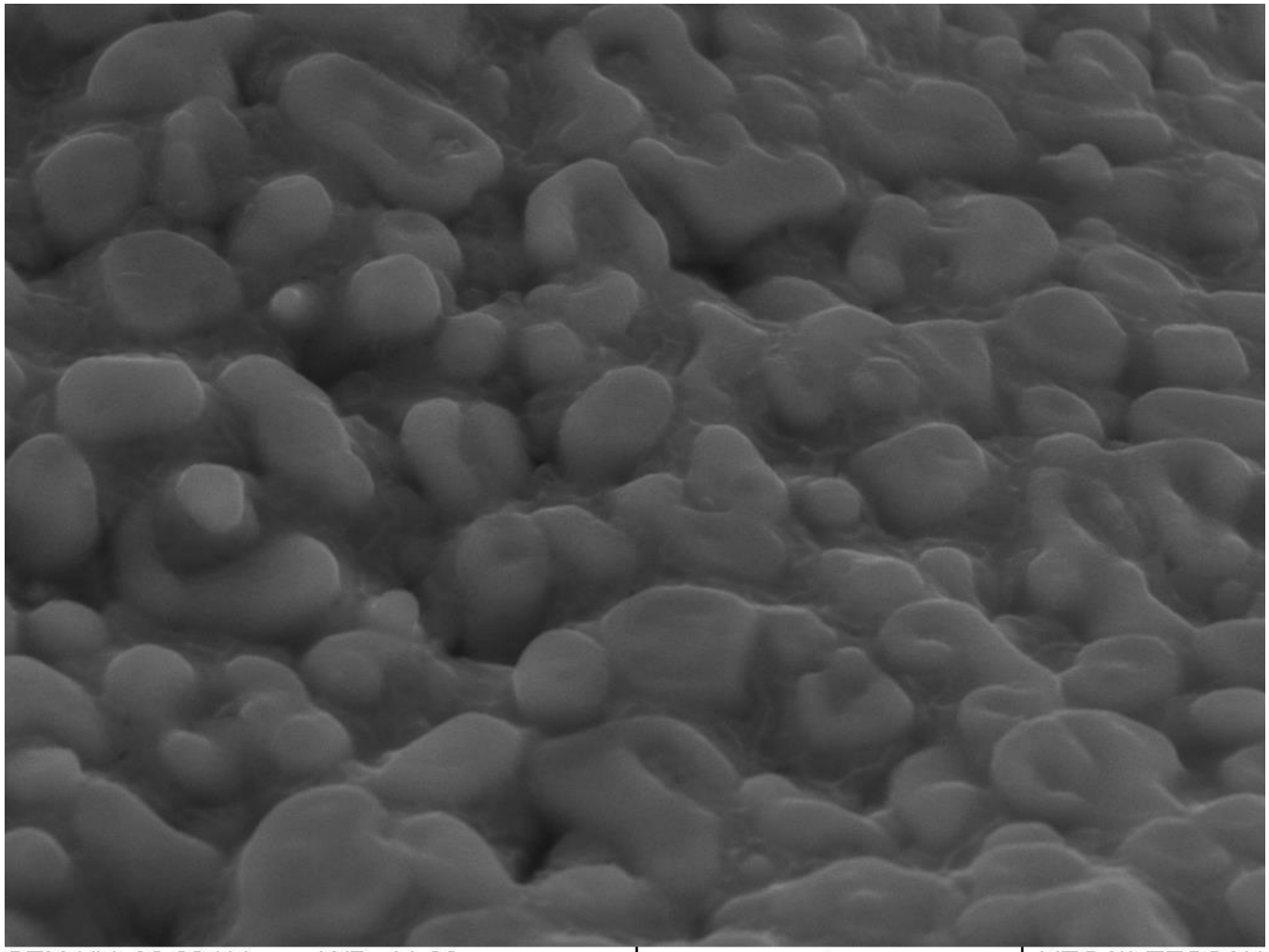
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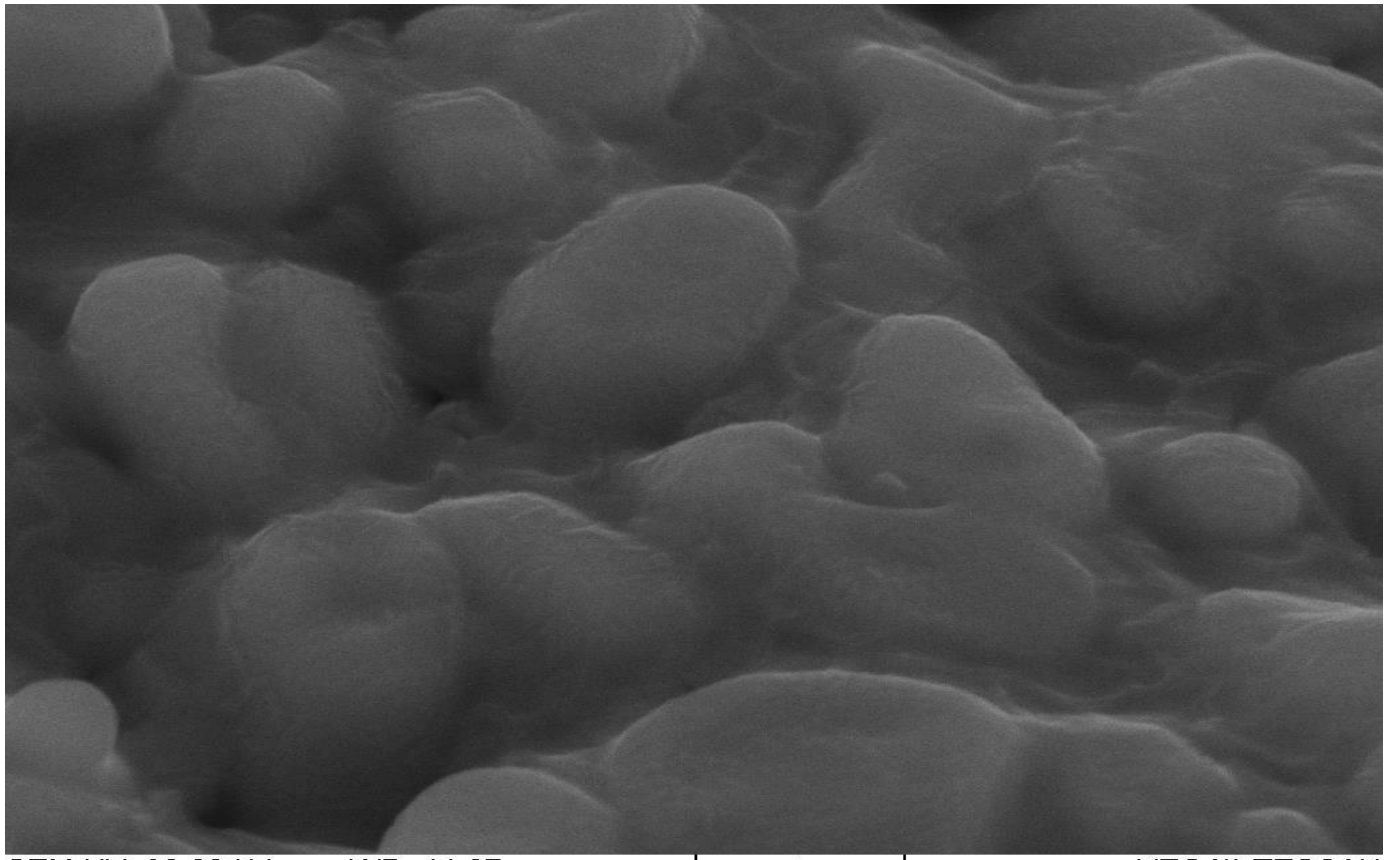
SEM HV: 30.00 kV
WD: 11.27 mm
VEGA\\ TESCAN
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Det: SE
2 µm
Vac: HiVac
Date(m/d/y): 05/25/16
NEXT - LNF - INFN

Main Results

The experiments, after calibration under vacuum and He gas with power applied ONLY to Pt (to avoid sintering of the nanoparticles at Cst surface due to high temperatures under vacuum or inert/noble gas atmosphere), were performed using D_2 at high (2 bar) and low (0.2 bar) pressures.

The combined effect of pure D_2 (a cracking agent to reduce dimensionality of surface nanomaterials) and several cycling Low→High→Low temperatures, further promotes the Deuterium absorption into the bulk of Cst: *usefull aging effects*.

In order to increase the wire temperature (beneficial for AHE) keeping as low as possible the external Pw applied, was added to pure D_2 the Xe gas (extremely low value of thermal conductivity) at 50% ratio.

Most of the results are reported with Xe-D_2 mixture, mainly at 0.2 bar total pressure.

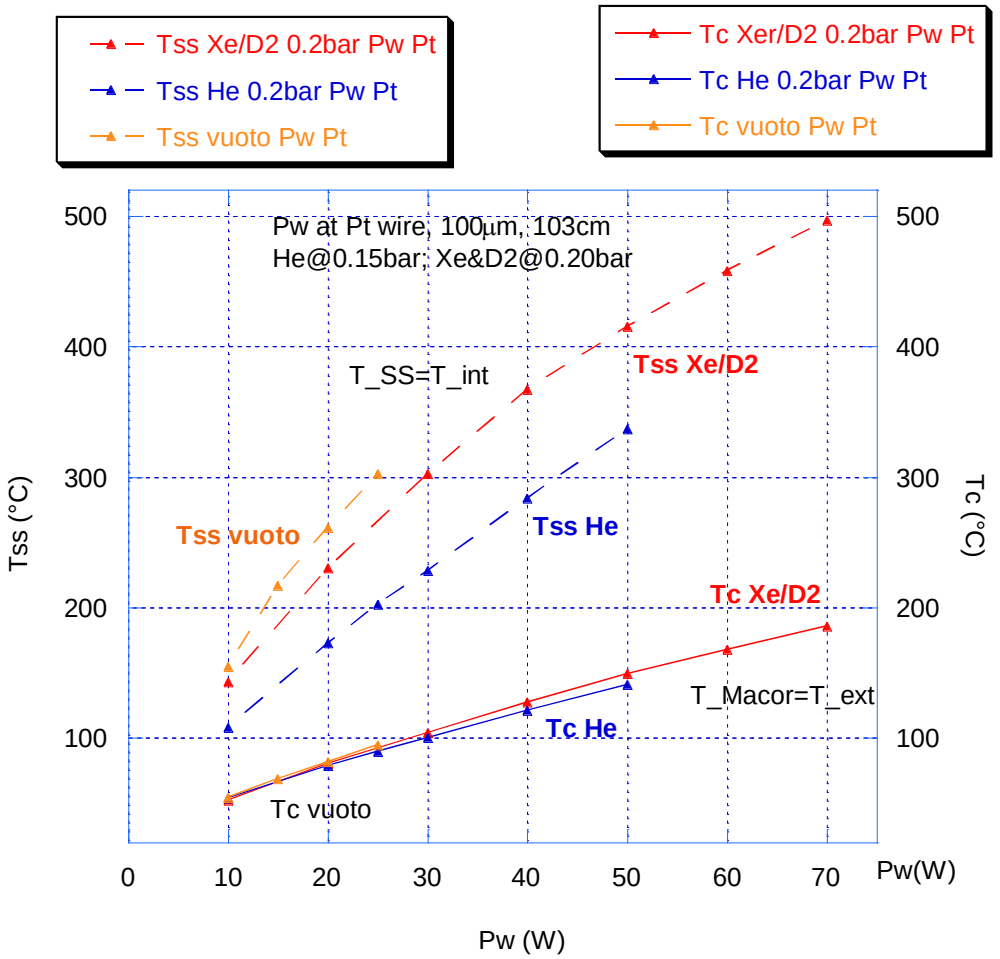
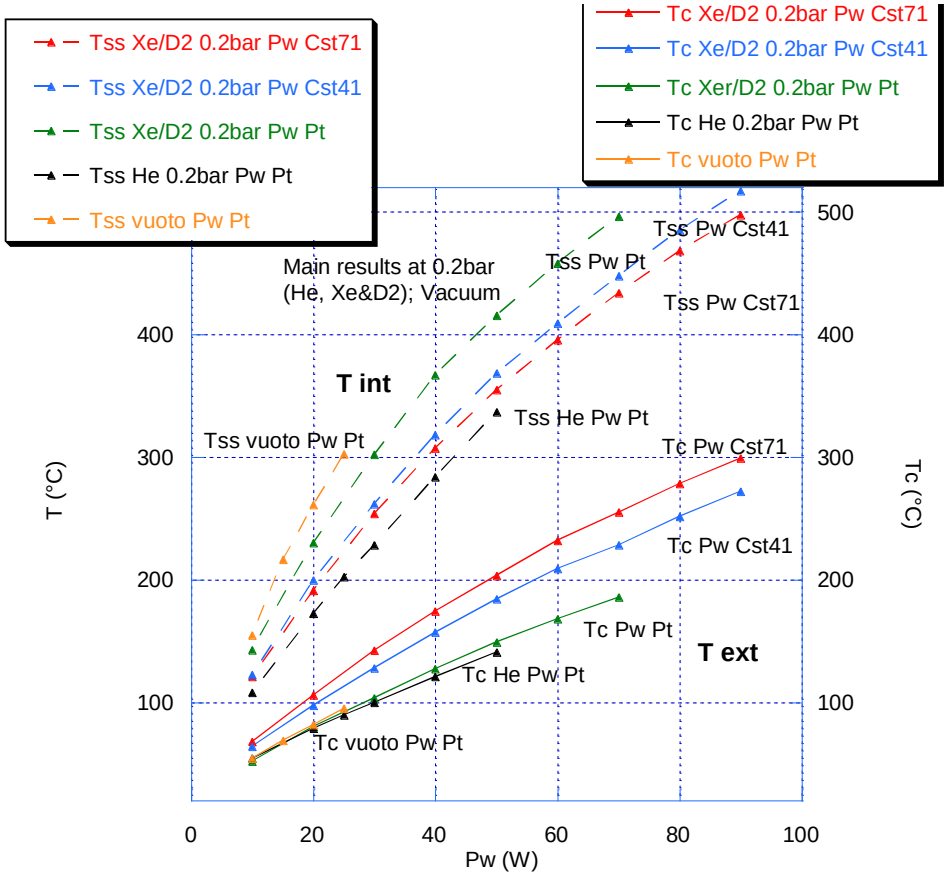


Fig.4. Pw at Pt wire. Calibrations by Vacuum and He. Both temperatures, internal (Tss) and external (Tc), increase with gas= Xe+D_2 (0.2bar). The extra temperature at constant Pw, with active gas D_2, could be induced by AHE on both CNM wires. The mixture of Xe+D_2 gas (50%-50%) has lower thermal conductivity than pure He.



. Fig. 5. Overview of the main experiments performed, using Vacuum (Pt), He (Pt), Xe+D_2 mixture (Pt, Cst41, Cst71). It is clearly discernable a large increase of external temperatures, in respect to Pt, with Pw applied at Cst41 and Cst71 (best result).

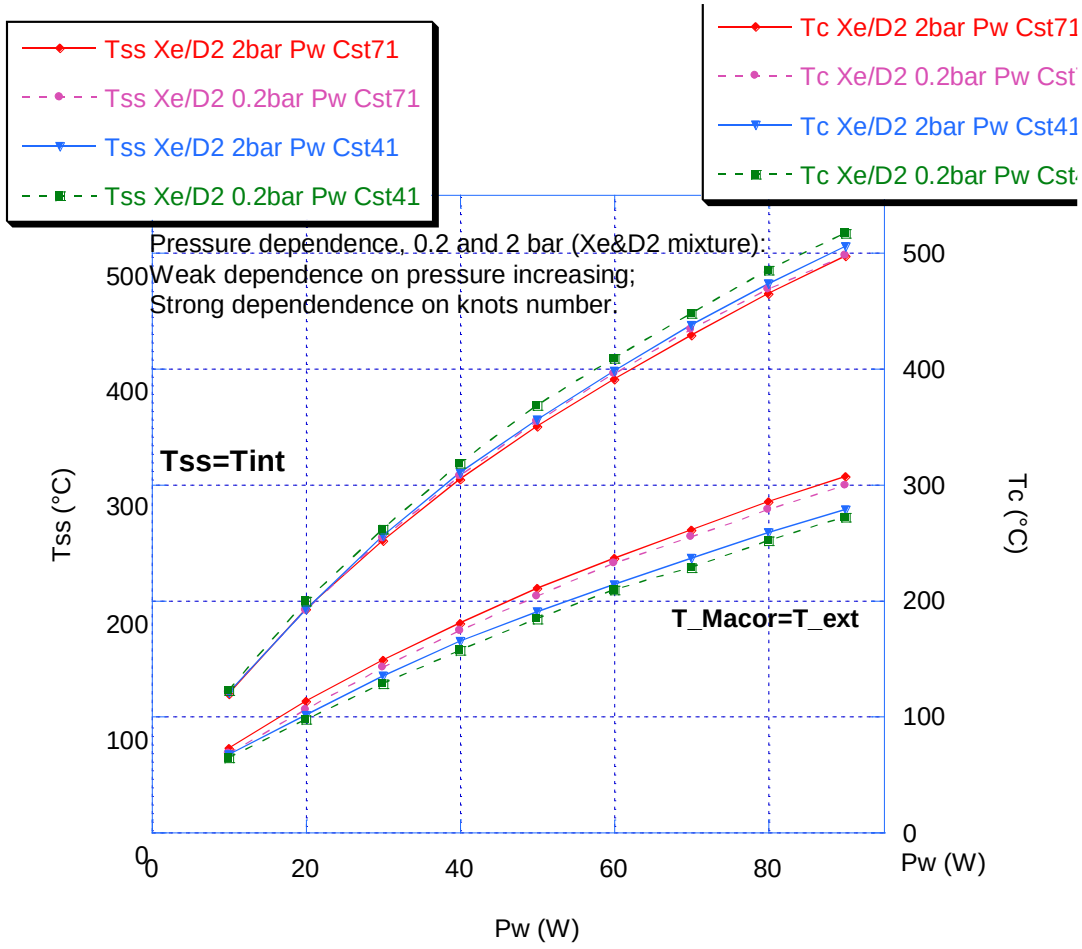


Fig. 6. Dependence of internal and external temperatures on gas pressure of Xe+D_2 mixture: 0.2 and 2 bar. The effect is quite low: the main temperatures changes, at constant input power, depend on the number of knots, not on pressure.

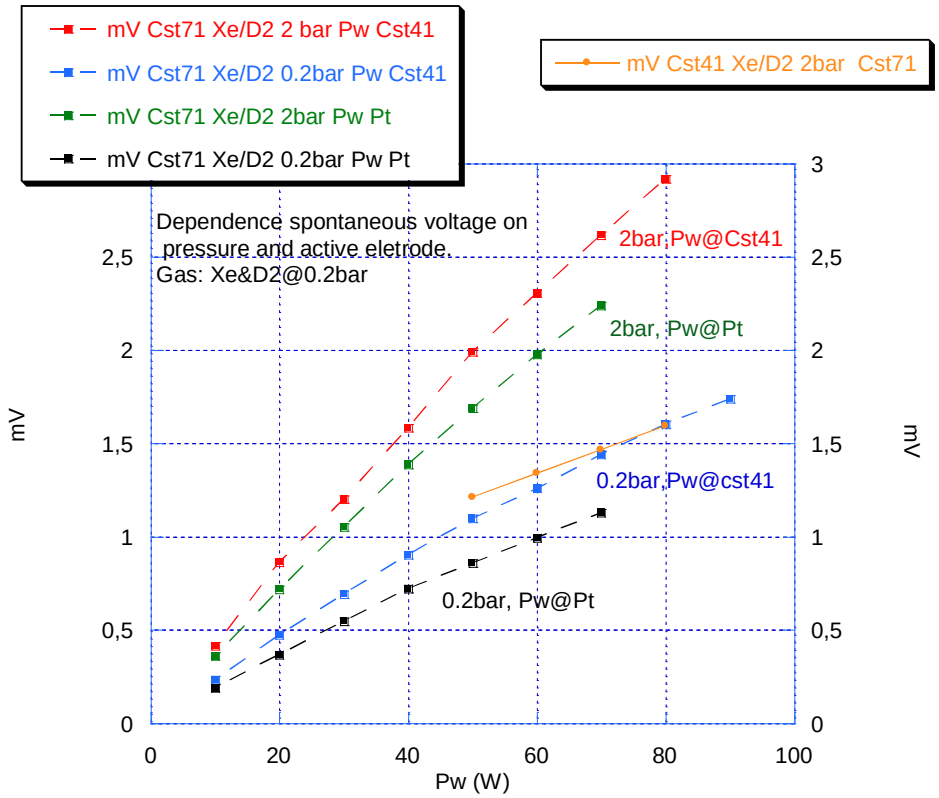


Fig.7. Spontaneous voltages (on 10M Ohm load) measured at the ends of Cst71 wire, NOT connected. The current produced (at 2 Ohm load) depends only on the internal resistance of the wire (around 18 Ohm).The value depends, apart internal temperature, on gas pressure (increases at larger pressure) and on the "amount" of atomic D allowable. With Pw applied to Cst41 (a guessed good catalytic material) the results are larger in respect to Pt.

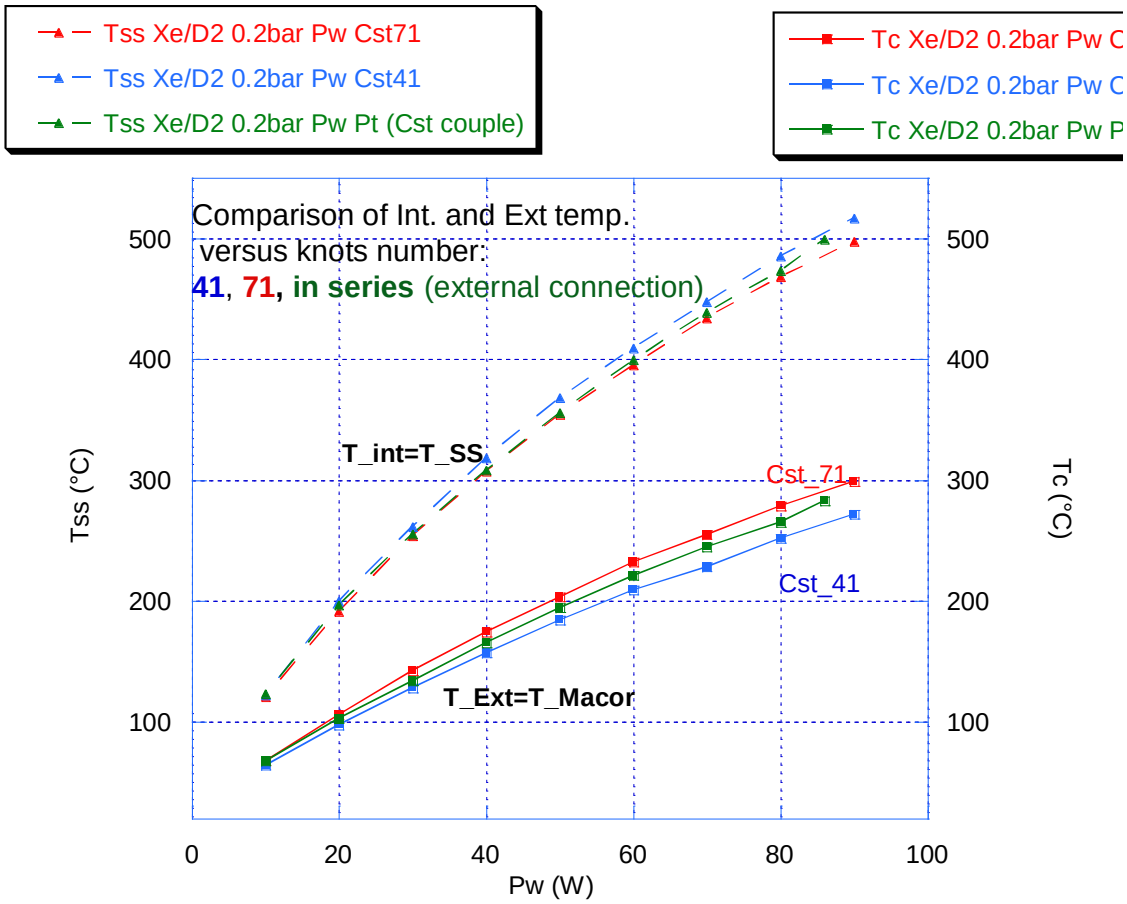


Fig.8. Experiment were the 2 Cst wires were connected in series. It can be argued that the AHE, at constant Pw, is NOT strongly related to the wires internal temperatures (individually lower because lower voltage drop) due to the current flowing, but to the number of knots.

Highlights

- **The addition of Fe-Mn-K mixed oxides, to our experimental setup, has shown a significant increase of the AHE, in respect to our previous similar experiments.**

- **Data point toward a complex of phenomena mediated by atomic hydrogen/deuterium formation, transport and recombination despite pressure and temperature (unfavorable for atomic hydrogen).**

- **In facts the energy gain seems to strongly correlate with the process of dissociation and recombination of atomic hydrogen/deuterium.**

- **We also have early indication that the migration of the active specie is enhanced by current and voltage in the Constantan.**

- **On the base of our observations we propose a simplified model of the experiment where atomic hydrogen/deuterium is firstly formed by exothermic adsorptive dissociation on the surface of Constantan (Ni/Cu), migrates through the Fe-Mn-K impregnated fiberglass sheath and exothermically recombines on MACOR ceramic.**

- **The energy for sustaining this process comes in form of current on Constantan (via Joule heating and a possible electrochemical factor) and from a *yet unknown process, possibly atomic/nuclear in nature and justifying the excess heat.***

- **Future work will be dedicated to identify and better understand the active sites of energy generation.**

- **At the same time special care will be given to exclude any “electrochemical” Peltier-like effect capable of mimicking certain of the reported thermal anomalies.**

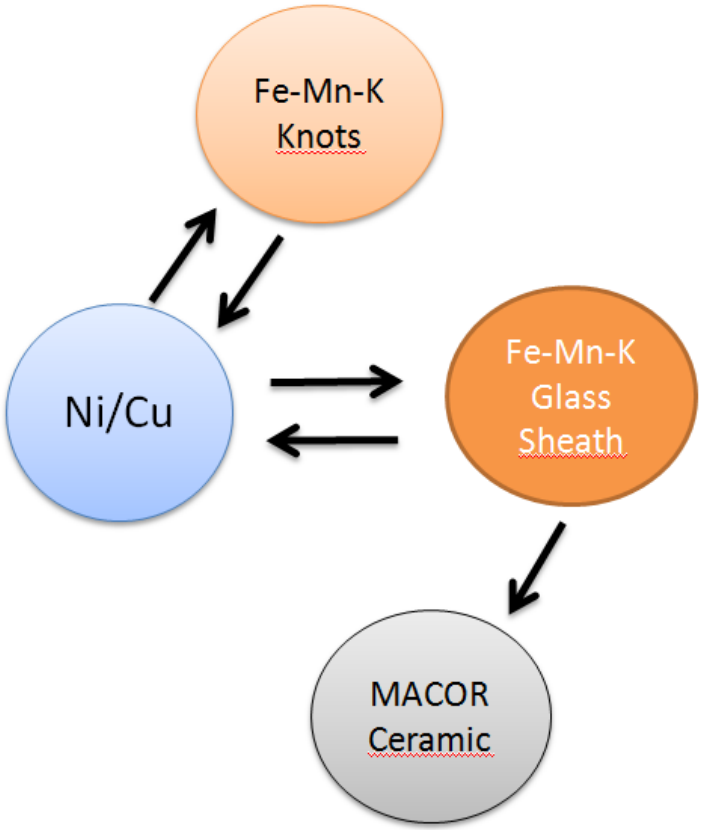
Final Remarks

- **Transport of atomic hydrogen may possibly occur from dissociation to recombination sites.**

- **These transport phenomena appears to be enhanced by current in Constantan, hence calling for a possible intervention of charged hydrogen/deuterium species.**

- **Dissociation/transport/recombination makes difficult to identify sites where excess heat occurs.**

- **A minor risk for some sort of Peltier-like effect mimicking certain observations cannot be yet excluded.**



Possible Exchange of atomic hydrogen in current experiment

Conclusions

1) One of the weakest point of LENR studies in general, i.e. not at all satisfactory reproducibility, in our specific experimental situation seems to be overcome by the addition of some Fe-K to our Constantan wires and glassy sheats (saturated by SrO, its role is the emission of electrons even at low tempertures, i.e.600 °C).

2) The effects seems related also to the concentration (or iper-concentration according to some Authors, like Leif Holmlid and Svein Olafsson) of Hydrogen or Deuterium in specific sites,Fe included (absorb H only at High temperatures, >500°C), once that the H_2 or D_2 were dissociated in situ.

3) The role of glassy sheaths, due of its specific composition and geometry/porosity, seems to have an important role, similar to that of alumina “support” in the field of car cathaliser for exaust gas conditioning.

4) The observation of “strange” nuclear effects, even included a reduction of ambient radioactivity and/or the emission of some low energy photons (<250 keV), has to be still now fully elucidated. Anyway, such phenomena arise only after LONG time (several months) operations of the reactor and large/stable “loading”.

5) The effect of so-called “spontaneous voltage”, firstly observed by us, by chance, on June 2014, is real and, since the first observation, was increased of over one order of magnitude about power produced. It seems like a new type of TEG (Thermo Electric Generator) induced by Hydrogen or Deuterium in the bulk and (perhaps) at the surface of Cst wires: practical application???

6) **Further, MULTIDISCIPLINARY work is mandatory to exploit the full potentialities of (our) Cst and LENR in general. Several phenomena are really unexpected and are NOT experimental errors:**

Some of the new phenomena observed have the potentialities for a practical application.