Steps to identification of main parameters for AHE generation in sub-micrometric materials: measurements by isoperibolic and air-flow calorimetry.

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OUTLINE

A) Introduction and background: motivations of present work (*after ICCF20, 2016; IWAHLM-12, 2017*). One of the motivations (from practical point of view) is comparison between previous measurements, performed with isoperibolic procedures, that maximize NON-equilibrium situations, and flow calorimetry that minimizes them.

B) Details on preparations of Constantan wires:

- **Photo sequence of high power pulsing of wires with knots (by video camera);**
- **Volt-Current-Power-Time plot (by oscilloscope); evaluation of energy needed;**
- **SEM observations;**
- **EDX analysis.**

C) Air flow calorimeter with calibrator inside the "box". Some key results

D) Conclusions and next steps.

Acknowledgements

A) The experimental work object of this presentation was carried out mainly at INFN-LNF; some (key) trials were done at the premises of a LENR Laboratory located in a Metallurgical Company of North Eastern-Italy: they helped also about financial aspects (since 2011).

B) Innovative/unconventional types of glassy sheaths were designed and made by SIGI (Società Italiana di Guaine Isolanti) among a joint collaboration of our Group and the Metallurgical Company.

C) Previously selected experiments have also been attempted within Martin Fleischmann Memorial Project (i.e. M. Valat and B. Greeyner): Live Open Science approach.

D) I am indebted to Prof. Brian Josephson, Nobel Laureate in Physics (from long time involved on LENR field), because he wrote an "Open Letter" to also the Scientific Coordinators of INFN asking that I could continue the LENR Research in Frascati even after the age pension limit.

E) Starting from October 2017 an (important) political Group in Italy (North League), "pushed" the President of INFN to allow the continuation of my experiments in the Frascati Laboratory, although I reached the age-limit of retirement. The main people involved were: Francesco Malagoli, Filippo Panini, Paolo Varini (from Modena City). All of them followed the LENR studies since 2010, among their (key) political framework/project of environmental conservation and pollution reduction.

F) SEM and EDX analysis performed by Antonino Cataldo and Stefano Bellucci (NEXT collaboration) at INFN-LNF.

G) Expenses at ICCF21 partially supported by IFA organization (main topics are "Water and Energy")-Italy.

Introduction

- **A research program has been initiated in 2011 aiming to increase the magnitude and reproducibility of Anomalous Heat Effects (AHE), possibly using *low-cost materials*.**

- **This program focused on the study of Constantan, an alloy of Nickel and Copper ($Cu_{55}Ni_{44}Mn_1$, *ISOTAN*, shortened by us as *CNM or Cst*) instead of metals such as Pd(*1), Ti(*2), and Ni(*3) that have been deeply studied before. The CNM was developed by Edward Weston in 1887 (Germany) and patented its application in the field of electro-technology (very *constant* value of resistance with changing temperatures, from RT up to 500°C).**

- **This alloy, unusual in the field of LENR research, was selected at the beginning of 2011 after the hypothesis/guess of a *"hidden factor"* behind AHE successful generation in early experiments of A. Rossi (most likely this factor was not immediately recognized).**

- **At the best of our knowledge, such successful experiments occurred around 2007 while A. Rossi was collaborating with B. Ahern in the United States.**

- ***We would like to highlight that the (detailed) real reasons for the selection of Constantan in our experiments were presented in public, for the first time, at IWAHLM-12 (5--9 June 2017, Asti-Italy).*** **They were, in deep, discussed at such International Workshop (organized, since 1996, by B. Collis).**

- **Later-on several Scientific meetings on LENR field were devoted to analyze/discuss such arguments. Among others, the most important were held at:**

  A) the Aeronautic section of Italian Army (end of June 2017), Rome;

  B) the 70° Anniversary of ANDI (Associazione Italiana Degli Inventori, i.e. Italian Association Of Inventors) held on November 2017, Rome. Such Association was found (on 1947) under the agreement/sponsorship of the President of Italian Republic.

(*1) F. Paneth, 1926; M Fleischman-S. Pons, 1989.

(*2) S. Jones, F. Scaramuzzi, 1989.

(*3) F. Piantelli, rods bulk shaped, 1991; A. Rossi-B. Ahern as powders, about 2008.

Background: motivation for choice of Constantan. Deeper informations.

- **Since 2011 we made the hypothesis that the "real and main catalyst or initiator" of the reaction with gaseous Hydrogen was the *thermocouple* inserted within the Ni nano-powders, and not the Ni itself.**

- **We refer to the *J-type* (*Fe-Constantan*), particularly suitable for the temperature range of most experiments (<750 °C). This thermocouple is particularly convenient also because of its low cost and high sensitivity for temperature measurements (i.e. about 50 μV/°C). Some technological know-how about the J-type fabrication was provided by Researchers at TCDirect Company (Turin-Italy).**

- ***Furthermore, we had reason to believe that these thermocouples* could be partially damaged after several tests, leading to activation of its surface; we believe also that the insulating and protective material covering the thermocouple (a kind of glass) may contribute to the observed phenomena.**

- **Later that year (June 2011), we found an article (computer simulation, by S. Romanowski et al., 1999) predicting that Cu-Ni alloys, such as Constantan, may provide an extremely large energy for the catalytic dissociation of Hydrogen from molecular to the atomic state ($H_2 \rightarrow 2H$). The value of energy is as large as 2-3 eV, furthermore (as a gift) among a wide range of compositions (i.e. Cu/Ni ratio), according to Tab.1. Shortly, as comparison, the most used (but costly) material, i.e. Pd, provides for H_2 dissociation only 0.424 eV.**

Reprint from S. Romanowski (Tab. 1) shows predicted values for the energy of dissociation of Hydrogen molecule to atomic Hydrogen. Some of these data have also been confirmed experimentally/qualitatively by the same Authors.

Material composition	ΔE (eV) for Hydrogen dissociation ($H_2 \rightarrow 2H$)
$Ni_{0.375}\,Cu_{0.625}$	3.164
$Ni_{0.625}\,Cu_{0.375}$	2.861
$Ni_{0.8125}\,Cu_{0.1875}$	2.096
Ni	1.736
$Ni_{0.1825}\,Cu_{0.8175}$	1.568
$Ag_{0.8125}Pd_{0.1875}$	0.572
$Ag_{0.625}Pd_{0.375}$	0.560
$Ag_{0.325}Pd_{0.675}$	0.509
$Ag_{0.1875}Pd_{0.8125}$	0.509
Pd	0.424
Cu	-1.110
Ag	-1.416

Tab. 1. Adapted from S. Romanowski et al. (*Langmuir 15 (18), 5773-5780*)

Best catalyzer is $Ni_{0.375}\,Cu_{0.625}$ at +3.164 eV; worst is Ag at -1.416 eV.

- **A similar behavior can also be expected for Deuterium gas, and it was retroactively noticed in electrolytic experiments carried out by our group since 1989 (mainly because of contamination problems, considering that Cu was used as electrical contact, although protected by PTFE shrinking sheaths; Ni often adopted as "reference") , later-on gaseous.**

- **It is also worth to mention that in Summer 2012, Giorgio Vassallo (co-Author), brought to our attention patents filed in 1993 (published as US5770036, US5411654, US5674632) where B. Ahern is the first inventor. These patents reported that *Cu-Ni* could be a good candidate to obtain AHE, especially when at *nanometric dimensions* or prepared alternating layers of copper and nickel.**

- **This information made us more confident on the "Cu-Ni hypothesis" as a trigger of AHE phenomena. Furthermore, it encouraged the use and additional developments of our techniques to prepare a multilayer and nanostructured texture using electrical, short duration, pulses of very high peak power.**

The INFN-LNF main steps and related improvements.
Wires Pulsed/Flash Oxidation

- **To increase the surface area of *CNM wires*, we applied to them several hundred electric pulses (typically 50 ms duration, recently up to 70 ms) with a rather large peak power (15-20 kVA/g). This allows us to reach surface temperatures of 700-1000 °C in air, where *Constantan* starts to oxidize showing the formation of a spongy sub-micrometric texture.**

- **We think the cause is the fast rising in temperature followed by fast cooling (quenching), according to SEM. This also leads to some phase separation, with the formation of separated islands of nickel-rich or copper-rich phases: confirmed by EDX analysis.**

- **It is likely that a *skin effect* (pulse rise-time 1 μs) concentrates most of the current (power) at the surface of the wire, causing increasing temperatures and large gradients.**

- **Interestingly the wire surface shows, after pulsed oxidation, the *formation of mixed oxides of copper and nickel (*Cu_xO_y, Ni_wO_z), sometimes arranged in a multilayer and sub-micrometric structures.**

- **Highlight: pristine *CNM* is the substrate where sub-micrometric materials, of various composition, are supported. The material prepared per this procedure shows a reduced tendency to self-sintering. In other words, the inert material which is often added to reduce sintering problems (e.g. ZrO_2 chosen by Yoshiaki Arata-Japan, since 2002, for nano-Pd) is replaced with "active" structures that, among others, can possibly absorb some amounts of Hydrogen and eventually take part in the exothermic reactions.**

- **For the sake of clarity, we do not have a precise control of dimensionality of nanoparticles as in Arata's procedure. In facts, Arata obtains 2-15 nm nano-particles with Pd_35%-over ZrO_2_65%, using the (sophisticated) melt-spinning and quenching processes.**

- **Weak points. We have observed that the oxidized and partially reduced structures (by H_2 or D_2 interaction) on Constantan wires may detach and fall off at the bottom inside glass reactor (low temperatures). When such phenomenon occurs, the experiment outcome may be negatively affected. The disadvantage of detaching increases, sometimes dramatically, after H or D absorption and several thermal cycles (e.g. 20→400--700→20 °C).**

Importance of "non-equilibrium" and "knots"

- **In almost all our experiments (since 1989) we realized, and "advocated", that having Non-Equilibrium conditions is a key factor to induce, and possibly increase, any "anomaly" in the H/Metal systems, AHE effects included. On the base of such observations, in 2015 we introduced a new type of wire geometry, aiming to increase local thermal (and possibly concentration) gradients, without adding extra power from outside.**

- **In other words, we developed simple procedures to get thermal gradients along the wire, thanks also to fiber glass sheaths that, for several aspects, are step-discontinuity to heat transfer from the wire to local gaseous environment. After various attempts, we realized that the simplest approach was to introduce several knots in the *CNM* thin wire. This effect is increased further if the wire has a current flowing in it, as usual in our experiments, i.e. large voltage drop along the wire to get *NEMCA Effect* (C. Vayenas, 1996).**

- **The procedure worked very well and first results were presented at ICCF20 (Sendai-J, October 2016). Moreover, we realized that the AHE value is positively correlated with the number of knots. *The optimal distance between knots is still under investigation.***

Reactor assembly, including knots preparation (presented at ICCF20, pub. on JCMNS, Vol 27).

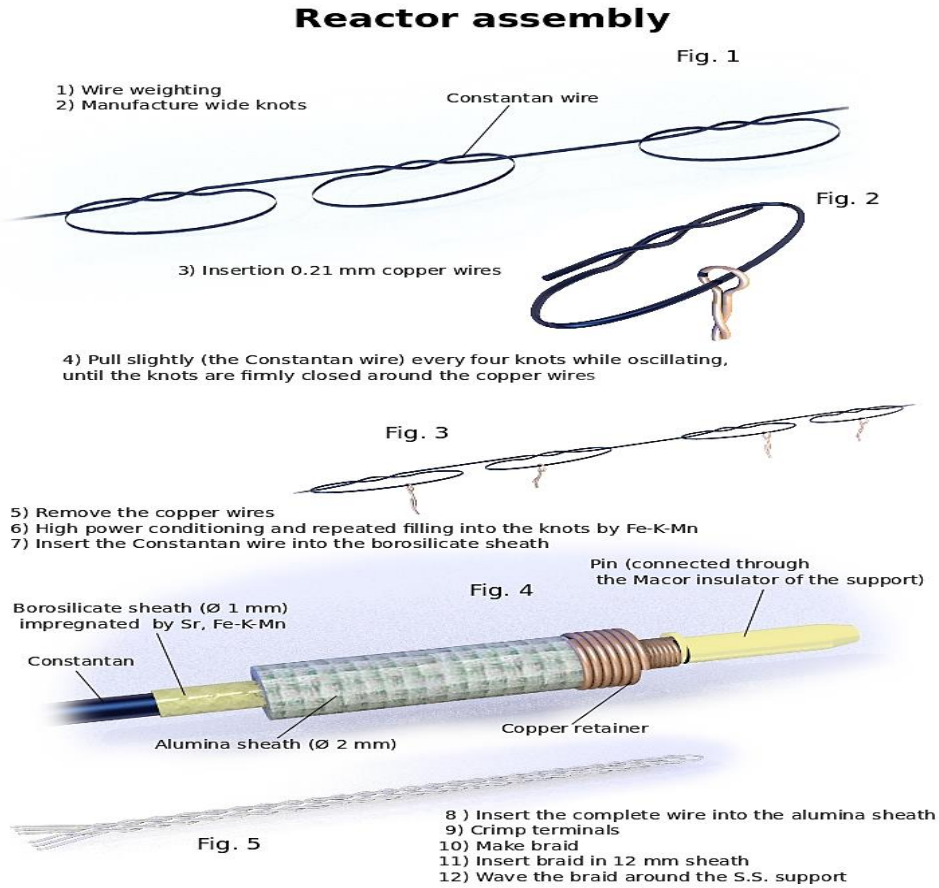

***Figure R1**: Preparation of the knots in the Constantan wire. **Figure R2**: Insertion of the copper wires in order not to completely tighten the knot. **Figure R3**: Critical point of tightening the node due to possible stress of the Constantan wire. **Figure R4**: After conditioning, insertion of the wires inside the several glass sheaths. **Figure R5**: Braid of the three wires to be waved around the SS support.*

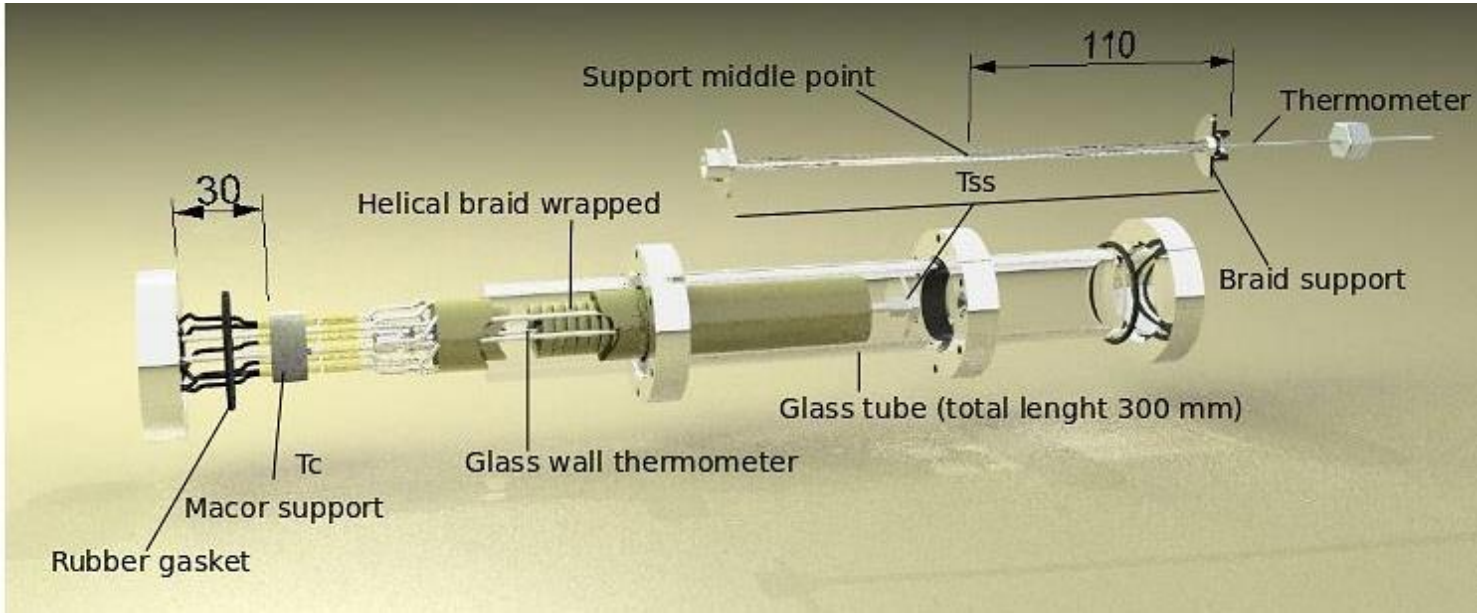

Fig.2 Overview of the reactor once assembled. Regarding temperatures, Tc is external, Tss is internal to the reactor core.

Video (time frame about 20ms, pulse duration 70ms), of HPP (High Peak Power pulses), 200 micron diameter wire with knots, each every 2 cm.

* Diameter of holes inside the knots <200 μm, for 200 μm wire diameter.

- Using the 100 μm diameter wire the holes diameter are less defined, ranging from almost 0 micron up to 100 μm.

The HPP pulser is based on the well-known capacitive discharge method. One of its peculiarity is the quite large value of (multiple) capacitor bank used (6600 μF, parallel of ceramics, polyesters, pulse-operated electrolytic), relatively large repetition rate (0.1Hz), fast rise (<1 μs) and fall time. The electronic switch is based on 4 Power MOS in parallel (up to 100 A of current capability).


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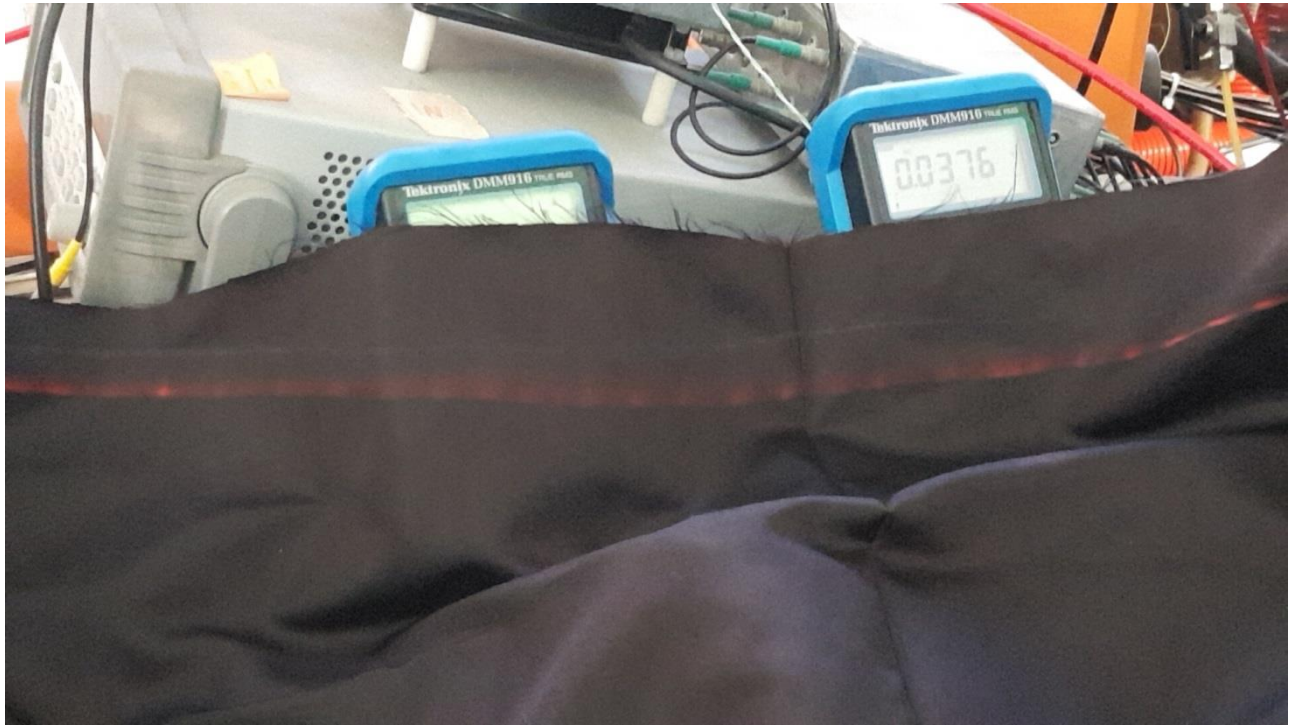
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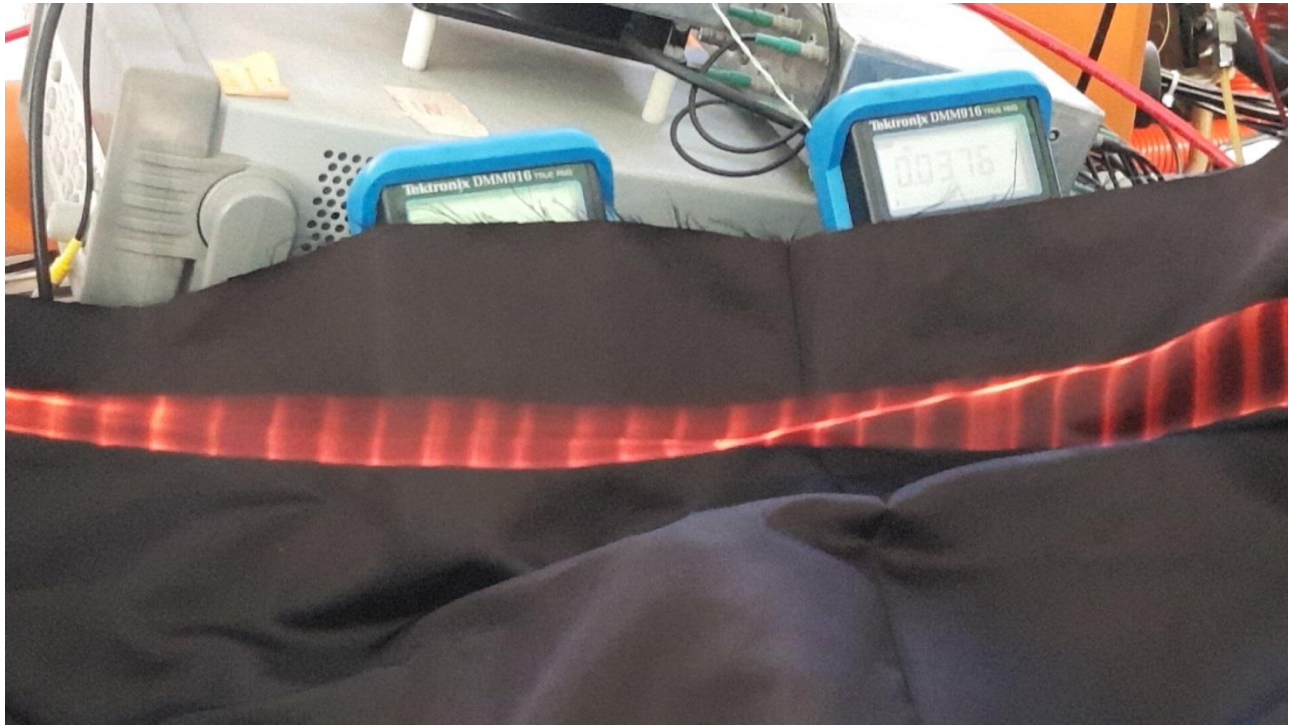
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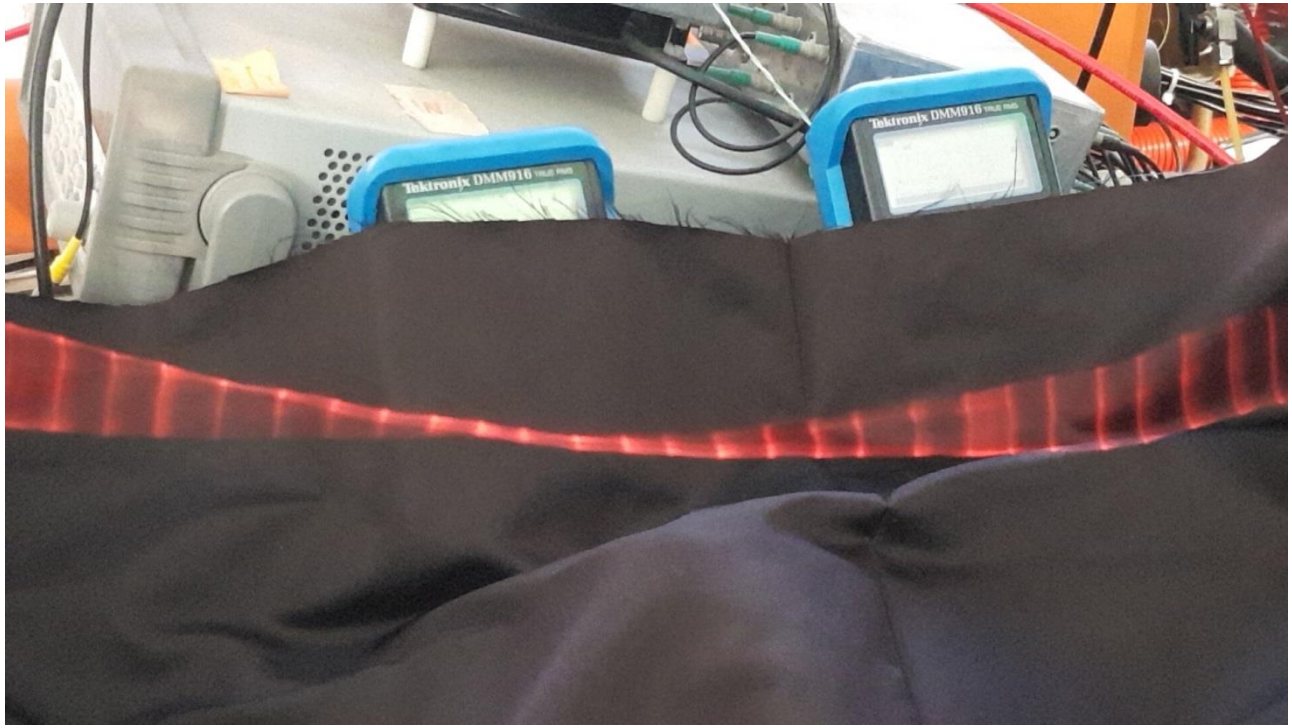
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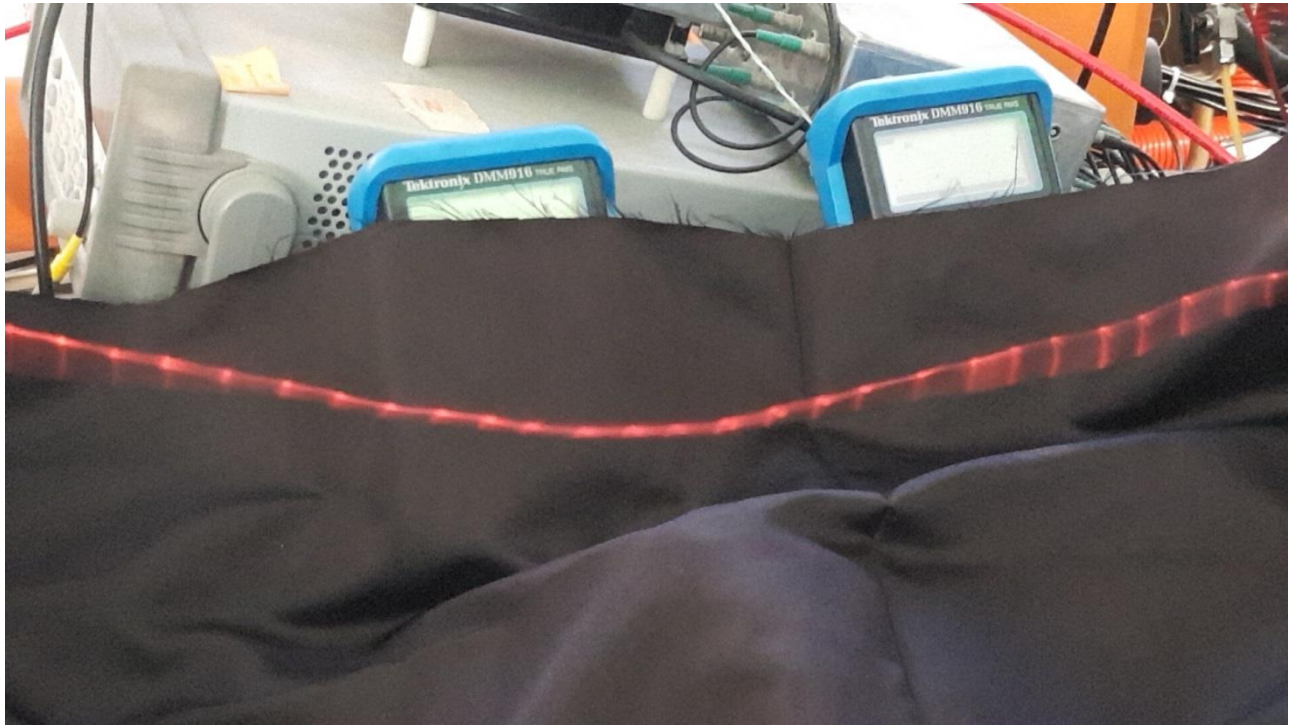
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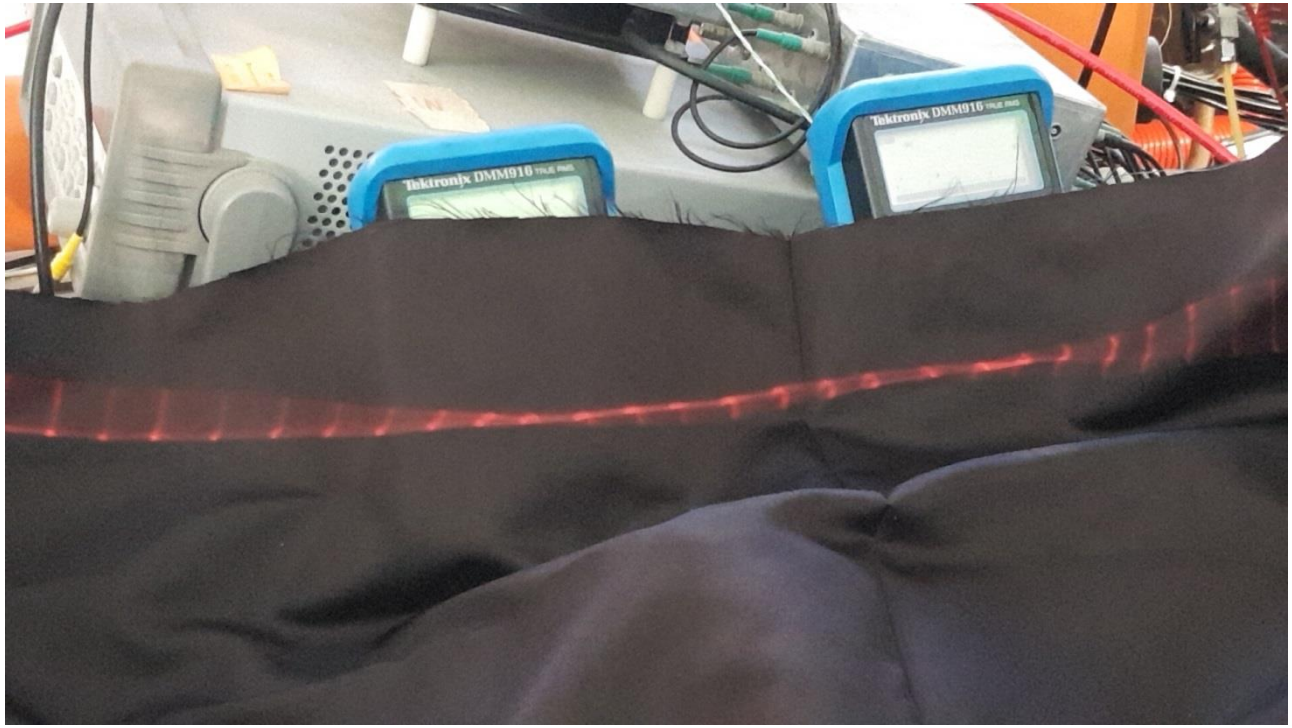
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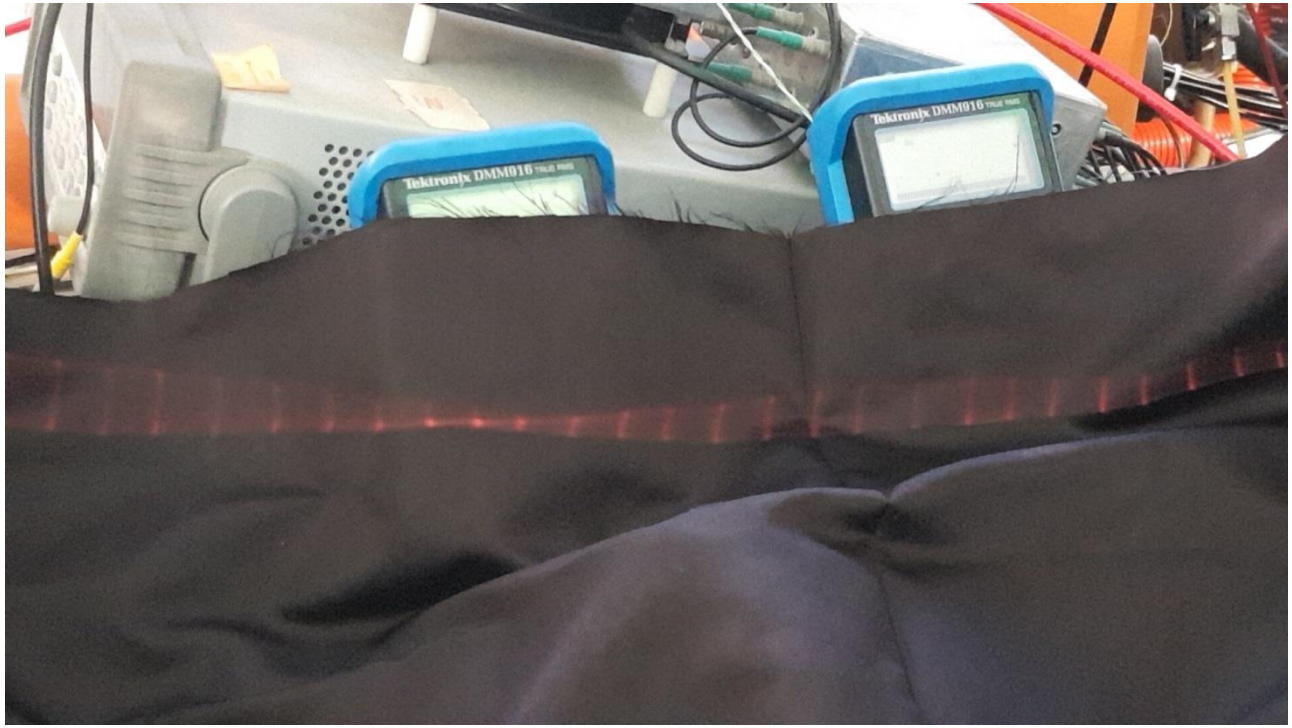
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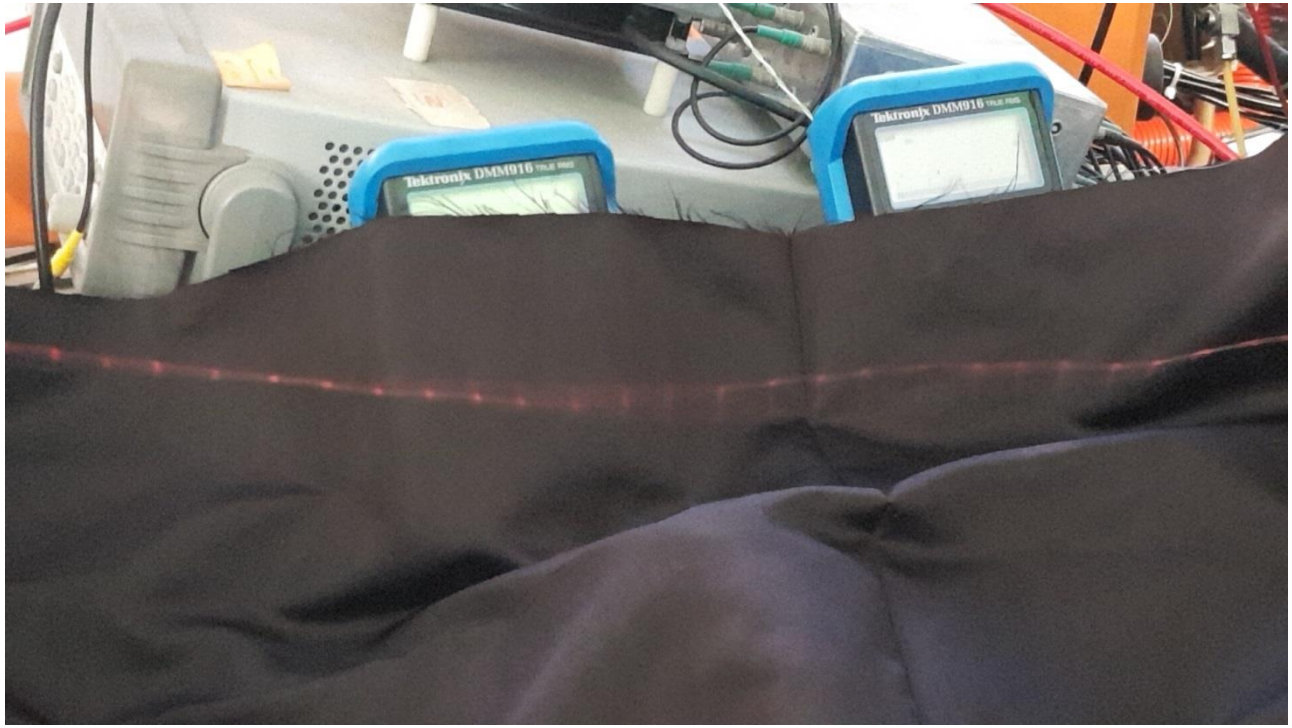
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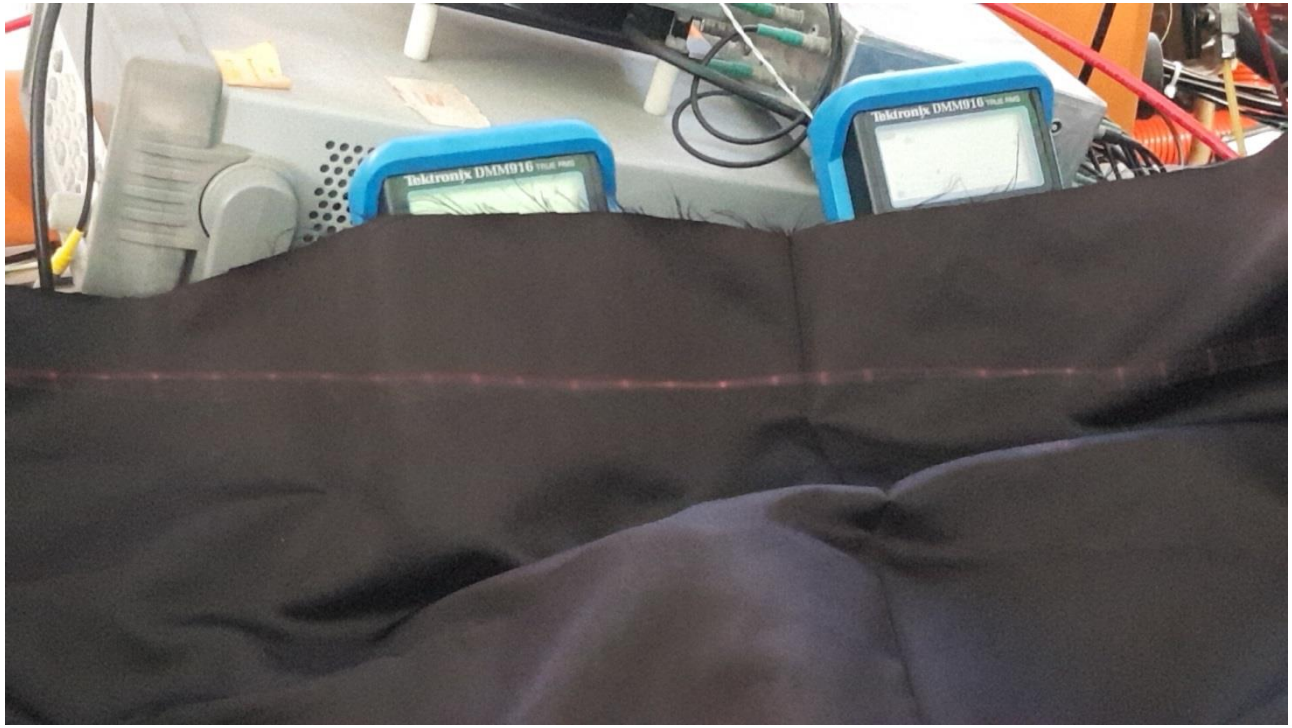
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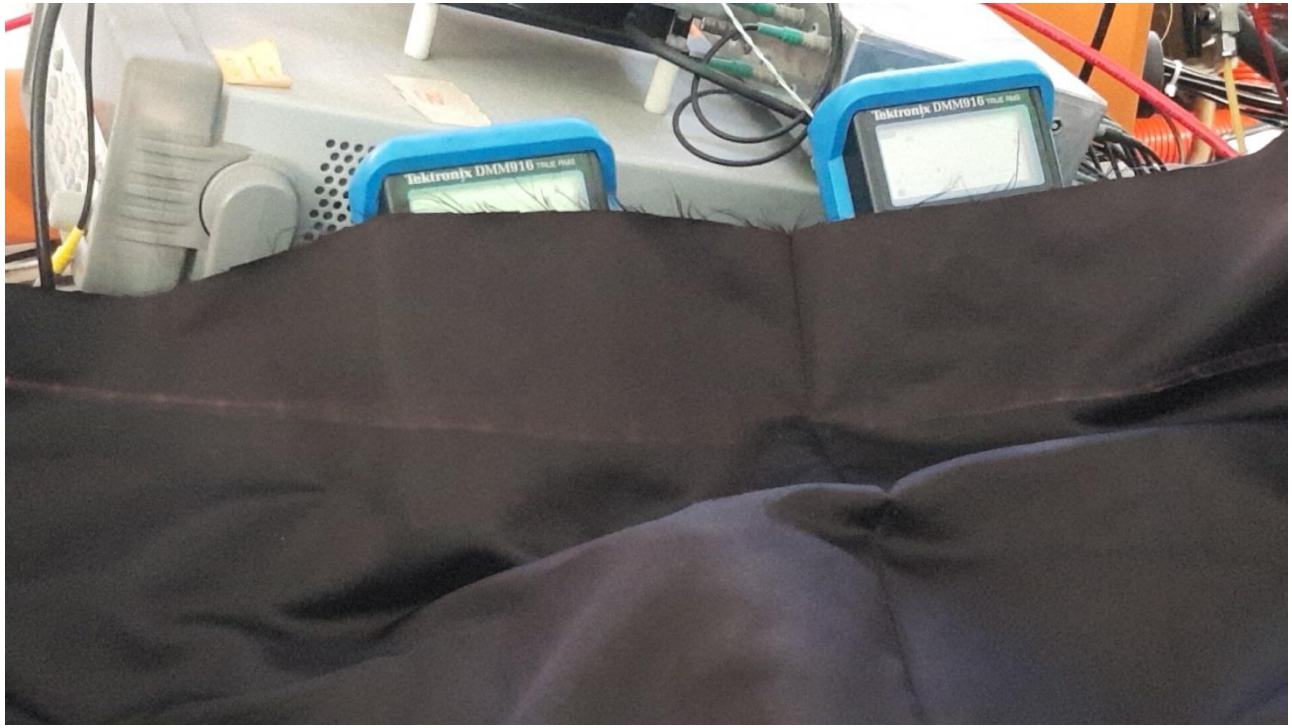
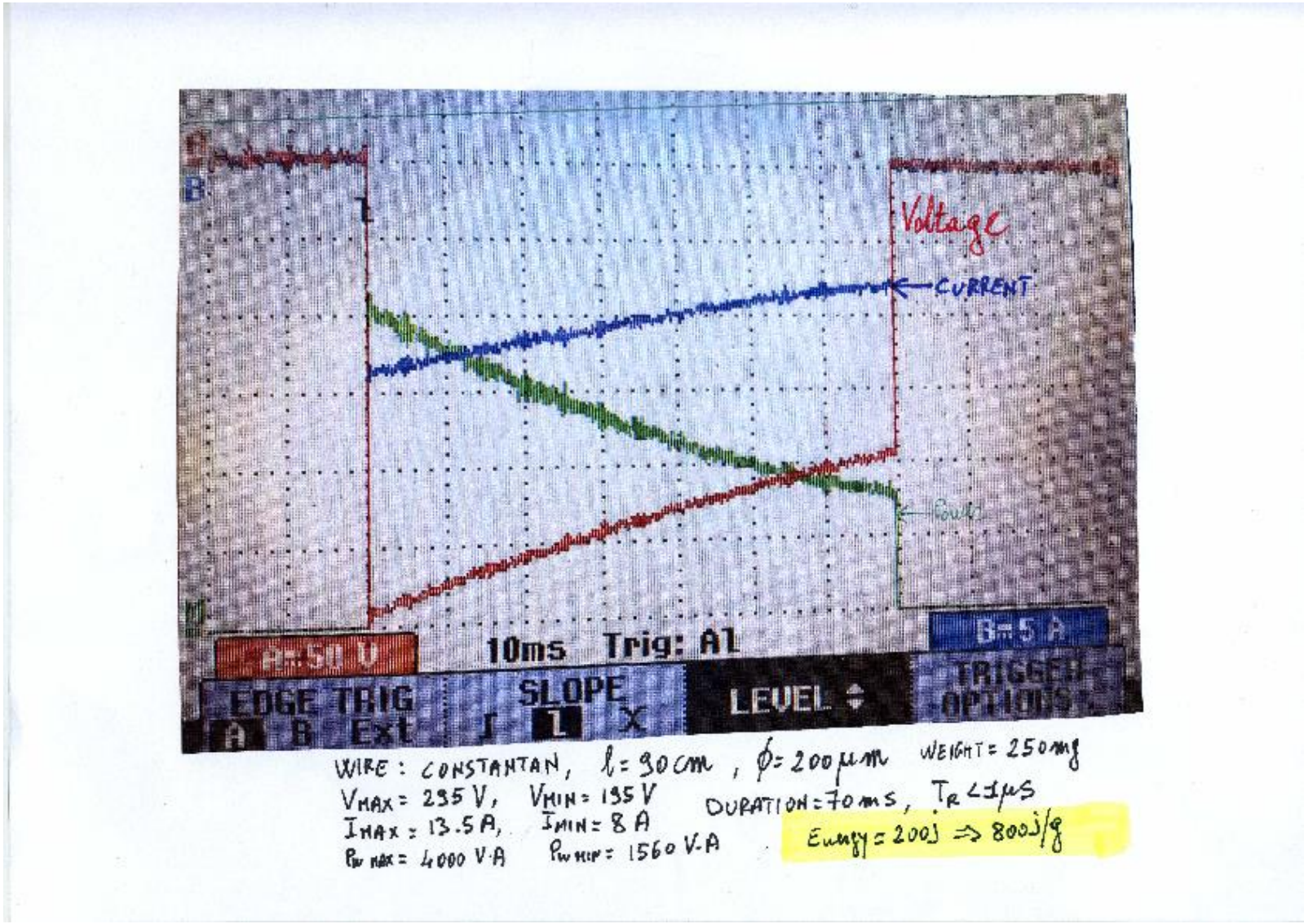


Observation: the area of the knots is more bright in comparison to normal wire, meaning higher temperatures.

Also in DC operations the area of the knots has temperatures higher with respect to the normal wire area, i.e. local thermal non-equilibrium.

Oscilloscope view of a typical HPP. Wire with knots. Pulse duration 70ms. Effective energy 200J



Evaluation of energy needed to increase wire temperature to 1000°C.

The HPP (High Peak Power pulser) has to provide the wire enough energy to feed for:

A) The thermal inertia of the wire ($m \cdot Cp \cdot \Delta T$), where m is the mass (g); Cp is the specific heat (J/g/°C); ΔT is the temperature difference (K);

B) The power (J/s) lost by irradiation (Stefan-Boltzmann): $P=5.67 \cdot 10^{-8} \cdot T^4 \cdot S$. T in K.

C) The Power (J/s) lost by conduction to the air: $10 \cdot S \cdot \Delta T$.

Considering our situation, we have, in the case of 200 μm wire, 1 g (normalized) of total mass, density of Constantan 8.90 g/cm^3: m=1 g; Cp=0.41 J/g/K; S ($2*\pi*r*l$)=22.4 cm^2.

A1) $1 \cdot 0.41 \cdot 1000$=**410 J**;

B1) $5.67 \cdot 10^{-8} \cdot 1273^4 \cdot 22.4 \cdot 10^{-4}$=333 J (in 1 second!!), i.e. **23 J** in 70ms.

C1) $10 \cdot 22.4 \cdot 10^{-4} \cdot 1000$=22.4 J (in 1 second), i.e.= **1.6 J** in 70 ms.

In conclusion, the energy of 800 J is enough for our purposes.

Typical results of EDX (Energy Dispersive X-ray spectroscopy) analysis of CNM sample after HPP procedures.

- **The max energy of electrons is 30 keV. With the main materials used, the depth of analysis is about 2 μm.**

- **It is observed that the relative composition of main materials (Cu, Ni) is STRONGLY dependent on the position along the wire.**

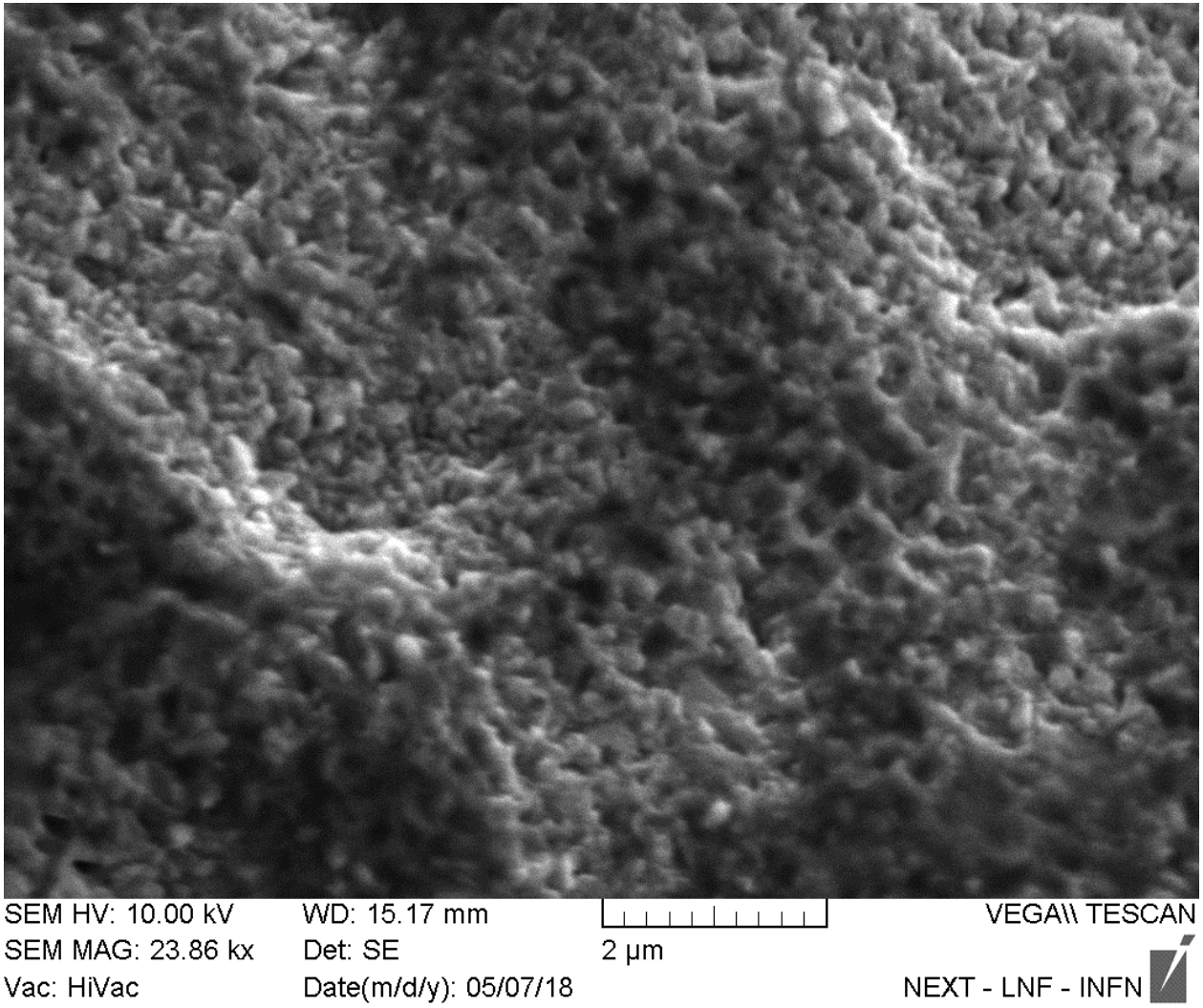
- **At the area of the knots (the most hot/bright according to the video-camera) the Cu content, at surface, is the largest because of partial decomposition of CNM alloy and moving (diffusion) of the elemental Cu. Cu has a lower melting temperature, in alloy mixture, with respect to Ni.**

SEM observations (3 points).

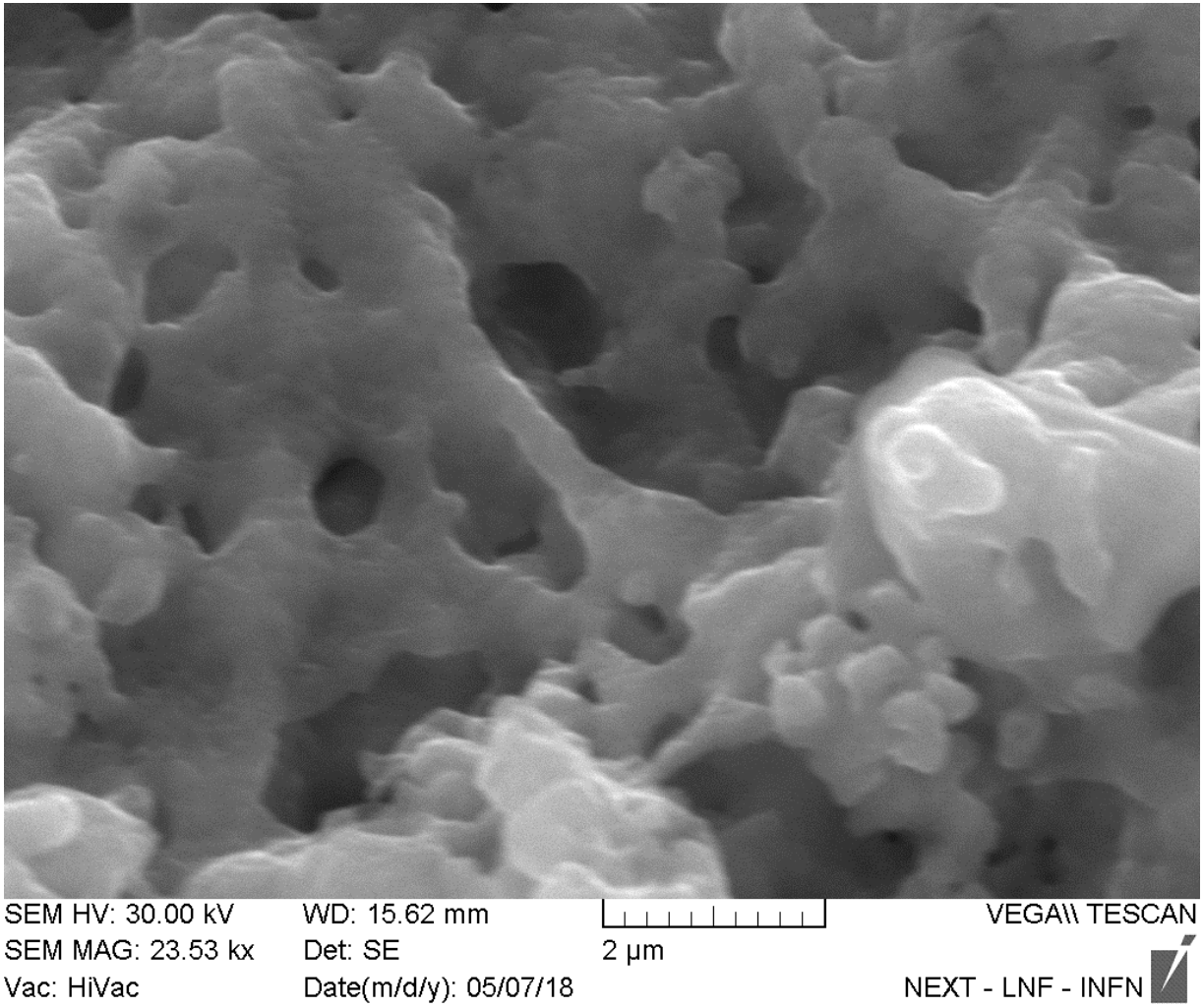
1) **Reference wire, just heated at 600 °C for 300 s in air.**

2) **External surface of a typical knot, after hundred HPP pulses and surface treated by nitrate mixtures of Sr-Fe-K-Mn. The treatment is several times repeated by "manual" deposition of liquids. The nitrates (Sr, Fe) almost immediately decompose to oxides just because large temperature reached (up to 700-1000 °C).**

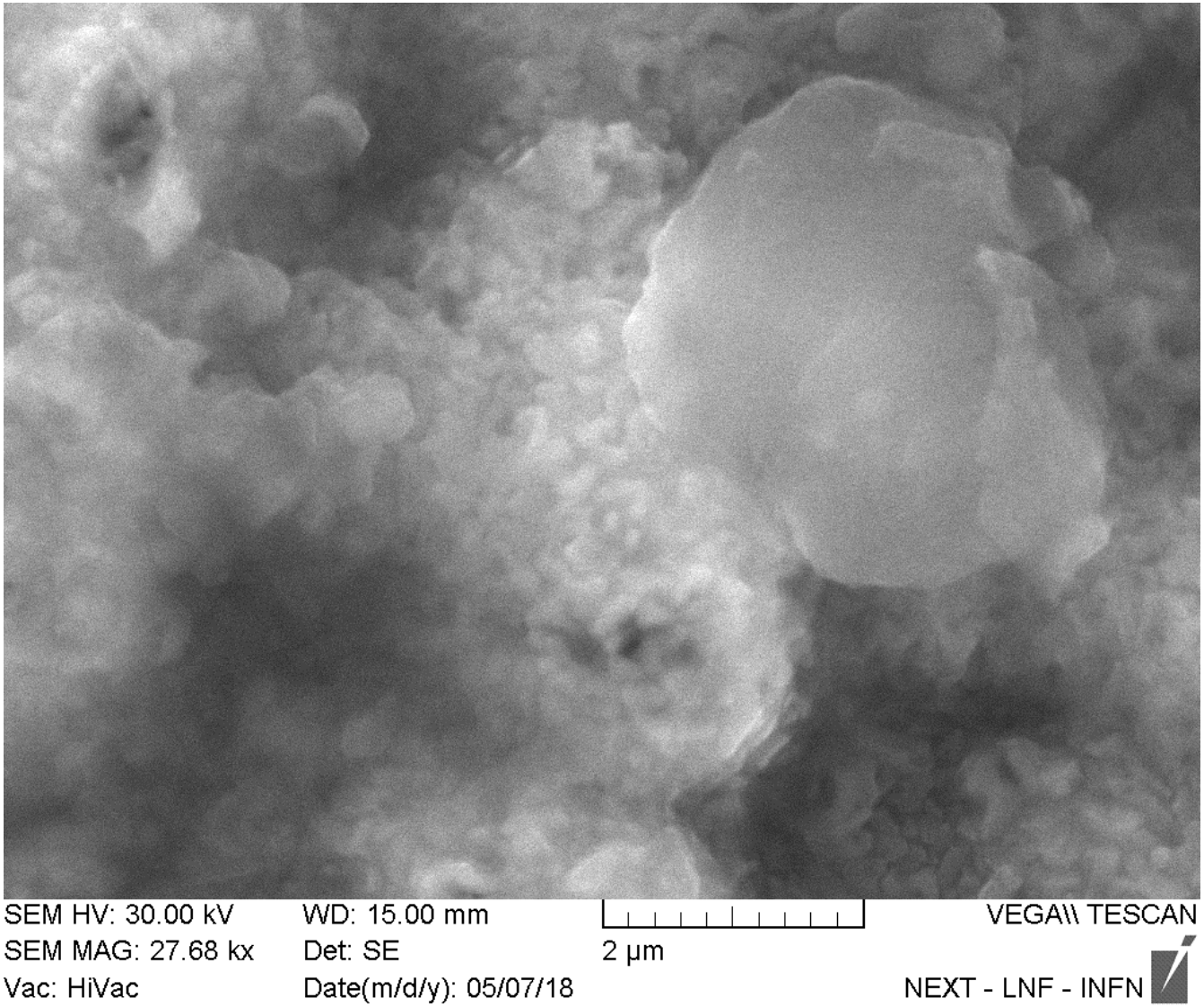
3) **External surface of an area between 2 adjacent knots, inter-distance 2 cm, after hundred HPP pulses and surface treated by nitrate mixtures of Sr-Fe-K-Mn.**



Reference: SEM observation of the final part of wire, Φ= 200 µm, just heated in air at 600 °C for 300 s.



Knot area. SEM observation of a typical area at the top of the knots.

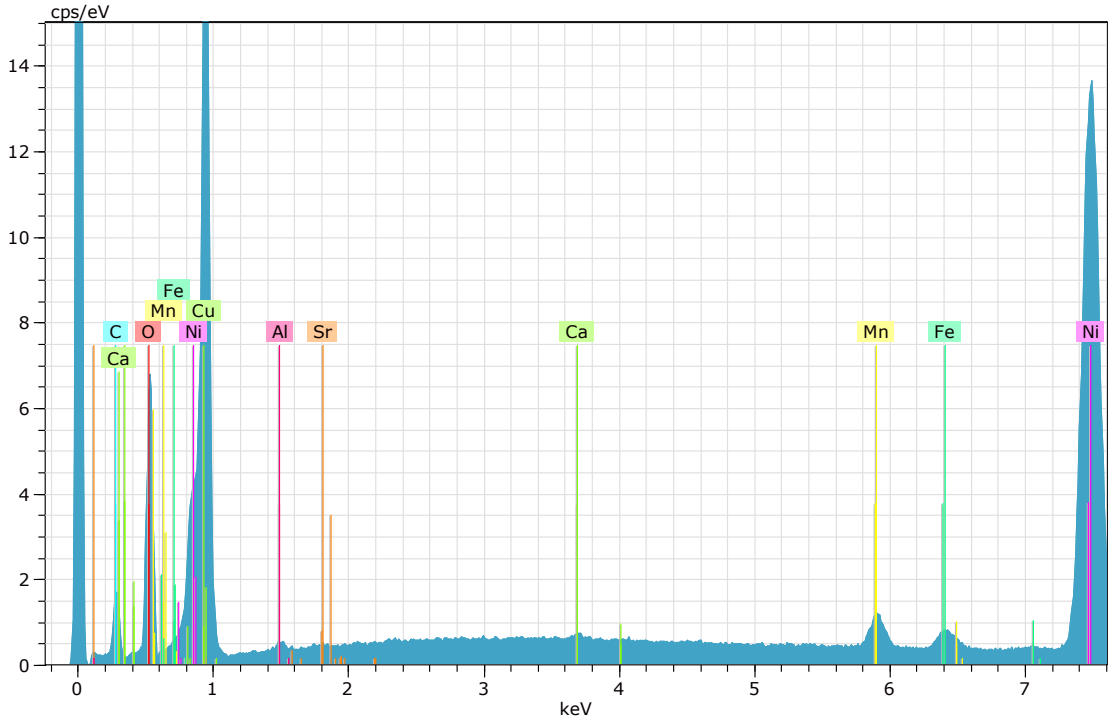


Area between 2 adjacent knots: SEM observation.

EDX analysis, 30 kV, typical depth 2 μm.

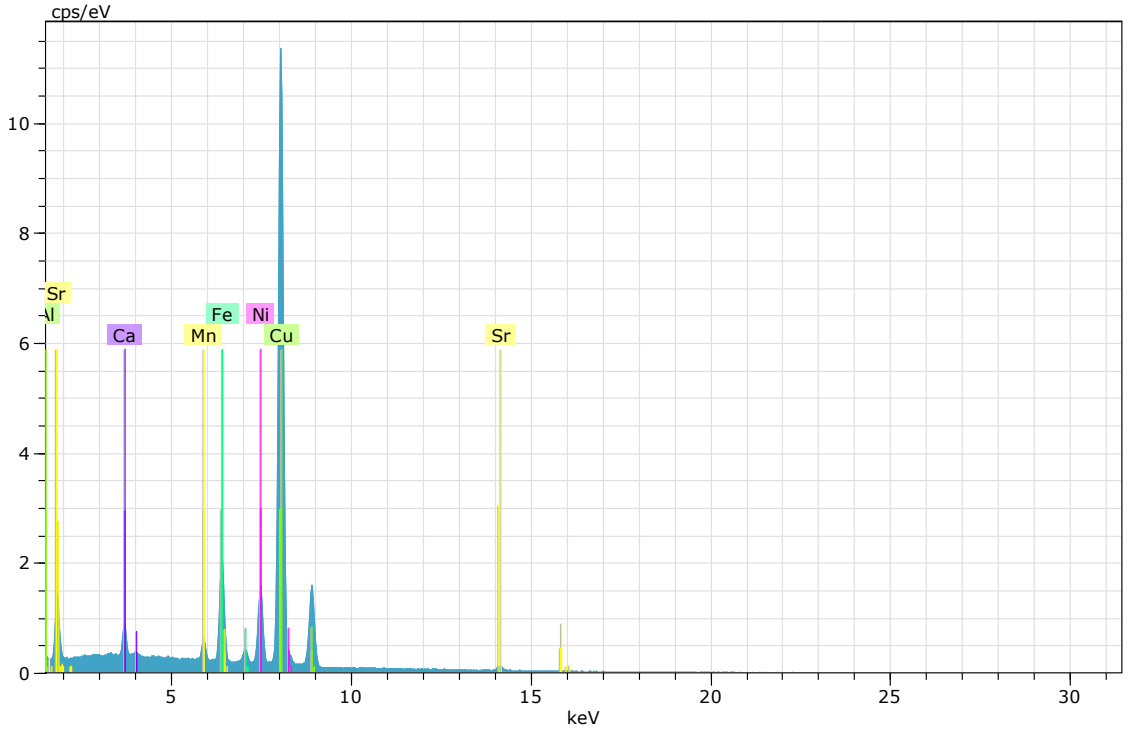
- **Measurements on: Reference point; top of knot; area between 2 adjacent knots.**

- **The ratio Cu/Ni, used as reference, varies largely, according to the treatments and geometrical position.**



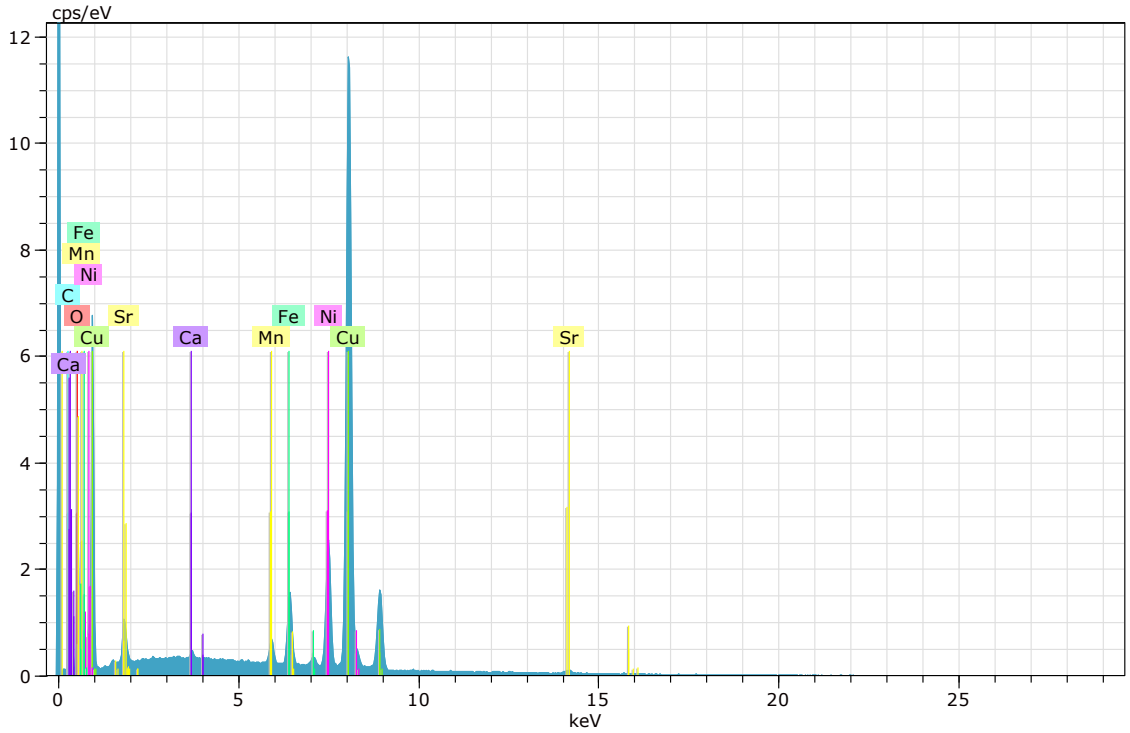
Net unn. C norm. C Atom. C Error (1 Sigma) **Reference: Cu=43.5%, Ni=31.6%**

				[wt.%]	[wt.%]	[at.%]	[wt.%]	
Cu	29	K-series	167934	42.65	43.53	23.71		1.07
Ni	28	K-series	149919	30.99	31.64	18.65		0.79
O	8	K-series	22943	12.95	13.22	28.59		1.66
C	6	K-series	5231	9.39	9.58	27.61		1.48
Mn	25	K-series	7629	0.97	0.99	0.63		0.05
Fe	26	K-series	4387	0.56	0.57	0.35		0.04
Al	13	K-series	723	0.24	0.24	0.31		0.04
Ca	20	K-series	899	0.12	0.12	0.10		0.03
Sr	38	K-series	71	0.10	0.11	0.04		0.04



Knot: Cu=58%, Ni=5.5%

			Net unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Cu	29	K-series	126198	58.16	58.40	30.91	1.46
O	8	K-series	11977	13.56	13.61	28.61	1.88
C	6	K-series	3187	10.58	10.62	29.74	1.83
Ni	28	K-series	15276	5.47	5.50	3.15	0.17
Fe	26	K-series	19339	4.96	4.98	3.00	0.16
Sr	38	K-series	1359	3.57	3.58	1.38	0.16
Ca	20	K-series	4517	1.07	1.08	0.90	0.06
Mn	25	K-series	3598	0.88	0.89	0.54	0.05
Mg	12	K-series	552	0.70	0.70	0.97	0.08
Al	13	K-series	820	0.64	0.64	0.80	0.07



Between knots Cu=62%, Ni=9.8%

El	AN	Series	Net unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]	
Cu	29	K-series	130832	62.34	62.50	36.21	1.56
O	8	K-series	11531	12.27	12.30	28.31	1.71
Ni	28	K-series	26185	9.75	9.77	6.13	0.27
C	6	K-series	2364	8.17	8.19	25.11	1.51
Fe	26	K-series	13484	3.44	3.44	2.27	0.12
Sr	38	K-series	938	2.45	2.46	1.03	0.13
Mn	25	K-series	4477	1.11	1.11	0.74	0.06
Ca	20	K-series	928	0.22	0.22	0.20	

Introduction of wires inside fiber glass sheaths and their impregnation

- **To prevent or minimize the separation of active (i.e. sub-micrometric) surface layers from core of the wires, we put them, immediately after their preparation, inside a *fiber glass sheath* (this procedure was systematically adopted since 2013).**

- **We have also reason to believe that this glass sheath may contribute to the generation of AHE under certain circumstances. This phenomenon was also observed in a previous set of experiments with Pd wires in 2008, with sheaths used for electrical insulation purposes.**

- **At that time, several types of fiber glass sheaths were tested. We realized that Alumina (Al_2O_3) based materials, although able to withstand quite large temperatures (>1200 °C), never gave useful effects. Only more common glass types (E Glass, i.e. boro-silico-alumino-calcic glass; e.g. made by SIGI-Favier, IT-F; a derivative of borosilicate glass with further addition of TiO_2, Fe_2O_3, Fe_2), although with limits on maximum continuous temperature of 550 °C, were showing synergistic effects with respect to AHE phenomena. We first thought that the glass itself could be an important factor but we had no justification for our experimental observations yet.**

- **Having in mind the idea of a "vessel" or containment made of micrometric fibers (diameter 4-6 μm, almost porous), we started modifying the sheaths by impregnation. In the initial experiments, impregnation was done just dipping the sheaths into a solution of $Sr(NO_3)_2$ in H_2O or D_2O: it was later decomposed to SrO at high temperature.**

- **Wires were later inserted in the sheaths impregnated per this procedure.**

- **Please note that SrO is one of the materials (e.g. CaO, Y_2O_3) that have a low working function for electron emission. The use of these materials is in accordance with procedures of Y. Iwamura (1999) in the field of transmutations (Sr→Mo, Cs→Pr) induced by Deuterium flux on multilayered (CaO, Pd) structures.**

Rediscovery of the work of Irving Langmuir

- ***We think that the impregnation step further enhances the intrinsic tendency of glass fibers to absorb or interact with atomic hydrogen, locally "produced" by Constantan.***

- **We also consider important to mention that the properties of some specific glasses with respect to their interaction with atomic hydrogen were discovered by *Nobel Laureate Irving Langmuir* around or before 1927.**

- **Specific experiments were presented at "*2014 MIT Colloquium on Cold Fusion effect*" (21-23 March 2014; MIT-Cambridge-USA). Performed 2 kind of experiments using 2 Constantan and 1 Pt wires, inserted inside glass sheaths, *at low and large amounts of additional glass.* H_2 atmosphere, different pressures. Effects larger at *low pressures*, in agreement with observations on Langmuir about 2H to H_2 recombination rate (higher at low pressures). *The 2H to H_2 recombination reaction is highly exotermic. In principle, the local temperature can be as high as 36000 K at very low pressures.***

Introduction of Iron and Potassium and their effects

- **Aiming to increase AHE, in a further development of such material, we added Fe (as mixed oxides) on the surfaces of wires and sheaths together with K (as a dissociation promoter of $H_2 \rightarrow 2H$ reaction, following Fischer-Tropsch like procedures, along 1920-1945, for gasoline synthesis starting from CO and H_2). The role of K, among others, was put to our attention by C. Lorenzetti (co-author) since 2014. The addition of iron was made before the pulsed, high power, procedure. Later-on (May 2016) we also added Mn in order to stabilize the observed effects: possibly because Mn may decrease K evaporation at high temperatures.**

- **Concerning Fe addition, we later realized that the first *CNM* batch that we used was prepared before 1970. We measured that the Fe content, either as impurity or intentionally added, was as large as 0.5-1% on average, while in certain spots we have measured up to 3-5% concentration, especially on the surface.**

- **In addition to the lower purity of starting materials (i.e. Cu, Ni, Mn), we have been suspicious of some surface contamination during swaging process and/or inappropriate storage in a rusted iron cupboard in Italy from where we got the "old" Constantan.**

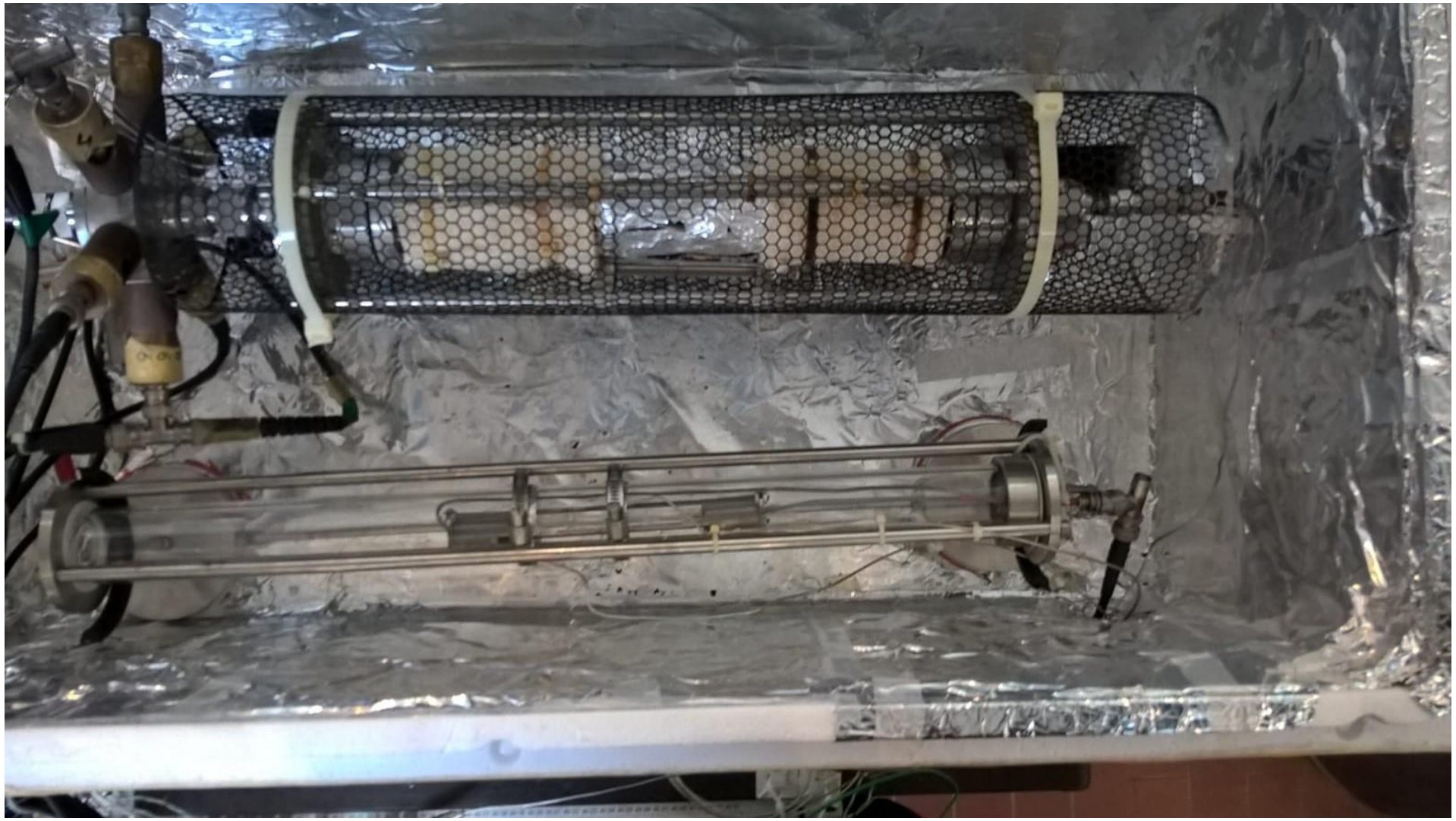
- **In facts, we observed that the first "vintage" batch of Constantan gave relatively large values of AHE (up to 5-20% of the input power at 50 W input). While AHE became much lower with "recent" batches featuring a higher purity, both with the same reactor operated in the isoperibolic regime.**

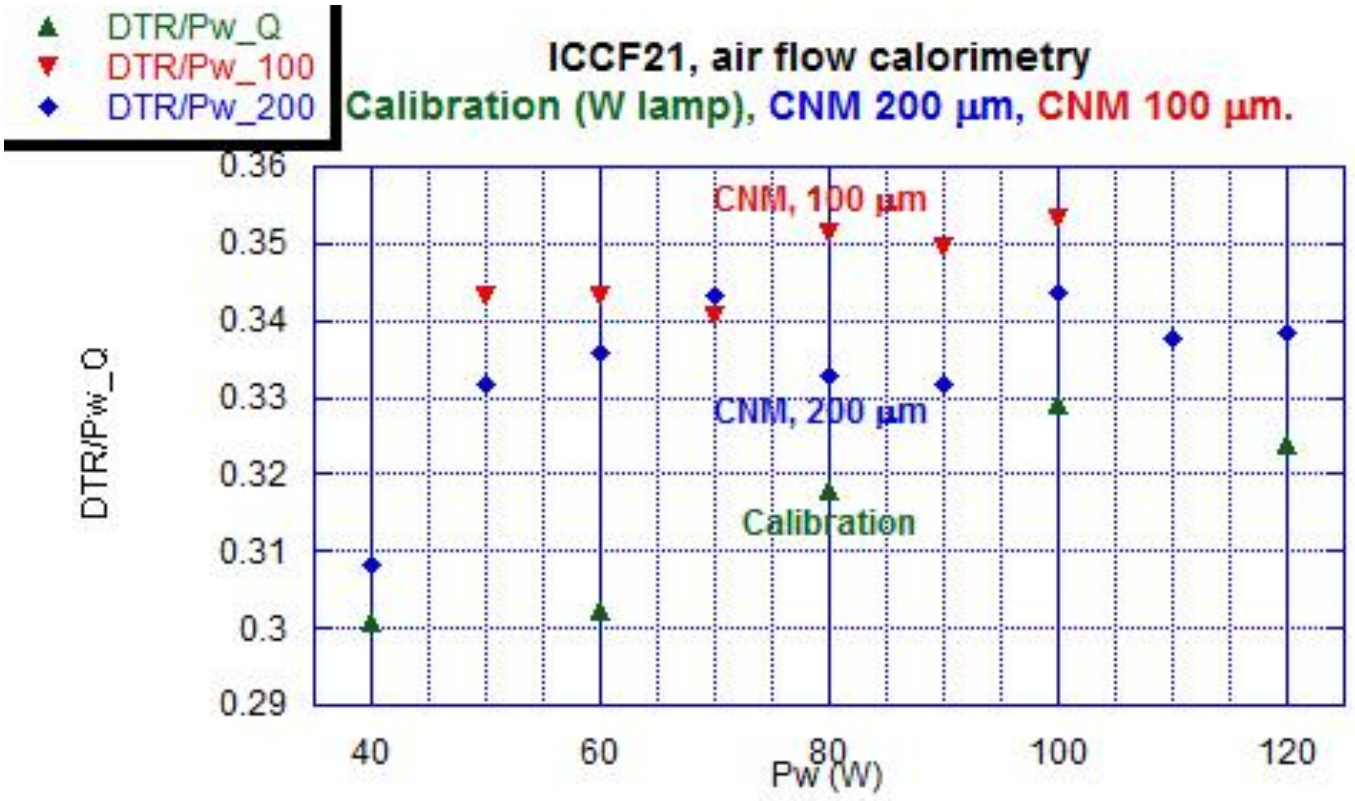
- **If we also consider that the J-type thermocouples, mentioned before, also contains iron, the case for its synergistic effect on AHE is strongly reinforced.**

- ***Finally, Fe has the unusual propriety of absorbing H_2 at high temperatures (>600°C) and releasing it at lower temperatures. So, an intrinsic counter-effect about Hydrogen storage could be properly used, especially from the point of view of NON equilibrium conditions.***

Latest results on air-flow calorimetry.

New calorimeter, air flow. Calibrator (W lamp) inside the insulating and reflecting box.





Comparison of results, expressed as ΔT (Out-In) of cooling fan, divided by input power (W). 1° measurement was performed by giving power to *Calibrator* (Halogen Tungsten lamp, max Power 230 W), *under-powered* to reduce the temperature. The lamp was put inside a borosilicate glass tube with same composition and dimension of the reactor (L=29 cm, Φ=34-40 mm). 2° measurement using *CNM 0.1 mm* diameter (broken after only 840 s at 100 W of input power➔ *very conservative data*; *data acquisition frozen: like EMP??*). 3° measurement with *CNM 0.2 mm* diameter. Gas=D_2 at 1.2 bar at RT. *All the data shown are row, i.e. without corrections for air density inside the thermal-insulating calorimeter box, decreasing at high temperatures. Only external RT, and humidity, are kept almost constant.*

Short comments and explanation of the heat results.

The air flow calorimeter operates according to the usual formula:

$$E=m\cdot C_p\cdot\Delta T$$

Where: m is air density [g/cm^3, or kg/m^3]; Cp the specific heat [kJ/kg*K]; ΔT is the temperature difference between Out and IN [K].

The problems arise because the air is a gas and its density changes largely (i.e. decreases) increasing the temperature while Cp increases but slightly. Moreover, the water content increases largely with temperature. The energy to be given inside the measuring chamber to increase water temperature (4.184 J/g) or even evaporate it (i.e. phase transition) can be large. Such water is a negative term in the system.

Some of the values are reported in the following Tab.:

Temperature (°C)	Density (kg/m^3)	Cp (kJ/kg*K)	Max H_2O content (g H_20/Kg air)
0	1.293	1.0037	4.9
5			6.82
10	1.247	1.0041	9.4
15	1.225	1.0043	12.8
20	1.205	1.0045	17.3
25			20
30	1.165	1.0050	30.4
35			36
40	1.127	1.0055	49.51
50			83
60	1.060	1.0068	130
80	1.00	1.0084	
100	0.946	1.0104	

Tab: Values of density, specific heat, water content of air in the temperature interval 0-100°C:

As a consequence, the simplest way to manage data is to make calibrations.

- **Moreover, with increasing temperature the heat losses, by external air convection to ambient, from the large insulating box, increase.**

- **Anyway, *some cross-check of results were made, time to time,* by calculations using the value of Tab. and measuring the temperature of the external wall of the insulating box, by a high sensitivity (0.1 °C) IR video-camera (Nikon).**

- **In short, the aim of our experiment is to increase the value of AHE, NOT to improve the accuracy of the measurements (up to 1% level), once obtained enough satisfactory results for our purposes.**

Photo of the borosilicate glass reactor after latest experiment: clearly visible, at center, internal partial melting due to local overheating, like *imprinting of sheaths*.

Conclusions

- We observed that even using flow-calorimetry procedures, that MINIMIZE the non-equilibrium situations, the whole of procedure adopted (i.e. wires of Constantan with surface sub-micrometric, several knots with low diameter of holes, multiple addition of material at low working function, Fe, K and Mn) allows the production of AHE, even at levels quite lower with respect to isoperibolic geometry. ***The value of wire temperature, enough high but avoiding sintering of sub-micrometric materials, seems to be a key factor***.

- The amount of AHE is inversely proportional to wire diameter, i.e. as small as possible.

- We observed, twice, that the 100-µm diameter of Constantan wire is NOT able to sustain, for enough long time, input power density close to 100 W in our experimental conditions (length about 130 cm, 115 cm after making knots). Anyway, in such regime the AHE values are growing very fast, perhaps in a positive feedback regime.

- Further systematic work is needed to keep under full control such extremely-interesting results/operating regimes. Perhaps the voltage drop along wire (i.e. ***NEMCA***) is important.

- It is interesting to observe that all 3 types of measurements performed during and after thermal treatments (fast camera, SEM, EDX) gave results each-other self-consistent.

- **It is useful to observe, from a futuristic/optimistic technological point of view, that the maximum excess power density, with 100-µm wire, was larger than 100 W/g, supposing that also the bulk of the wire, is operative, not only surface.**

- **From the other side, if we suppose that the glassy sheaths, wetted by the usual mixture of Sr-Fe-K-Mn (total weight of about 3 g), made a special role for AHE generation, the power density drops largely. Anyway, the industrial cost of glassy sheath is quite low: few €/g.**

- **Such values are qualitatively similar to the old experiments (up to 2008) when we used mainly long Pd wires (diameter of 50 and 100 micron), again inserted on glassy sheaths and treated, before insertion, with several cycles of deposition of specific materials (Th-nitrate, Sr-nitrate, liquid glass home-made). Sadly, almost all logbooks of such experiments, where all the details (i.e. know-how) were reported, were destroyed from people working at LNF on February 2015.**

- **For the last 10 days, just before the ICCF21, a new reactor has been operating, with minor changes about the overall geometry: our aim is to reconfirm the previous data and increase, step by step, the input power avoiding to get uncontrolled self-braking/burning of the wire. Up to now, at low powers, the results are similar to the previous experiment just reported.**

Introduction. We have introduced since 2011 the use of Constantan wires ($Cu_{55}Ni_{44}Mn_{1,}$ CNM), treated to have a sub-micrometric surface texture with an enhanced capability to dissociate Hydrogen (H_2) and/or Deuterium (D_2) from molecular to atomic state. Key reasons for the introduction of CNM were: cost reduction of *active material* (i.e. Pd and its alloys); improvement of the durability of the material, wires (Φ=50-200 μm, l=50-200 cm) activated using pulse heating (ultra-fast cycles from room temperature to 900 °C), and loading/unloading of Hydrogen or Deuterium.

Reactor materials. The reactor body is made of thick borosilicate glass (Schott) working up to 500°C; gas pressures (pure H_2, D_2 or mixed with noble gas Ar, Xe) between 0.05-3 bar. Since 2015 we have used, in the reactor, 3 wires of 125 cm length: Pt (ϕ=100 μm) used both as local thermometer and for calibrations; "standardized" CNM (ϕ=200 μm); "explorative" CNM wire (different ϕ, l, number of wires, thermal pre-treatments, coating, ...). All wires, except Pt, were initially treated with a series of high power electric pulses (up to 50 kVA/g ➔ 900 °C) in order to modify the dimensionality of smooth surface to sub-micrometric by oxidation, following the pioneering work of Y. Arata (Osaka Univ.) on nanomaterials. The specific surface increased thousand times as well as the efficiency of CNM as catalyzer of H_2/D_2 dissociation. Moreover, the surface is several times coated with Low Working Function materials (mainly SrO), according the intuitions/tests of Y. Iwamura (MHI, Yokohama) about the role of electron emission in LENR field. Each wire is inserted into glassy multi-filamentary sheaths, also impregnated by liquid solutions [$Sr(NO_3)_2$, $Fe(NO_3)_3$, $KMnO_4$; later decomposed to oxides], in order to reduce the drawback of sub-micrometric material detaching from wire surface. The borosilicate has the peculiarity of adsorbing large amounts of H (1927, I. Langmuir). Finally, we also made several knots (hole<0.1 mm) along the CNM wires to get non-equilibrium conditions due to the local thermal gradients and high magnetic fields (flowing current up 2.5 A, FexOy magnetism).

Results. In previous experiments we have evaluated AHE (anomalous heat energy) using an isoperibolic procedure being the most appropriated to produce *non-equilibrium conditions* in the system (thermal gradients in this case), as observed by several Researchers in the field. This allowed to measure gains near a factor 2 in the experiments at the highest temperature, although with limited stability over time. Recently, we have decided to compare previous results obtained from the isoperibolic approach with an air flow-calorimetry. During the new experiments, the external wall of the glass reactor has been covered with a double layer of black and thick aluminum foil to further homogenize the internal temperature. The calorimeters consist in a large insulating Styrofoam box with a layer of thick aluminum foil covering the internal surface for improved thermal homogeneity. The calorimeter assembly contains the active reactor and a W lamp inside a dummy reactor for calibrations; these are performed powering the lamp (0->120W->0W, step 20W). Best results to date are the following: A) with a CNM wire with ϕ=100 μm D_2 at 1 bar, internal reactor temperature 500°C, input power 90W the AHE was over 12 +-2W, i.e. over 150W/g, but after 1 day the wire was broken; B) with a CNM wire with ϕ=200 μm it has been necessary to have a Xe-D_2 mixture (each 0.1 bar) and input power of 120 W in order to obtain an AHE of 6-7 W stably for weeks. Qualitatively, such results, and dynamics, where observed twice with 2 different set of wires. It is worthy to note that the behaviors of thin wires was even similar to quite old experiments (50 μm Pd wires): sadly most of the documentation of our old experiments were destroyed by some people at LNF on Feb. 2015. Further work is necessary to improve reliability of the (nice) results (AHE=150W/g; integral of energy over 10MJ/g) obtained by 0.1mm wire.