

## OVERVIEW OF H-NI SYSTEMS: OLD EXPERIMENTS AND NEW SETUP

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This is an overview of our experimental activity during the last twelve years. We have been studying the Ni-H system at temperatures of about 700 K. Our investigations have revealed several interesting effects:

- energy production for long time
- neutron emission
- $\gamma$ -ray emission
- charged particles emission
- appearance of elements other than Ni on the surfaces of Ni samples.

These experiments were performed in several laboratories: the greatest part in Siena but also in Bologna, Colleferro and Pavia. A new laboratory in Colle Val d'Elsa (Siena) will become operative in the summer 2004.

### Introduction

Several kinds of metal samples were used: pure Ni, Nickel alloys and Nickel plated, having cylindrical or planar shape. In a typical experiment the samples were inserted in a cell, loaded with hydrogen at pressure in the range 100-1000 mbar and kept at temperatures between 420 and 720 K<sup>1-4</sup>. The reported temperature range represents the optimum choice for hydrogen loading in Nickel below a limit value 1000 K which guarantees the cell's integrity. Experiments were performed using three different kinds of cells built to hold one cylindrical sample, 4 cylindrical samples or three planar samples. A schematic layout of the experimental setup is shown in Figure 1.

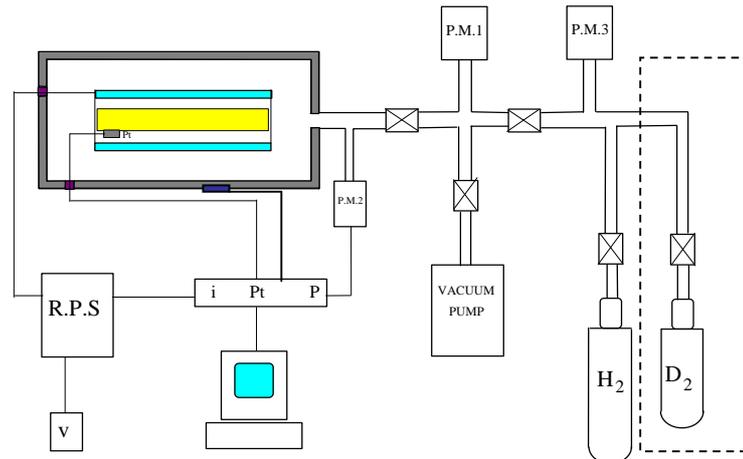


Figure 1. A schematic layout of the experimental setup.

The following sections describe several results obtained with these specimens, regarding their hydrogen loading, excess heat production, nuclear particles emission, and detection of new elements on the surfaces of loaded samples. Table 1 summarizes a selection of results obtained in the experiments.

Table 1. A selection of results obtained in the experiments.

AUTHORS	LAB	START	SAMPLE	H LOADIN G	$\Delta t_{\max}$ [d]	$\Delta P_{\max}$ [W]	TOTAL HEAT EXCESS [MJ]	NUCLEAR ASHES
F. Piantelli	Siena	January 92	Ni cylindrical	high	36	12	not valued	no $\gamma$ -ray or neutrons measures altered metal surface
S. Focardi, R. Habel, F. Piantelli	Siena	October 93	nickel- plated Ni alloy cylindrical	high	55	44	> 90	no $\gamma$ -ray or neutrons measures altered metal surface
S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Siena	Septemb er 94	nickel- plated Ni alloy cylindrical	very high	278	72	~ 900	$\gamma$ -ray neutrons altered metal surface
S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Siena	Novemb er 94	nickel- plated Ni alloy cylindrical	high	319	18	~ 600	$\gamma$ -ray no neutrons measures no measures on metal surface
S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Siena	March 96	Ni plane	medium	22	27	38	$\gamma$ -ray no neutrons altered metal surface
S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Siena	July 96	Ni plane	very low	0	0	0	$\gamma$ -ray no neutrons altered metal surface
E. Campari, S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Bologna	June 96	Ni alloy cylindrical	high	....	....	not valued	no $\gamma$ -ray or neutrons measures altered metal surface
S. Focardi, V. Gabbani, V. Montalbano ,F. Piantelli , E. Porcù, E. Tosti, S. Veronesi	Collefer ro	Septemb er 97	Ni alloy cylindrical	medium	147	8	~100	no $\gamma$ -ray or neutrons no measures on metal surface
S. Focardi, V. Gabbani, V. Montalbano F. Piantelli , S. Veronesi	Siena	Novemb er 97	Ni plane	low	0	0	0	$\gamma$ -ray no neutrons no altered metal surface
S. Focardi, L. Cattaneo V. Gabbani,V. Montalbano L. Nosenzo, F. Piantelli , A. Piazzoli, S. Veronesi	Pavia	Septemb er 01	nickel- plated Ni alloy cylindrical	medium	....	...	elab	no $\gamma$ -ray or neutrons measures no measures on metal surface

# 1 Hydrogen loading

Before loading the cells with hydrogen, air is pumped out. In some cases annealing cycles were performed at temperatures up to 820 K in vacuum and 720 K in hydrogen. The hydrogen loading in the metals is measured by the pressure decrease inside the cells. We observed in the experiments various hydrogen loading rates and also a temperature dependence. Such effect is clearly evident from Figure 2.

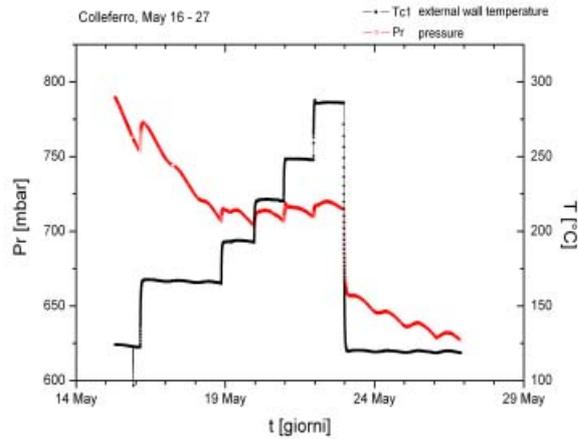


Figure 2. Hydrogen pressure (continuous curve) and Ni temperature (step curve) measured in a period of 10 days. The hydrogen loading depends on the temperature and increases for  $T < 200$  °C.

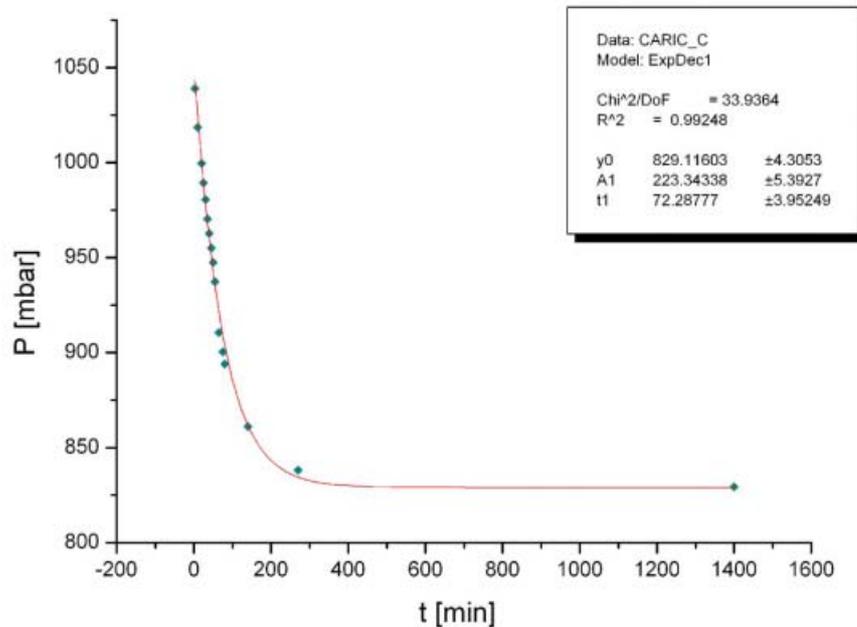


Figure 3. An example of fast hydrogen loading which corresponds to about 7000 mbar/day.

In this case, the mean loading rate resulted to be of the order of 50 mbar/day. The fast loading shown in Figure 3 corresponds to about 7000 mbar/day.

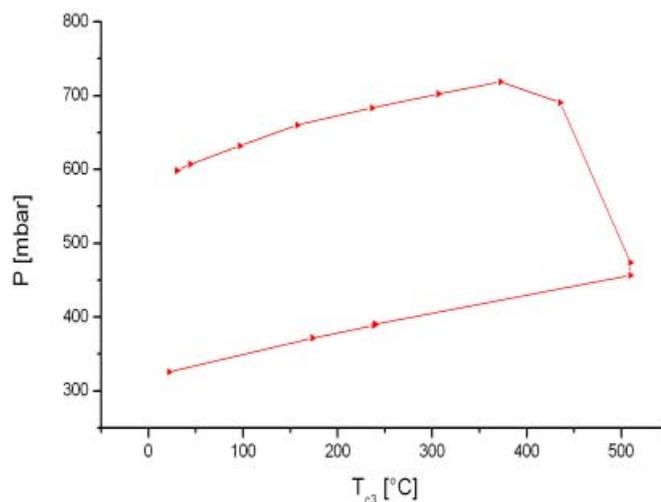


Figure 4. Pressure versus temperature plot showing a slow hydrogen loading. The pressure decrease which correspond to the temperature increase cannot be explained by the gas laws.

The hydrogen absorption is not completely reversible: as shown in Figure 4, in a thermal cycle, the final pressure is lower than the initial pressure at the same temperatures.

## 2 Excess heat production

Regardless of the cell type, excess heat production was detected using the same method. At the beginning of the experiment, a set of calibration curves was constructed plotting the temperature of several reference positions as a function of the input power<sup>9</sup>. A correction was done for the room temperature fluctuations.

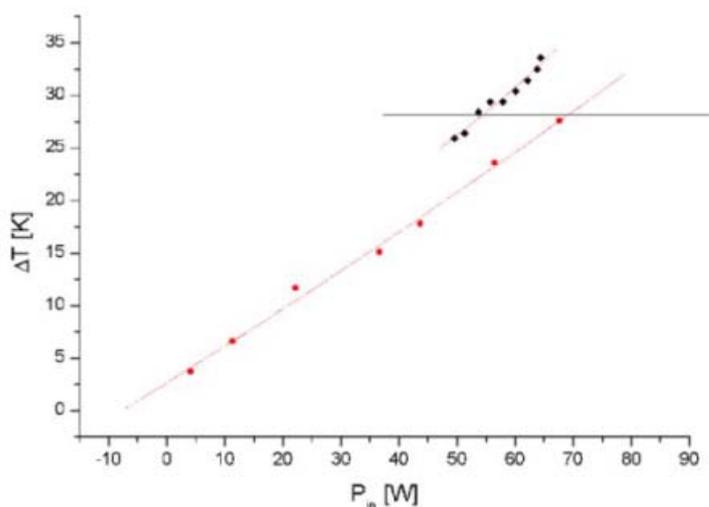


Figure 5. Calibration curve (line connecting experimental points) and points corresponding to heat production. The distance measured on the horizontal line directly provides the excess heat amount: in this case about 15 W.

A temperature increase with respect to the reference curve, at a given input power, is evidence for excess heat production. In Figure 5, which refers to a cell with a cylindrical sample, shows the calibration curve (lower) and the points corresponding to heat production (higher). The distance measured on the horizontal line directly provides the excess heat amount. In two cases, a considerable amount of energy was produced<sup>4</sup>: a cell with a single cylindrical sample (cell A) produced 900 MJ in 278 days; a second cell with four cylindrical samples (cell B) produced 600 MJ in 319 days. The samples were made of pure Ni, or an alloy of Ni, Cr, Mn and Fe.

### 3 Radiation emission

The excess energy release described in the previous section cannot be accounted for by any chemical reaction involving the masses of Ni and H inside the cell. Therefore same kind of nuclear process should be taken into consideration. This, in turn, justifies the search for radiation emission associated with the heat production. Several kind of detectors were

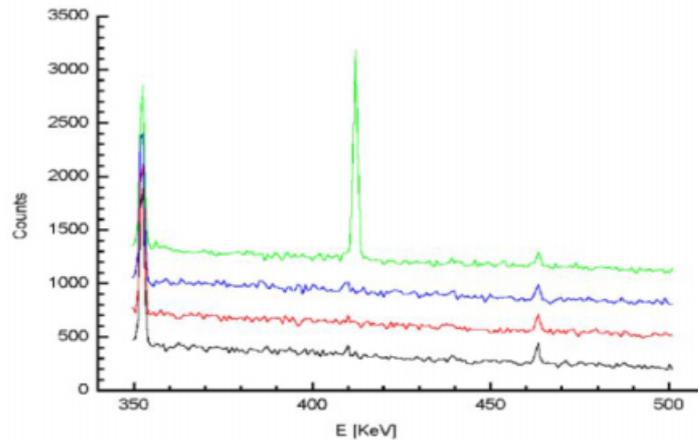


Figure 6. Four germanium  $\gamma$ -rays spectra: the upper curve (note the 411.8 keV peak) was obtained measuring the  $\gamma$  emitted from a gold sheet placed for 12 days near the cell A. The other curves correspond to laboratory background, gold sheet not irradiated, gold sheet after a 12 days exposition 10 m away from the cell.

used at this purpose: neutron detectors filled with  $^3\text{He}$  and shielded, for neutron thermalization, with paraffin or polythene; NaI scintillation counters; a germanium detector; X-rays films; a cloud chamber.

In one case, during the period of heat production from cell A, neutron emission was detected both by the neutron counters and the activation method<sup>5</sup>. For the latter, a gold sheet placed inside a paraffin box was kept near cell A. A neutron capture transmutes  $^{197}\text{Au}$  into  $^{198}\text{Au}$  which emits, while decaying, a 411.8 keV  $\gamma$ -ray. We detected such  $\gamma$ -rays with the germanium detector (Figure 6) and evaluated a neutron flux of about 6000 neutrons/s

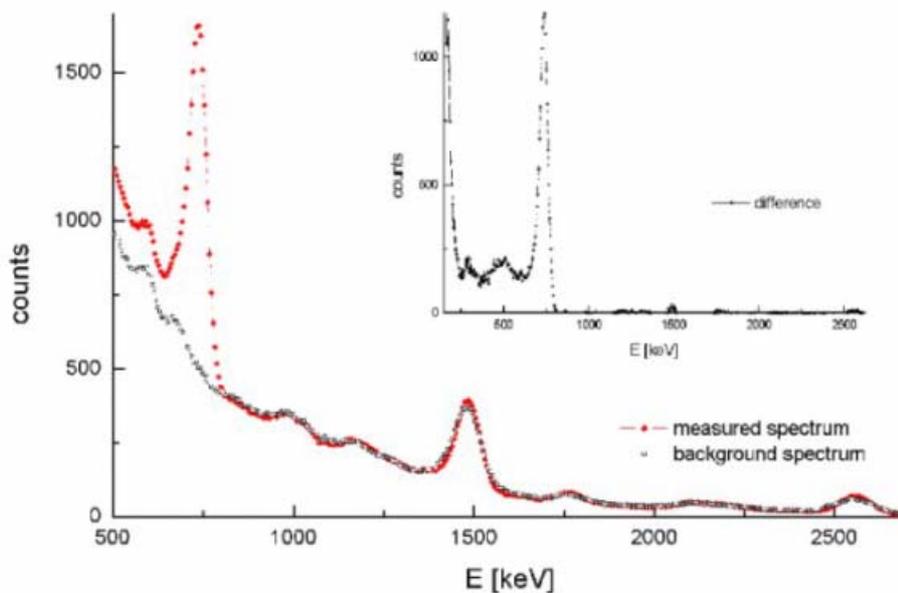


Figure 7. NaI  $\gamma$ -rays spectrum showing a peak superimposed to the background. The insert, obtained by subtracting the background, shows the typical structure of a  $\gamma$ -ray: photoelectric peak, Compton and backscattering peak.

In some other occasions, working with planar cells,  $\gamma$ -ray emission was detected with a NaI counter<sup>6</sup>. The spectrum (Figure 7) shows, superposed to the background, a peak centered to  $660 \pm 1$  keV. The  $\gamma$ -ray emission lasted for a few days and then ended.



Figure 8. Picture of a track of an ionizing particle emitted from the Nickel taken in a cloud chamber.

The cylindrical specimen which produced energy in cell A, at the end of experiment, was placed in a cloud chamber. As shown in Figure 8, we observed tracks of charged particles coming from the specimen<sup>8</sup>. We checked that no tracks were present when a reference nickel sample (not treated in a cell) was placed inside the cloud chamber.

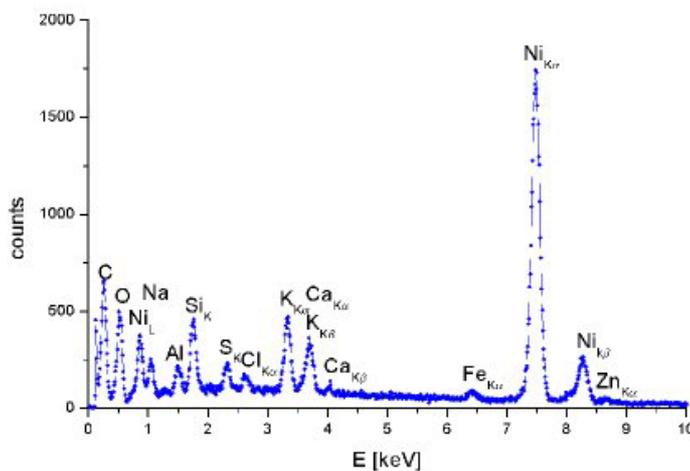


Figure 9. A typical EDAX spectrum observed in a little spot, showing the existence on the surface of Na, Al, Si, S, Cl, K, Ca, Fe and Zn.

At the end of each experiment, the sample surfaces were analyzed by using a SEM with an EDAX probe. Those samples which produced energy or absorbed a considerable amount of hydrogen exhibited regions where elements not present before the treatments were observed. Figure 9 is a typical EDAX spectrum of such regions. These elements are usually found in little spots or corroded regions. We found F, Na, Mg, Al, Si, P, S, Cl, K, Ca, Cr, Mn, Fe, Cu and Zn. Apart from light elements, which are not detectable by SEM, we found all the elements lighter than Ni plus Cu and Zn, except for Sc, Ti, V and Co<sup>7-8</sup>. Figure 10 is an optical image of one of those corroded regions where the above mentioned elements are found.



Figure 10. An optical image of a corroded region, where elements other than Ni can be found by using EDAX .

#### 4 New Laboratory in Colle Val d'Elsa

As a result of a collaboration between Siena University and Lumenergia (a firm based in Brescia), a new laboratory with 400 m<sup>2</sup> of floor space will begin activity in Colle Val d'Elsa, near Siena, by the end of 2004. In this new laboratory a facility for Ni samples production (Figure 11) has been installed: it will be used to produce Ni samples for the next experiments.



Figure 11. Colle Val d'Elsa laboratory facility for Ni samples production.

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