# VERY THIN NICKEL LAYERS HEATED OVER CURIE TEMPERATURE SHOW HIGH TEMPERATURE SPOTS IN HYDROGEN LOADING EXPERIMENTS. Ubaldo Mastromatteo - STMicroelectronics; Via Tolomeo, 1; 20010 Cornaredo; Italy.

#### Abstract

With the purpose to study the behaviour of thin Nickel layers in presence of hydrogen, a prototype microcell has been designed using a silicon chip of about 6 mm2 size. On one side of the chip it has been realised a structure including a low electrical resistance polysilicon heater (anode), a high Hydrogen content dielectric layer and a 0.1  $\mu$ m thick Nickel resistor (cathode).

Several experiments using that cell prototype have pointed out that it is possible in certain conditions of temperature and electrical biasing, the activation into the metallic lattice of Hydrogen absorption able to modify the electrical resistivity of the layer and also generating large fusion spots in the Nickel layer due to high and fast temperature rising. This high temperature is not explained by chemical exothermic reactions or by external power input instability.

If those should be led, as calculation about specific power needed to have such fusion spots say, to *cold fusion phenomena*, then a different configuration of the cell, more suitable for heat extraction and robustness of Nickel layer should be easy to prepare for excess power measurements and quantification.

We are actually working on the design of a new cell configuration with a completely integrate calorimeter (microbolometer) able to detect any small temperature increase (even locally) through thermal emissions measurements. This control system may be in the same cell environment and electrically connected to the input power control system to guarantee maximum system stability.

The actual cell dimensions are in the range of a small integrate silicon chip, because the purpose of the experiments is to realise a power generation device suitable for portable electric apparatus, but it is in principle completely scaleable for high power generation.

**Introduction:** solid and gaseous Hydrogen sources have been used to study the behaviour of thin Nickel film layers heated over the Curie temperature in presence of Hydrogen.

The structure showed in the enclosed schematic cross section (Fig. 1), has been realised on the surface of a small silicon chip of  $2x3 \text{ mm}^2$  area. On a side of the chip there is a heater resistor done by N+ doped polysilicon deposited on 2  $\mu$ m of silicon dioxide. On that heater, separated by a 2  $\mu$ m of silicon nitride layer, there is a 0.1  $\mu$ m thick Nickel resistor. On the other side of the chip there is, directly deposited on the same silicon dioxide, an identical Nickel resistor (used as reference).



The electrical connection allows to heat the silicon chip using either the silicon resistor or the Nickel resistors having so several biasing conditions. To reduce at minimum the power dissipation, the chip is suspended into a ceramic minidip package using only the aluminium bonding wires without any kind of die attach between the silicon chip and the ceramic package.

A suitable hermetic stainless steel chamber has been set up where is possible to put the sample and reduce the pressure below  $10^{-3}$  torr; in this way it is possible to minimise the power needed to reach the working temperature.

In these conditions using a power ramp up and monitoring the Nickel resistance it is possible to calculate easily the Thermal Resistance of the system and correlate the heating power with the temperature. The power usually needed to increase the temperature of 1  $^{\circ}$ C has been around 0.5 mW.





The curve has been plotted using a power ramp up and monitoring the electrical resistance of the Nickel resistor placed just over the heater. The power needed to reach the transition point allows an easy calculation of the cell thermal resistance Rth.

It is useful to remember that the system, as it has been realised and because the material used, does not work if there is not extraction of hydrogen from the solid source material included between the heater and the nickel layer. This extraction is activated by the temperature [1],[2].

### Experimental design, procedure, results.

The aim of a first group of experiments was the study of several aspects of the Nickel behaviour when the temperature activates the Hydrogen release from the solid source.

### **1.** Nickel resistivity versus temperature.

It is well known that Nickel shows a non linear behaviour from temperature below 20 °C till 360 °C, where is positioned the Curie temperature (Tc) and where the Nickel looses the ferromagnetic properties [3]. Over that temperature the resistivity continues to rise linearly.

Just using the power needed to reach the Curie temperature it is quite easy to determine the thermal resistance (Rth) and then the temperature of the nickel as function of the input power. In fact it is not more possible to use the resistivity of the Nickel to know the temperature because it changes when the Nickel Hydrogen absorption begins.

#### 2. Resistivity variations with hydrogen absorption.

Once the cell temperature has been fixed, the resistivity variation, if no oxidation phenomena occur, should be due to the hydrogen absorption in the Nickel lattice; hydrogen being available from Si-H and N-H broken bonds. In the plasma deposited silicon nitride the amount of these bonds is very high (more than 10% of total bonds) [2].

The high temperature of the cell is necessary to have enough H+ generation in the solid source.

If we suppose an exponential generation rate like:

 $G(H+)=Go \exp -Ea/KT$  and the main part of H+ produced being absorbed by the Nickel, we should have an exponential variation of the resistivity versus the temperature and a linear variation versus the time. This has been observed in our experiments and the value of the activation energy Ea is closed to what reported in the literature (0.5 - 0.7 eV) [2].





Using a fixed stressing time of 600s it has been stressed at three different temperatures the structure heater/solid source/Nickel resistor. Going back to the ambient temperature it has been measured the R/Ro value for the Ea calculation. It is in the range of 0.5/0.7 eV.

### 3. Nickel stability versus thermal cycles.

The reference Nickel resistor realised directly on silicon dioxide reaches the Curie temperature with the same power of the resistor on the silicon nitride. This means that the temperature over all the chip is uniform (the system under vacuum helps for that).

Taking as reference a first cycle as showed in Fig. 4. A new identical cycle is shifted towards R/Ro higher values (Ro is the initial value of the first cycle).

The reference resistor after identical thermal cycles does not show the same shift because higher distance from the hydrogen solid source. Some shift has been noticed only after long time at high temperature correlated with crystallographic modification of the layer structure.



After a ramp up to 500 °C and stressing for 600s the solid source cell a second cycle is shifted towards higher R/Ro values indicating a resistivity change of the layer.

#### 4. Nickel cell behaviour in Hydrogen atmosphere.

With the purpose to confirm what observed in the experiments with the hydrogen solid source, a second group of experiments have been performed filling the chamber with Forming gas (5% of H2 in N2) and monitoring the temperature and the resistance of the Nickel resistors. In this case the absorption can occur also at lower temperatures being the Hydrogen available in the gaseous phase.

The tests done with H2 gas source required much more input power to reach the same temperature used during solid source experiments. This was due to the increased gas pressure inside the chamber that reduced of about one order of magnitude the thermal resistance Rth of the cell.

In spite of that, together with the modification of the nickel resistance, indication of hydrogen absorption, in all the experiments occurred high temperature spots breaking the resistor under test. The sudden occurrence of the resistor interruption did not allowed quantitative measurements neither of excess heat production nor of activation energy. The nickel resistor usage as heater seems to accelerate the hot spot occurrence.

By the way the hot spot occurred also in resistor branches where no current was flowing. In all cases, especially when the resistor interruption is in areas where no current was flowing, it is impossible to justify the observed melting of the Nickel layer (1453 °C) thinking to a temperature rising through chemical reactions. It is necessary an energy density 2/3 order of magnitude higher.

From this point of view the cell prototype used for these experiments, designed to point out only the electrical Nickel behaviour and not excess heat coming from nuclear reactions, was very useful to analyse the resistivity of the Nickel resistor versus the temperature, but extremely fragile versus excess power occurrence, at such a point that was impossible to maintain the cell working as long as we need to try different bias control conditions.

The design of the cell was really bad concerning the possibility of power extraction from the Nickel layer: the layer is between two very low thermal conducting materials. We are actually working on a new cell design to overcome this kind of concern.

# Conclusions

The experiments performed using the cell prototype described above, showed that is possible under certain temperature and biasing conditions, to induce absorption of Hydrogen into the lattice of a thin Nickel layer. The Hydrogen can be absorbed from a solid source or from a gaseous source and it changes the electrical resistivity of the Nickel layer. In all the experiments we had, early or late, the physical interruption of the Nickel resistor due to the melting of part of it. These phenomena can not be explained thinking to instability of power supply or exothermic chemical reactions. If, as calculation about energy density authorise to hypothize, this power spots may be attributed to "cold fusion" phenomenology, the design of a new cell suitable for excess power extraction should be quite easy. In that case having thin layer over a more efficient heat sink material should be the ideal condition for very high yield and controllable "cold fusion" processes.



Fig. 5 Nickel resistor showing melted area.

## References

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## Acknowledgements

The author is indebted to Stefano Manzini for technical assistance in cell design and automatic data monitoring and collection; to Francesco Celani for fruitful discussions and ideas exchange.