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The 2007 Edward G. Weston Summer Research Fellowship — Summary Report

Electrochemical Synthesis of Nanothermocouples

by **Aleix G. Güell**

The thermocouple is one of the most commonly used devices for temperature sensing, due to its simplicity and easy integration into electronics. Basically, a thermocouple functions as follows: two different metallic wires in electric contact at one single point generate an output voltage that depends on the difference of temperature between the *hot junction* and the *cold junction*. Recently, it has been demonstrated that thermocouples of nanometric dimensions led to faster response times than conventional ones,¹ opening a wide range of new applications in the fields of photonics, renewable energies and biosensing.

In this work, the dimensions of the thermocouple has been further reduced by using lithographically patterned nanowire electrodeposition (LPNE).² This method allows the synthesis of nanowires with a high control of the nanometric dimensions of their cross-section, and also their position and macroscopic shape. It is also the most suitable method for the design of complex nanowire devices and it is easily mass-scalable.

Our devices consist of two crossed nanowires made of gold and palladium because those metals present a high Seebeck coefficient difference, high chemical stability, and could be used in a wide range of temperatures. As described elsewhere,² the synthesis of these nanowires started with an initial nickel evaporated layer on glass partially covered by a photoresist layer that is etched by nitric acid. After some minutes, the etching process takes place also underneath the photoresist, generating a nanoband. This nanoband is filled by the electrodeposition of the first metal, in our case gold. A complete removal of the photoresist layer and the remaining nickel layer left the electrodeposited gold nanowire on glass. Repeating all this process with the second metal, Pd, led us to the final device: a crossed Au nanowire and Pd nanowire with a nanoheterojunction.

Figure 1 shows one of these heterojunctions between two single nanowires, a gold one of 80 nm, and a palladium one of 250 nm. Nanowire dimensions can be controlled as desired by setting different electrodeposition parameters.

As a proof-of-concept that devices like the one shown in Fig. 1 behave as a thermocouple, we measured their output voltage at different temperatures (see Fig. 2). The experimental data has been fitted to a lineal behavior, with a slope of about 10.5 $\mu\text{V}/^\circ\text{C}$. This slope corresponds to the sensitivity or Seebeck coefficient of the thermocouple, and it is in agreement with the theoretical value of this Au-Pd couple.^{3,4}

The devices present a high reproducibility and stability with time and use. Even after iterated heating-cooling cycles, the responses presented in Fig. 2 are stable and no hysteresis has been detected.

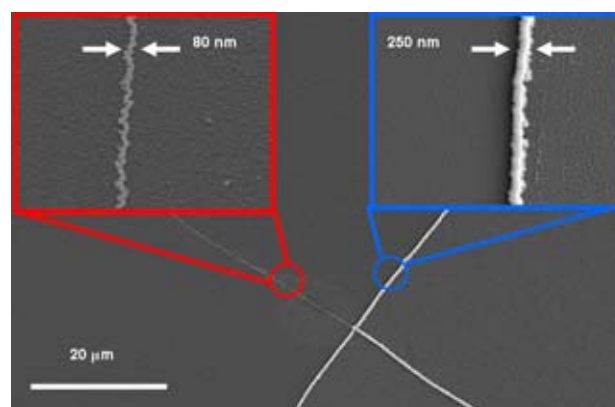


FIG. 1. Scanning electron microscopy of a LPNE nanothermocouple. The red and blue boxes are magnifications of the gold and palladium nanowires, respectively.

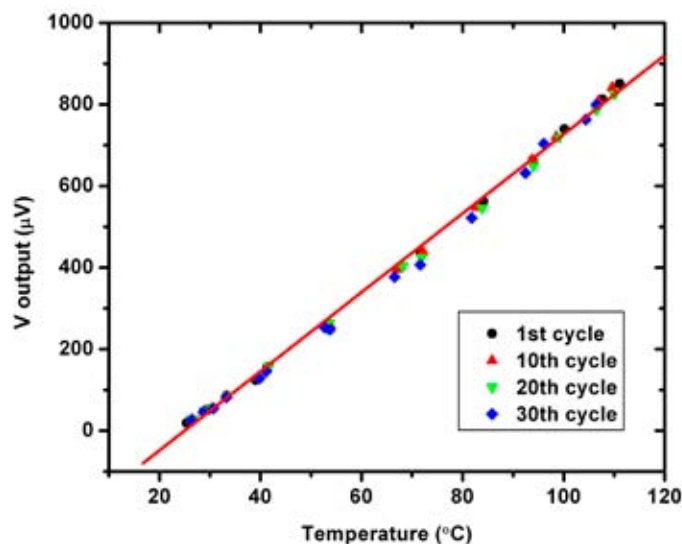


FIG. 2. Calibration curve of Au-Pd LPNE nanothermocouples. Data from the first, tenth, twentieth, and thirtieth cycles are presented. The red line corresponds to the lineal fitting of the data.

In summary, this work presents a proof-of-concept of the first nanothermocouple synthesized by LPNE nanowires. Future experiments are being designed to determine the response time of these nanothermocouples, and also to establish a relationship between properties and nanowire dimensions.

Acknowledgment

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References

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About the Author

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The 2007 Colin Garfield Fink Summer Research Fellowship — Summary Report

Fabrication, Structure, and Electrochemistry of Iodated Microporous Carbons of Low Mesoporosity

by Prabeer Barpanda

Electrochemical double layer capacitors (EDLCs), often known as supercapacitors, are increasingly popular in various energy storage applications ranging from consumer electronics to hybrid electric vehicles.^{1,2} EDLCs employ high surface area, mesoporous activated carbons, in which charge storage is primarily due to the formation of electrostatic non-Faradaic double layer reaction and interfacial charge separation. In recent years, there has been ongoing and successful effort to produce high capacity carbon electrodes by careful tuning of the meso- and micro-porosity of the carbons. We have recently focused on the improvement of the volumetric capacity of such carbons for intermediate time constant applications. In an alternative approach, we have reported significant rise (200-500%) in volumetric capacity of activated carbons by iodine modification.^{3,4} We have shown iodination of activated carbon indicates the formation of carbon-polyhalide compounds due to charge transfer (CT) reaction,

thereby altering the existing physical and electrochemical properties of carbon in the non-Faradaic development of reversible Faradaic reaction. The polyiodides were preferentially located in the micropores. The focus of this work is to study the fabrication, validation and physico/electrochemical properties of a microporous carbon from low cost precursors.

Microporous carbon (IUPAC definition: 0-2 nm) was prepared from coal following chemical activation method.⁵ Anthracite coal was grounded and mixed with KOH solution in water at 60°C for 2 h. The solution was dried overnight at 110°C. The resulting slurry was taken in an alumina crucible and was annealed at 750°C for 1 h for activation in nitrogen atmosphere. The annealed powder was mixed with HCl solution to neutralize the existing KOH. Upon drying the powder overnight, pure microporous carbon was successfully obtained.

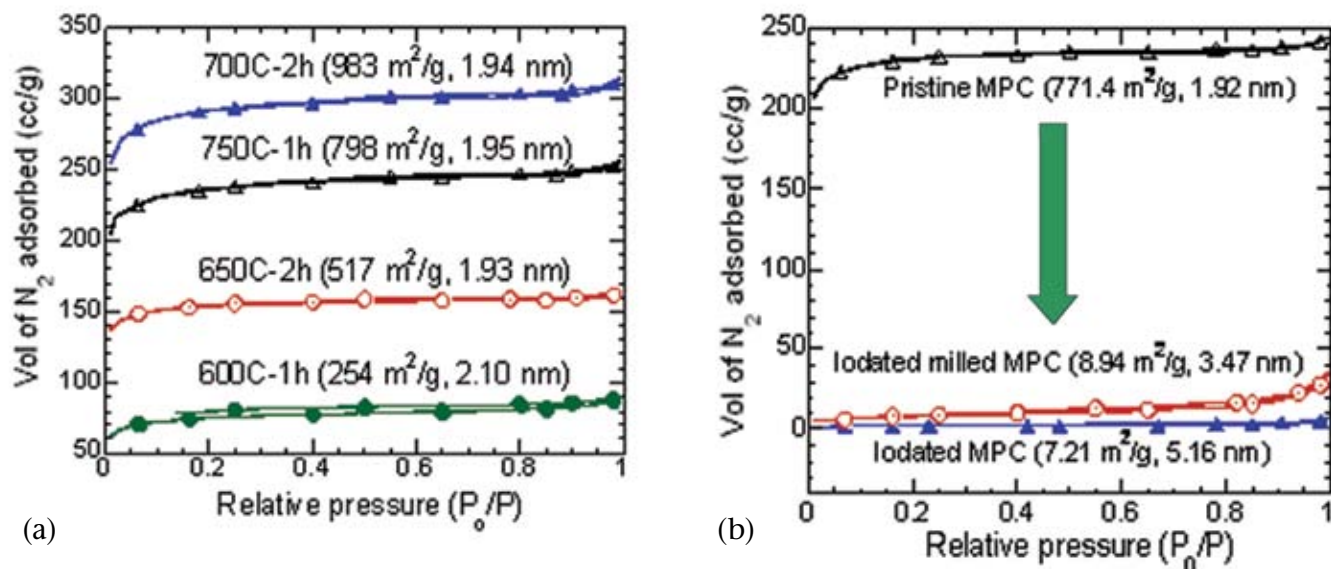


FIG. 1. (a) The BET isotherms of microporous carbons produced at different activation condition (annealing temperature and soaking time). They resemble the IUPAC type-I pattern proving the presence of only microporosity. (b) The BET isotherms showing the drastic decrease in N₂ adsorption isotherm upon iodination (as shown by arrow) indicating the filling of micropores with iodine particles. (The numbers in brackets indicate surface area and average pore size respectively.)