Journal of ELECTRICAL ENGINEERING, VOL. 60, NO. 6, 2009, 346–349

COMMUNICATIONS

WIRELESS TEMPERATURE MEASUREMENT IN THE HOT FILAMENT CVD REACTOR FOR DEPOSITION OF CARBON NANOTUBES

Michal Kolmačka [*] — **Magdaléna Kadlečíková** [*] — **Juraj Breza** [*] — **Filip Lazišťan** [*] — **Lenka Zajíčková** [**] — **Marek Eliáš** [**] — **Karol Jesenák** [***] — **Katarína Pastorková** [***]

The experimental work deals with measuring the temperature in the hot filament chemical vapour deposition (HF CVD) reactor by a purpose-built electronic circuit. The objective is to optimize the substrate temperature, one of the key technological parameters in the synthesis of carbon nanotubes. The main outcome of the work is the design and construction of a functional thermometer, its precision calibration and improved accuracy of temperature measurement.

K e y w o r d s: Carbon nanotubes, chemical vapour deposition, isolation amplifier

1 INTRODUCTION

The amazing properties of carbon nanotubes (CNTs) make them a very popular material for various applications. The main problem is their effective preparation. At present, CNTs are prepared by diverse methods, most often by employing an arch discharge, laser evaporation and chemical vapour deposition. To make the fabrication process efficient and suitable for creating CNTs of desired properties, it is necessary to optimize the deposition conditions. The topical objective is to optimize the substrate temperature, one of the key technological parameters in the synthesis of CNTs.

2 TECHNICAL REQUIREMENTS

Deposition of CNTs is carried out in a hot filament chemical vapour deposition reactor, where the precursor is activated by five tungsten filaments heated up to 2200 °C. The working atmosphere is a mixture of methane and hydrogen. Previous experiments confirmed these best preparation parameters: deposition time from 2 to 30 minutes, pressure 2 or 3 kPa, temperature 600 °C, and the presence of plasma bias. Previously, the substrate temperature used to be checked by two thermocouples placed in the substrate holder. The measured voltages were connected from the rotating holder via ring contacts to the input of an amplifier. During deposition, the temperature was measured with accuracy of 100 °C. The status is shown in Figs. 1 and 2.

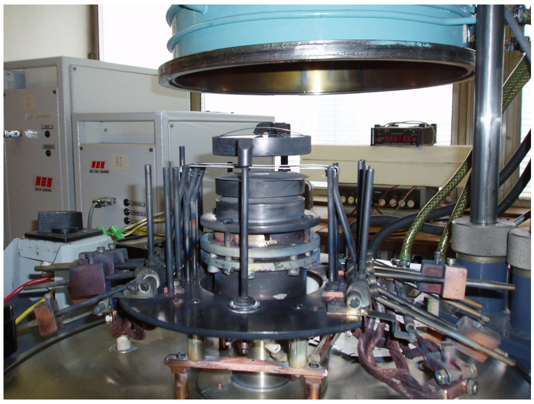

Fig. 1. Interior of the HF CVD reactor - One can see the gas shower, tungsten filaments in molybdenum holders and a rotating substrate holder.

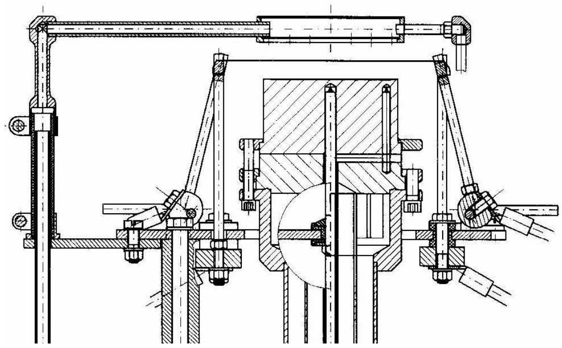

Fig. 2. Detail of the upper part of the holder - Two thermocouples are placed in the cavity of the holder.

It was inevitable to remove the leakages due to the conductive connection of the thermocouple with the holder

[*] Department of Microelectronics, Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Ilkovičova 3, 812 19 Bratislava, Slovakia, juraj.breza@stuba.sk [**] Department of Physical Electronics, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic, [***] Department of Inorganic Chemistry, Faculty of Natural Sciences, Comenius University, Mlynská dolina CH-2, 842 15 Bratislava, Slovakia

ISSN 1335-3632

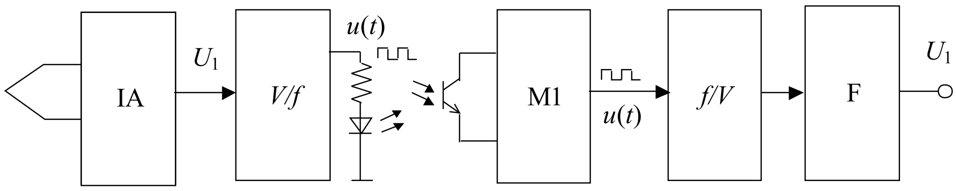

Fig. 3. Block scheme of the measuring assembly: IA -isolating amplifier, U/f (f/U) - voltage-to-frequency or frequency-to-voltage converter, M1(2) - optical-to-electrical signal converter, F - filter

(most often by carbon black from the atmosphere). This is why the new gauge is based on a circuit with an isolation amplifier and optical signal transmission, whereby the effect of the contact resistance on the copper contacts of the rotating part of the reactor below the vacuum chamber is eliminated.

For the purpose of separating the thermocouples from other parts of the measuring electronics it is necessary to use an isolation amplifier (IA) with a linear characteristic in the whole range of voltages. The IA should be able to isolate voltages up to 200 V or even higher. The IA will replace the amplifier presently installed in the plant. Therefore, its amplification and offset must match with those of the present amplifier. Nonlinearity of the transfer characteristics should not exceed 0.1 %. The input of the A/D converter has a range from 0 to 10 V and the amplification is set so as to allow measuring the temperatures up to 2000 °C. At a temperature of 1000 °C, when the thermocouple generates a voltage of about 9.6 mV, the present amplifier has an output voltage of approx. 5 V.

After comparing with similar products, we chose the integrated circuit HCPL-7840 [1] that is an isolation amplifier with optical coupling. The circuit works on a modulation principle, therefore it requires a well balanced differential filter at its input. On properly setting the time constant of this filter, the circuit is applicable as an isolation amplifier for the thermocouple. However, the HCPL-7840 is designed for voltages in the range ±200 mV, hence it is inevitable to insert an operational amplifier at its input so as to utilize the whole range of voltages of the HCPL-7840. The catalogue declares states nonlinearity about 0.0037 % in the range of input voltages ±200 mV.

A simple method how to create wireless two-channel transmission of thermovoltages consists in converting these voltages into a frequency of pulses and their transmission from the rotating part of the holder to the static part by optical coupling. The two channels are easy to distinguish as one of them uses IR radiation, the other one optical radiation. Voltage-to-frequency and backward frequency-to-voltage conversions must be performed with high linearity as this determines the accuracy of temperature measurement. For our purposes we used a converter consisting of the integrated circuit VFC32 [2] and several external resistors and capacitors.

The output signal of the f/U converter is not a smooth DC voltage. The shape of the signal depends on the input frequency to be converted into the voltage. In general the signal contains a DC component onto which a triangular voltage is superimposed with a linear growth and exponential decay of the voltage. The time and nonlinearity of the tail edge depend on the frequency of the signal at the input of the f/U converter. The input of the A/D converter should only be fed by the DC component, thus by the average value of the signal. This is obtained by means of a low-pass filter that suppresses all superimposed harmonic components in the signal.

3 CONSTRUCTION DETAILS

The block scheme of the temperature measuring system satisfying the requirements discussed above is shown in Fig. 3. Detail circuitry is given in Fig. 4 and the list of devices was published in [3]. The circuit board was designed in EAGLE 4.11. Finally, a photograph of the functioning temperature measuring system installed in the HF CVD reactor is shown in Fig. 5. The supply voltage for both input and output circuits is 5 V. The voltage regulator 78L05 [4] was employed as a stabilizer of the supply voltage.

Ultra-precision op-amp OP177 [5] in inverting connection was used as a preamplifier. The negative feedback was accomplished by means of a multiturn trimmer allowing to set the gain. In the feedback there is also a parallel capacitor whereby the preamplifier acts simultaneously as a low-pass filter.

On the input of the preamplifier there are two voltage dividers, one of them with multiturn trimmer P1. This circuit solution is a modification of the connection recommended in the catalogue list of the OP177. Passive temperature compensation of the so-called cold end was omitted because the measuring system itself contains temperature compensation. Therefore the Si diode in the preamplifier is not needed and by its removal the circuitry was simplified into the applied form. The gain of the preamplifier is approx. 20.

The preamplifier along with the input part of the HCPL-7840 must be fed by a symmetrical supply with a galvanically separated input and output. For this purpose we used a DC/DC converter FDD03-15D [6]. It is applicable in a relatively broad range of voltages, 20 to 60 V, and at its output it delivers a voltage of ± 15 V needed to supply the preamplifier with OP177. It can create a current of 100 mA, its isolation resistance is 1 GΩ and isolation voltage 1500V ± 10 %. The converter contains its own impulse generator controlling the electronic trigger in the input circuit of the converter and with every wave it takes a relatively large current form the supply,

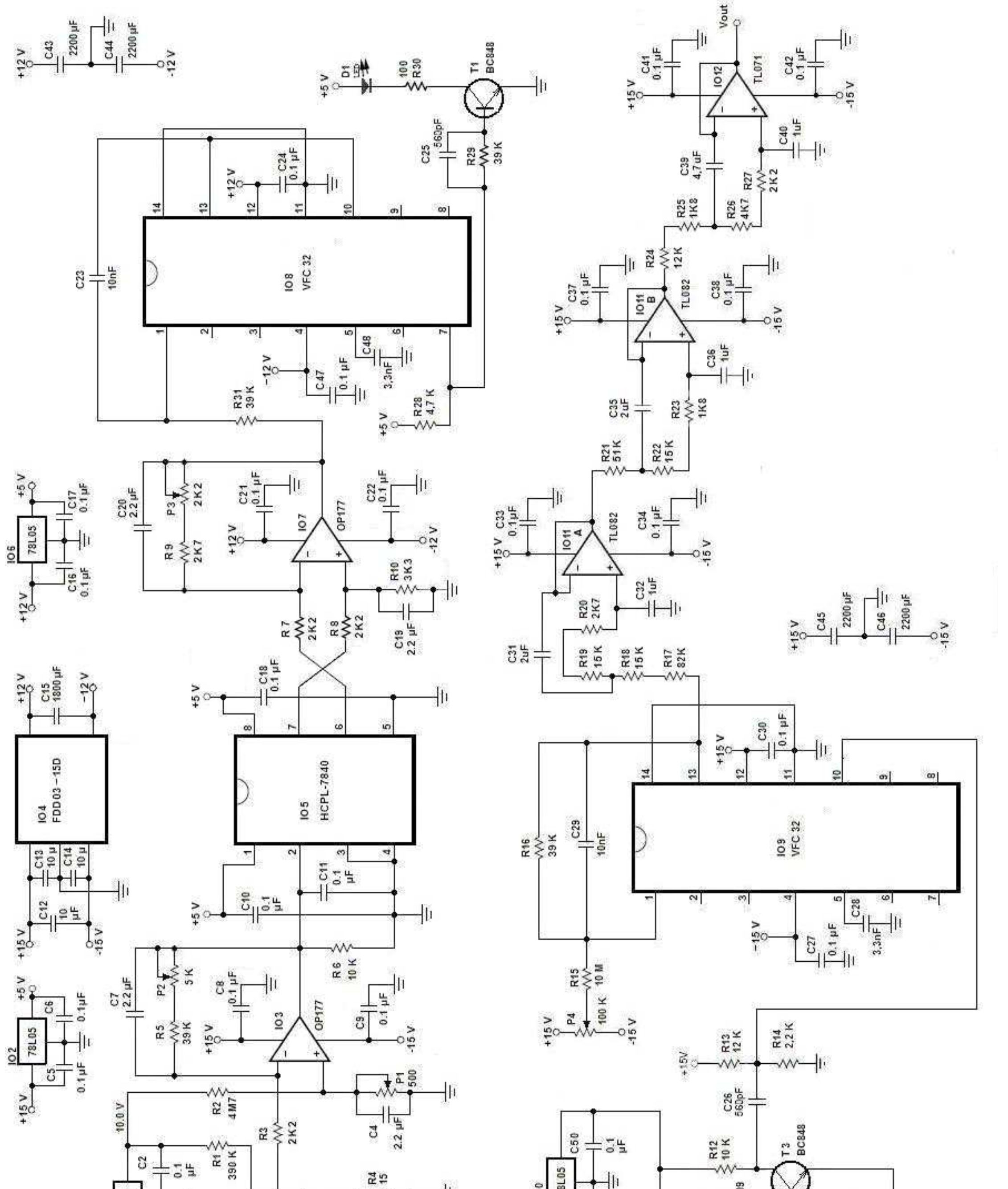

Fig. 4. Circuitry of the temperature measuring assembly - Integrated circuits: IO1 = 78L10; IO2, IO6, IO10 = 78L05; IO3, IO7 = OP177, IO4 = FDD03-15D; IO5 = HCPL7840; IO8, IO9 = VFC32; IO11 = TL082; IO12 = TL071

which results in a drop of the voltage due to the parasitic impedance of the leads. To eliminate the voltage drop it is necessary to insert at the input of the DC/DC converter an electrolytic capacitor with a capacitance of at least 1800 μF as determined empirically. At the output of the converter there is a 10 μF tantalum capacitor.

The differential filter at the output of the HCPL-7840 consists of the op-amp OP177 with resistors R5 and R6 and trimmers R3 and R4 allowing to balance the device. The output of the differential filter is fed directly to the integration element of the U/f converter of the circuit VFC32. The converter employs the method of the so-called charge balancing to convert DC voltage into frequency. It consists of three basic parts: integrator, comparator (two op-amps) and a controlling circuit that, based on the signals from the comparator, switches the output transistor on or off and discharges the capacitor in the feedback of the integration element.

By varying the values of the external elements one can set the frequency range and the pulse duration. The output transistor with an open collector is in the integrated circuit VFC32. It requires a supply voltage of 5 V through a 4.7 kΩ resistor. The collector current in the switched-on state is roughly 1 mA. The catalogue contains a chart for setting the values of external elements for single frequency ranges. In the selected frequency range from 0 to 10 kHz the converter has a nonlinearity below 0.01 %.

The emitter of light pulses for the optical channel consists of a high luminosity LED. For achieving its sufficient brightness a current of 15 mA should be flowing through the LED. Because of this reason a switch was inserted at the output consisting of transistor BC848 [7]. Additionally, without using the transistor switch the light signal would be inverted. Since at the output of VFC32 there is an open collector of a bipolar transistor, this collector must be connected to the supply voltage (+5 V for TTL logics) through a so-called pull-up resistor. The receiver of light impulses consists of phototransistor SFH309FA [8] connected in series with the resistor at the input of the transistor switch with BC848.

At the output of the switching transistor there is a differentiation element. The received light impulses are hereby changed into short triggering signals that control the comparator in the integrated circuit VFC32. This circuit can also be connected as an f/U converter. This converter, however, suffers from the "bug" mentioned above. At its output it does not deliver a smooth voltage but a triangular voltage superimposed on a DC component. The frequency of the triangular component is the same as the frequency of the trigger impulses controlling the comparator of the f/U converter. For achieving a smooth DC voltage at the output of the converter it is necessary to use a filter. The transfer by means of the frequency of impulses is set in such a way that there is always the same voltage at the output of the filter as the voltage at the output of the differential filter of the isolation amplifier. Amplification of the whole assembly is the same as that of the older amplifier in the HF CVD reactor. The output voltage of the device is connected by a lead to the output node of the initial amplifier.

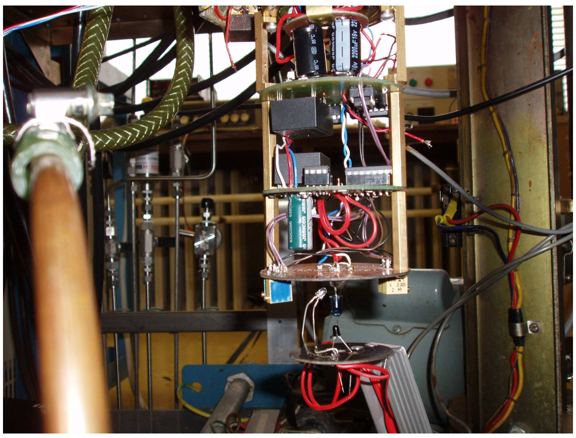

Fig. 5. Temperature gauge with wireless signal transmission. The substrates are inserted as replaceable segments into the lower part of the reactor. Transmitting diodes and tuning trimmers can be seen at the bottom.

4 CONCLUSION

The method of measuring the temperature in the HF CVD reactor has been changed fundamentally. The new measuring system is based on a circuit with an insulating amplifier and optical transmission of the information on temperature via optical elements. The insulating amplifier has a linear transfer characteristic and insulation ability up to 250 V. The amplifier is built using a linear optocoupler consisting of an AlGaAs infrared LED radiating on two output PIN photodiodes, one of them being feedback connected. The transmitter is an electronic circuit employing the modulation principle in which the amplified voltage is linearly converted into the frequency of optical impulses. The receiver converts the frequency of optical pulses to corresponding voltage levels. Then the signal is processed by a filter and fed to the input of an A/D converter.

Calibrations showed that the measurement of temperature was carried out with inaccuracy of ±5 °C in the temperature range from 100 to 300 °C and with inaccuracy of ±10 °C in the temperature range from 300 to 600 °C. Above this temperature, the inaccuracy is ±20 °C

Acknowledgement

This work has been supported by grants VEGA 1/0746/09 of the Ministry of Education of the Slovak Republic, and MEB CZ-100/SK-CZ-0078-07 (NANOCARBON) of APVV and by the Czech Ministry of Education under the project MSM 002162241 and by the Grant Agency of the Czech Republic, contract 202/07/P523.

References

[1] http://www.datasheetcatalog.org/datasheet/hp/HCPL-7840.pdf.

[2] http://www.noise.physx.u-szeged.hu/Education/Dlabor/Docs/VFC32.pdf.

[3] KOLMAČKA, M.: Diploma Thesis, Slovak University of Technology in Bratislava (2007).

[4] http://www.datasheetcatalog.org/datasheets/208/402911_DS.pdf.

[5] http://www.analog.com/en/amplifiers-and-comparators/operational-amplifiers-op-amps/OP177/products/product.html.

[6] http://www.alldatasheet.com/datasheet-pdf/pdf/125588/ETC/FDD03-15D.html.

[7] http://www.datasheetcatalog.org/datasheet/kec/BC846_7_8.pdf.

[8] http://www.alldatasheet.com/datasheet-pdf/pdf/45657/SIEMENS/SFH309FA-5.html.

Received 9 April 2009

Michal Kolmačka (Ing) born in Detva in 1984, graduated in microelectronics in 2007. Currently pursuing his PhD study in microelectronics at the Faculty of Electrical Engineering and Information Technology, Slovak University of Technology in Bratislava. His field of study are carbon nanotubes, their growth and analysis. His research interests include construction of new electronic circuits with sensors, too.