Construction and characteristics of a thermometer in a hot filament CVD reactor for synthesis of nanocomposites based on carbon nanotubes

Magdaléna Kadlečíková, Michal Kolmačka

This study presents the crucial parts of the final construction of a combined temperature meter with wireless transmission of temperature data. The wireless transmission of temperature information is realized by optical coupling between the photodiode and the phototransistor through the air, not through an optical fibre. The power source is structurally unique in that its output terminals have mechanical freedom and thus the possibility to rotate without using mechanical contacts. Transmission of the supply energy is mediated by the magnetic field in a pot core transformer. A linear symmetrical post-regulator stabilizing the output voltage and ensuring the symmetry of the output voltages is included at the output of the source.

Keywords: chemical vapour deposition, nanocomposites, carbon nanotubes, switched mode power supply, pot core transformer

1 Introduction

The main objective of the research is the synthesis of nanocomposites based on carbon nanotubes in a hot filament chemical vapour deposition reactor (HF CVD). The reactor operates on the principle of pyrolysis of methane and hydrogen. The experimental material is placed on a molybdenum holder, several (7 to 10) millimetres below hot (2200 °C) tungsten filaments and its temperature during synthesis is optimized to approximately 600 °C. Design of the temperature gauge in the HF CVD employing a method of sensing the thermal voltage from ring contacts was published in [1]. The reconstructed measuring system was 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 was built using a linear optocoupler consisting of a LED radiating on two output PIN photodiodes. The temperature meter has a preamplifier and an isolation amplifier that are galvanically isolated. This isolation was achieved by DC/DC converters. Reconstruction of the supplying circuitry made the whole transmitting part independent and completely separated from all other circuits of the measuring system. During operation of the facility a severe drawback was encountered, namely instable supplying resulting in temperature fluctuations in the reactor. This is why another solution and reconstruction [2, 3] were chosen utilizing a switched mode power supply.

The measurement string was simplified by removing the mobile contacts and isolation amplifiers that behaved incorrectly when they became overheated and distorted the data on temperature.

The result of the present work is the design and second reconstruction of the circuitry supplying the temperature meter in the HF CVD reactor. The design of the power supply is unique. The output terminals have mechanical freedom and hereby the possibility of rotation without the ned to use mechanical contacts. Transmission of supply energy is mediated by the magnetic field in the pot core transformer.

2 Experimental details and results

The temperature sensor of the holder rotating at a constant speed during the synthesis is an N-type thermocouple placed under the holder. The generated thermal voltage is amplified and converted to the frequency of voltage pulses. The focus of the design of the electronic temperature meter is the construction of a switched voltage source which includes a balancing electronic circuit [4]. Figure 1 shows the realized construction of a switched voltage source, the key component of which is a pot core transformer whose circular geometry is used to transmit the electrical signal. The transmission of the supply energy is mediated by the magnetic field in the pot core transformer. Two isolated windings wound on a separate frame of the pot core transformer form the power source for the linear

Faculty of Electrical Engineering and Information Technology, Slovak University of Technology in Bratislava, Ilkovičova 3, 812 19 Bratislava, Slovakia magdalena.kadlecikova@stuba.sk

https://doi.org/10.2478/jee-2024-0030, Print (till 2015) ISSN 1335-3632, On-line ISSN 1339-309X

© This is an open access article licensed under the Creative Commons Attribution-NonCommercial-NoDerivatives License (http://creativecommons.org/licenses/by-nc-nd/4.0/).

regulator. The secondary windings are connected via the J1-J2 connector to the printed circuit board. The circuit of a linear DC voltage regulator follows ensuring the symmetry of the output voltages on the ± 12 V terminals

of the J3-J4 connector with an error of 0.5 V, which is a permissible symmetry error. The balancing circuit ensuring the symmetry of the output voltages is implemented as described in the diagram show in Fig. 2.



(a) Winding of the static (primary) side of the pot core transformer.



(c) Exposed primary part of the switching source. The static part of the transformer together with the phototransistor is connected to the switching source by a cable. The arrow shows the safety element of the source – the fuse.



(b) Here, in the rotary part, is the secondary side of the transformer and a linear regulator stabilizing the output voltage at ± 12 V.



(d) Signal transmission from the photodiode to the phototransistor. The phototransistor in the static part is illuminated by an orange LED emitting light pulses in the axis of rotation. The LED rotates together with the electronic part.

Fig. 1. Current realization of the electronic temperature meter of the rotating sample holder in the reactor chamber for chemical vapour deposition of carbon nanotubes and synthesis of nanocomposites



Fig. 2. Circuit diagram of the DC voltage regulator. In the grey frame there is a circuit ensuring the symmetry of the output voltages on the +12 V and -12 V terminals of the J3-J4 connector.

The voltage source for the linear regulator consists of two windings isolated from each other wound on a separate frame of the pot core transformer. The number of turns of both secondary windings $N_s=17$ is the same as the number of turns of the auxiliary winding. The secondary windings are connected via the J1-J2 connector to the printed circuit board, where damping RC elements with resistors R1, R2 and capacitors C3, C4 are included. These have the task of reducing the number and frequency of oscillations after switching on the switching transistor, as the capacities of the terminally polarized rectifier diodes D1 and D2 create a resonant circuit with the stray inductance of the transformer. Electrolytic capacitors C1 and C2 perform the function of collecting capacitances to maintain the potential of the output voltages during the switching on of the switching transistor. Electrolytic capacitors generally have parasitic series resistance, due to which the voltage at the terminals of these capacitors contains a larger ripple of different frequency components superimposed on the DC voltage. LC low-pass filters with L1, L2, C5, C6 are included to filter out this ripple. To reduce the quality of these filters, damping RC members with R23, C15 and R24, C18 were added to their outputs. The output voltage after filtering is used to power a symmetrical regulator.

The regulator consists of voltage references Ref1 and Ref2 formed by the LM285 circuit. The reference value of the voltage is 1.25 V. These references are fed through the primary resistors R3 and R4, and the capacitors connected in parallel to the references perform the function of filtering ripples or under-oscillations from power sources. Reference voltages are compared by operational amplifiers LM2904 (IC1A/B) with output voltages of resistor dividers formed by resistors R10, R11 and R19, R20, which sense the voltages at the regulator outputs. For the stability of the controller, frequency compensation in the form of RC members C7, R8 and C10, R14 is necessary, which adjust the frequency characteristic so that the phase safety is optimal.

The values of these RC components are determined based on simulations of frequency characteristics as well as analysis of the controller's time response to deviation from the equilibrium state using step changes in the load current. Capacitors C13 and C14 are the suppression capacitances on the IC1A/B power supply. The output voltages from IC1A/B, as a result of the comparison, go to the inputs of the current-enhanced voltage followers with a current limit of approximately 70 mA. These voltage followers consist of complementary transistors PBSS4560 and PBSS5560 (T2 and T8) which are driven by current sources. These current sources consist of transistors T1 and T7 with a bias voltage on their bases created by diodes D3, D4 and D11, D12. Source currents are defined by the values of emitter resistors R5 and R17. The excitation current for these current sources is created by resistors R6 and R18. Transistors T2 and T8 are de-excited by transistors T3 and T6. Diodes D5 and D8 perform the function of disconnecting the error amplifiers in the event of a short circuit at the output of the regulator in order to protect the base-emitter transitions of transistors T3 and T6 from high reverse voltage. Resistors R9 and R16 also aid in this protection by lowering the base-emitter voltage of the respective transistors. Resistors R7 and R15 increase the efficiency

of frequency compensation of error amplifiers due to the voltage drop that occurs on them and, together with capacitors C19 and C20, form low-pass filters improving the suppression of interference from the power supply for higher frequencies.

Diodes D6 and D9 perform the function of protecting transistors T2 and T8 from high reverse base-emitter voltage. Capacitors C8 and C11 fix potentials at the output of the regulator and help stabilize its output voltages. Transistors T4 and T9 biased by the diode voltage of diodes D7 and D13 together with resistors R12 and R21 sense and evaluate the symmetry of the output voltages of the regulator. If a voltage drop of more than $\approx 200 \text{ mV}$ occurs on one of the output voltages of the regulator, one of the transistors T4 and T9 is activated, the collector current of which is strengthened by the transistor T5 or T10 and deexcites the power transistor on the opposite source so that the symmetry of the output voltages of the regulator is maintained. Resistors R13 and R22 serve to reduce the sensitivity of the balancing detection to its output currents less than 8 µA. Capacitors C5 and C12 serve to stabilize the balancing element. Symmetrical voltages together with the positive output of the 14.5 V switching power supply are output from the printed circuit board using connectors J3-J4. Maintaining the symmetry of the output voltages of the linear DC voltage regulator which has the task to stabilize the voltages at \pm 12V with accuracy better than 5% is patented [5].

3 Conclusion

The implemented power source is structurally unique in that its output terminals have mechanical freedom and therefore the possibility to rotate without using mechanical contacts. The transmission of the supply energy is mediated by the magnetic field in the pot core transformer. The rebuilding of the transmitting part also allowed to implement compensation of the heating of the ends of the thermocouples and symmetrizing the output voltages and protection against overcurrent. The switching source with a flyback topology works with current mode control. A linear symmetrical postregulator stabilizing the output voltage at ± 12 V with voltage regulation accuracy of $\pm 5\%$ is included at the output of the source, ensuring the symmetry of the output voltages even in the case of a forced drop of one of these voltages. Voltage symmetry is sufficient in the range from 2 V to full voltage.

The electronic temperature gauge is characterized by two decisive electrical characteristics, namely the stability of the feedback of the flyback converter, since after step changes in the load current its output voltage does not show any damped oscillations and a sufficient phase reserve for the stability of the post-regulator even for the no-load condition of operation. The result of the calibration process is an interval estimate of the combined uncertainty of temperature measurement by the implemented temperature meter of ± 9 °C in the range from 22 °C to 500 °C and ± 13 °C in the range from 500 °C to 700 °C.

Acknowledgement

We are grateful to the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic for financial support of project VEGA 1/0789/21.

References

- M. Kolmačka et al., "Wireless temperature measurement in the hot filament CVD reactor for deposition of carbon nanotubes," *Journal of Electrical Engineering*, vol. 60, no. 6, pp. 346–349, 2009. http://iris.elf.stuba.sk/JEEEC/data/pdf/6_109-07.pdf
- [2] M. Kadlečíková et al., "Redesign of the supply electronics of the temperature gauge in a HF CVD reactor," *Electroscope*, no. 1, 2016. http://hdl.handle.net/11025/17670
- [3] M. Kadlečíková, M. Kolmačka, J. Breza, Ľ. Vančo, "Elimination of the leakage inductance of the primary winding of an impulse transformer for a contact-less temperature gauge," Electroscope, no. 2, 2019. https://dspace5.zcu.cz/bitstream/11025/36523/1/Kadle%c4%8 d%c3%adkov%c3%a1.pdf
- [4] M. Kolmačka, Optimization of nanocomposite synthesis technology based on carbon nanotubes in a HF CVD reactor, PhD Thesis, in Slovak, FEI STU Bratislava 2021, 129 s.
- [5] M. Kadlečíková, M. Kolmačka, "Udržiavanie symetrie výstupných napätí lineárneho regulátora jednosmerného napätia," (Ensuring the symmetry of the output voltages of a linear regulator of DC voltage), Patent SK289142 B6, Úrad priemyselného vlastníctva Banská Bystrica, Vestník ÚPV SR č.: 24/2023, 21. 12. 2023. https://wbr.indprop.gov.sk/WebRegistre/Patent/Detail/64-2021?csrt=6263135209920922156#

Received 1 February 2024