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### AUTOMATED COMPLEX FOR THERMORESISTIVE PROPERTIES INVESTIGATION OF NANOSTRUCTURED FILM SYSTEMS

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Using industrial controllers of Advantech ADAM-4000 Company and system of graphic programming LabVIEW 8.6 the automated complex for thermoresistive properties investigation of film systems is developed. Approbation of the complex on example of Ni/Fe film system obtained by layered condensation is carried out.

Keywords: THIN FILM, LabVIEW 8.6, THERMAL COEFFICIENT OF RESISTANCE.

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#### **1. INTRODUCTION**

Present-day investigations require high accuracy of experiments and data reproduction. As a result recently the automated systems of scientific experiment became widespread, and attempts of new automated complexes formation are often made. Such systems allow to increase an accuracy of obtained results and a "resolution ratio" of experiments (see, for example, [1, 2]). This, in turn, allows to discover new phenomena, which earlier could remain unnoticed. Basic principles of such systems formation can be found, for example, in [3, 4].

Investigations of film materials are of great interest in modern science. First of all, this is conditioned by application perspectiveness of these materials. Now film systems are widespread in such field as production of sensitive elements and sensors. In [5], for example, thin film investigations of nanocrystalline  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> are described. Revealed, that its electrical resistance varies under changes of relative air humidity and temperature. Based on investigations it was concluded about application of such film system for humidity sensors creation in operating temperature range up to 373 K. In [6] it was proposed to use silica film system as a sensitive element of humidity sensors. Conception for the film sensors creation by example of thermo- and tensoresistors based on  $Pd_{0.87}Cr_{0.13}$  is presented in [7]. In [8] the formation technique of sensor arrays based on Pt/polyimide and NiCr/polyimide film systems for temperature and deformation measurements is described; and data of temperature measurements in the range up to 500 K and relative deformation up to 2% by developed experimental sensory couples is presented. In [9] the investigations of thermoresistive properties of Pt thin films on MgO $SiO_2SiO_2$ , and  $Al_2O_3$  substrates are stated, based on which it was concluded about application perspectiveness of such systems for high-temperature sensor creation.

Analysis of scientific literature confirms applicability, perspectiveness and incompleteness of investigations of thermoresistive properties of film systems. Therefore for further implementation of such investigations we have created and tested the automated complex for the thermoresistive properties investigation of film systems.

Programming language LabVIEW 8.6 by National Instruments Company became the necessary software. It proved to be a suitable and flexible tool for creation the automated systems of scientific experiment (see, for example, [3, 10, 11, 12]). LabVIEW is a graphical programming language since it has a user-friendly interface. Arrays, data clusters, cycles, queues, and structures are reflected as pictograms on a block-diagram. Interaction between these elements is realized by the drawing wires for data transfer.

### 2. HARDWARE COMPONENT OF AUTOMATED COMPLEX

Developed automated complex consists of 8-channel 16-bit sigma-delta ADC ADAM-4118, interface converter ADAM-4520, oven heating control circuit with stepwise motor, and personal computer. General diagram of developed complex is presented in Fig. 1.



Fig. 1 – Diagram of the hardware component of automated complex

All measurements are performed in vacuum created by the vacuum plant VUP-5M. Direct electrical current pre-rectified by stabilizer is passed through a sample. Voltage drop on a sample, temperature, and voltage drop on a standard resistance data arrives from working space to the ADC as analog signals. ADC ADAM-4118 converts analog information to a digital one and transmits it via the interface RS-485 to the interface converter ADAM-4520. Converter sends data to the COM-port of a personal computer via the interface RS-485. High reliability, accuracy, software control of the measurement limits, and relatively ease-of-integration into systems of information collection and processing are combined in the controller ADAM-4118.

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In developed complex three ADC inputs are enabled: to one of them a thermocouple of K type (chromel-alumel) is connected for measurements of a sample temperature; two other inputs measure sample voltage directly in the vacuum chamber and on standard resistance plugged to the ADC input.

For measurements the four-point scheme is used. Using two pins an electrical current is delivered to a sample, and using other two pins a sample voltage drop is measured. This allows to measure a sample voltage drop only, therefore wire length (i.e., their resistance) does not influence on calculated sample resistance.

In addition to stabilizer, the use of standard allows to increase the accuracy of resistance measurements. Any random, even minor changes of a current strength do not influence on calculations of a sample resistance, since it is calculated with the Ohm's law.

Data from controller is transferred to the control program, where it is processed and stored. Oven heating control is performed using the monitor signals sent by control program as a binary code to the computer LPT-port. Signals arrive to the control circuit of the stepwise motor, which, in turn, is connected with current regulator in oven heating control circuit. This leads to oven heating decrease or increase.

Since to obtain the reliable results the gradual uniform temperature change is necessary, the PID-control (proportional, integral, and derivative) algorithm was used for regulation and steady speed maintenance of oven heating. Controlled parameter is considered as a process variable (in this case it is the temperature rate-of-change). User inputs tasks, i.e., a necessary value of process variable. PID-control defines a value of control action (in our system it is a rotation speed of the stepwise motor). This value influences on process variable and leads it to the fixed value [13].

#### **3. SOFTWARE COMPONENT OF AUTOMATED COMPLEX**

The control program of developed system should simultaneously perform some tasks, namely, to carry out the collection and processing of experimental data, to display data in graphical and character forms on a user form for analysis, to control an oven heating.

User form of developed application is shown in Fig. 2. Button group "START OPERATION" is responsible for start or completion of experiment. Using button group "OPTIONS" the input of controller and experiment options is performed. Button group "RESULTS" is responsible for the data saving to file.

Indicators of current values 2 display the latest data about voltage drop on a sample and a standard, and the temperature fixed by the controller. Graphic indicators 3 display dependences of measured and calculated during the experiment values.

The basis of developed software is the State Machine based on queue (see [14] in detail). The application operates with four parallel data flows: the interface flow, which is responsible for user events; the flow of data collection from the controller; the flow of obtained data processing and display; and the flow, responsible for an oven control.

Sequential commands execution is realized by the queue using. Clusters, which consist of Enum Type Definition and Variant data type (4, Fig. 3), are the elements of the queue. The first element is responsible for selection of the necessary operation (2, Fig. 3), and the second one performs the data

exchange between parallel operating systems (3, Fig. 3). Cluster elements are sequentially removed from the queue by the function "Dequeue element" (1, Fig. 3) and are divided as a result of each program iteration. Then the necessary code page of the structure Case is selected and executed. Elements are added to the queue using the function "Queue element" (5, Fig. 3).



**Fig.** 2 – User form of the control program: 1 – buttons of the program control; 2 – indicators of the current values of measured parameters; 3 – graphical indicators of the dependences dT/dt(t), T(t), and R(T)



**Fig. 3** – Block-diagram State Machine on the queue basis: 1 – function "Dequeue element"; 2 – selector of the Case structure; 3 – data of the Variant type; 4 – array of the queue cluster elements; 5 – function "Queue element"

All collected data and inputted settings are recorded and stored in the main cluster. Results of the experiment and the dependence R(T) are stored in files of .xls and .lvm format, that allows to carry out their further processing and analysis.

Data exchange between a computer and the controller ADAM can be performed by using both a usual text transmission format of the commands "string" and the protocol Modbus RTU.

Protocol Modbus is the protocol of application level. During interaction controllers use the client-server model based on transactions, which consist of queries and answers. In automated complex personal computer can be the master device (server), and the ADC can be the slave one (client). In answer to client commands server performs the appropriate actions or transmits required data. Block-diagram of the virtual device, which is responsible for interaction with the controller via the protocol Modbus, is shown in Fig. 4.



Fig. 4 - Block-diagram of the virtual device based on the Modbus protocol

Block-diagram of the virtual device used in developed application for interaction with the controller is presented in Fig. 5. It uses the text transmission format of ASCII commands. A command consists of command identifier, controller virtual address in the web, and symbol of carriage return. The last symbol is equivalent to the button "Enter" pressing on a keyboard. New settings of the controller, numbers of the necessary channels, bands and type of measured data, etc can be pointed in commands as well.

# 4. APPROBATION OF AUTOMATED COMPLEX

Using developed automated complex we carried out investigations of the thermoresistive properties of Ni(15)/Fe(35)/S (S is the substrate) film, obtained by layered condensation. Based on the obtained dependence of resistance versus temperature R(T) (Fig. 6a) the temperature dependence of the thermal resistance coefficient (TRC) was calculated (Fig. 6b). During the experiment three cycles of annealing and cooling were performed. TRC decreasing cycle by cycle can be explained by defects reduction and recrystallization processes.



Fig. 5 - Block-diagram of the virtual device with the text commands

Inset in Fig. 6a shows a quantity of points obtained during the whole experiment (~ 5500) that is impossible for manual mode. PID-control using for a feedback during the oven governing allowed to improve the experiment.



**Fig. 6** – Experimentally obtained dependences: R(T) (a);  $\beta(T)$  (b). 1-3 are the numbers of cooling cycles. On the inset: dependence R(T) in the temperature range from 495 to 535 K

Taking into account obtained results we can conclude that the LabVIEW language allows rapidly and effectively create software for automated complexes of scientific experiment. Use of industrial controllers of the ADAM-4000 type by Advantech Company, which combine high accuracy, reliability, and low costs, leads to more effective approach in development of automation systems of scientific experiment.

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