The equipment for controlling the structure and functional properties of nanostructured composite films

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The equipment for controlling the structure and functional properties of nanostructured composite films

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Abstract. The article is devoted to the creation of an instrumental system allowing evaluating the functional properties and current-voltage characteristics of nanostructured composite films at different temperatures and other environmental parameters. The system is based on an assessment of current-voltage characteristics of a nanostructured film material. The main components of the system are a chamber and a unit for current-voltage characteristics measuring. The stage with the test material and the contact system are provided with a heating element and a cooling system thus allowing warming to 150 °C and fast cooling to negative temperatures by liquid nitrogen circulating. The chamber body leak proofness against the external environment allows forming a composition of the atmosphere at a predetermined humidity level, which is essential for the measurement of current-voltage characteristics of polymer materials. The article describes the design features of the instrumental system and results of its application used for determining the properties of polymer nanostructured composite films.

1. Introduction
Laboratory evaluation of the structural state of the thin film made of composite materials provides sample preparation for X-ray diffraction and electron microscopic studies. In contrast to the research laboratories conditions preparation of samples at factories and plants the evaluation of the material’s structural state is incorporated into the technological process chain only in the form of sampling. Counting the fact that the formation of nanostructured thin film materials is the so-called ‘thin technologies’, there is the need to have equipment that enables controlling the process without preparation of special samples. For example, to evaluate the structural state of the surface layer of engineering products, the measurement of hardness or the damping parameters of ultrasonic vibrations, which are sensitive to the structural state of the material, are widely used. Under the conditions of technology compliance with these characteristics it is possible to reliably assess the compliance of the material structure with initial parameters and to judge about accordance of its surface layer properties with specified ones in the manufacturing documentation. Benefits of using structure-sensitive characteristics are the possibility of taking measurements at various stages of the manufacturing process of each product without disrupting its integrity and availability.

Hardness testers and ultrasonic devices are of little use for assessing the structural state of the nanostructured thin film materials. In this case, measurement of electrical resistance which is a structure-sensitive characteristic would be the most convenient way to assess the state of the material [1-6]. The use of the current-voltage dependence allows one to evaluate the structural condition of the...
nanostructured thin film material, if one knows the relationship between them. Since the electrical conductivity and current-voltage characteristics of all types of nanostructured films are affected by the temperature at which measurements are taken, the equipment for the control of current-voltage dependencies must ensure its variation and stabilization during measurement. For films with polymeric components, a chemical composition and environment humidity are essential, and for the light-sensitive semiconductor films the measurements at certain level of light should be provided. Thus, we have solved the task of equipment creation which allows: simultaneous measurement of conductivity (current-voltage characteristics) and X-ray analysis for film structure assessment; measurement of conductivity (current-voltage characteristics) under different external conditions. Equipment was originally designed for evaluating the photovoltaic characteristics of the solar cell semiconductor polymer materials, as well as for studies of the structure and properties of the active material from which these elements are made. The created equipment is applicable to assess the structural state of nanostructured thin films of various compositions.

As thermal treatment is one of the ways to improve the reliability of welded joints, the aim of this work was to study the structural transformations occurring during heat treatment of the welds between the pearlite and high-chromium-nickel austenitic steels, obtained by flash butt welding.

2. Description of the developed equipment

The main devices for monitoring the current-voltage characteristics of nanostructured thin film materials are an in-situ chamber and the software-hardware system. The in-situ chamber (Figure 1) is a leak-proof prismatic box (position 1), bounded with X-ray transparent plastic film windows from four sides (position 2). All windows are removable and are attached to the chamber through sealing. The front window is connected to the stage (position 3), which is supplied with a ceramic heating element and the thermocouple.

![Figure 1](image-url)  

**Figure 1.** An in-situ chamber for control of structure and current-voltage characteristics of thin composite film materials.

The heating element is located under the top plateau (position 4) and thermocouple (position 5) is fixed in the center of the stage by a threaded connection. The stage design provides its cooling by liquid nitrogen pumping through the channel located in the lower part under the heater. Pumping of nitrogen is carried out through unions with collet fastening (position 6), which are connected by heat-
resistant polymeric tubes with a liquid nitrogen tank on the one hand and with the pump – on another one. All materials used in the chamber should work trouble-free in the temperature range from –40 to +150 °C.

The substrate up to 20 × 20 mm in size with a controlled film is placed on an insulator (position 7), which is on the top plateau (position 8) of the stage. Limiting the size of the controlled film and, accordingly, the size of the chamber is caused by its use in the step of finding the relationship between conductivity or current-voltage characteristics of the films with their structure. Such studies are carried out using an X-ray diffractometer with limited space to place the chamber. The insulator is made of PTFA and has a cave with conductive coating of gold. Four electrical contacts in the form of leaf springs are brought to the surface of the controlled film or to the conductive coating through the isolator (position 9). The chamber is installed in the X-ray diffractometer with high intensity radiation with positioning at the base (position 10). The leak proofness of the chamber allows making necessary environmental conditions by pumping gas composition through four fittings (position 11) mounted on the sides of the chamber. In the study and control of film materials, which functional properties related to photovoltaics, an installation of a sunlight simulator is provided in the chamber which ensures adjustable intensity of the film surface radiation up to 1500 W/m². The printed-circuit board (PCB) of the sunlight simulator was developed by the innovative enterprise ‘Ekoenergotekh’ at Novosibirsk State Technical University and executed on an aluminum substrate with an oxide insulating layer. The PCB incorporates RGB + UV + IR light-emitting diodes providing a range with a wave band from 0.3 to 1.3 μm which is close to sunlight in intensity of the spectral structure. The need to use a simulator of original development was caused by the requirement to ensure sufficient chamber leak proofness. Power supply of the simulator is carried out through the leak proof cable input of PG7 (position 12) from an adjustable source of direct current for the purpose of illumination pulsation exception. The PCB simulator is protected from the chamber environment by PET glass which passes all range of sunlight.

The designed and manufactured chamber is used together with a hardware-software system which consists of the block of current-voltage characteristics measurement and the microprocessor control block; the former is guided by the latter. The hardware-software complex functions firstly in the mode of voltage generation in the range from 0 to 10 V with an accuracy of ±1 mV and current measurements in the range of 1 pA to 1 mA with an accuracy of ±1 pA in the range of 1-1000 nA measurement and ±10 % within measuring of 0.001…1 mA and, secondly, in the mode of measurement of current-voltage characteristics of photo cells at various levels of electric load.

3. A sequence of equipment use
The current-voltage characteristic of the material and its structure is in complex functional relationship, which has to be established at the stage of film formation technology development.

This step is a research and it requires the use of analytical tools and, above all, the X-ray analysis while studying the nature of changes in conductivity or current-voltage characteristics under various conditions. The second stage is designed for the production conditions and consists in monitoring of the film material state at various steps of the process. It will be enough to measure the current-voltage dependence to assess the structural state of the nanostructured film material at this stage.

3.1. The mode of the studying of the relationship of current-voltage characteristics and the film structure
An example of using the developed equipment was the research of technological modes for creation of nanostructured films on the basis of polymeric mixtures P3HT/PCBM which are applied for organic solar elements creation.

Figure 2 shows the current-voltage characteristics of nanostructured films with simultaneous recording of X-ray, allowing judging the structural changes taking place in the film material. GIWAXS study of the film revealed that there is a two-stage increase in the current strength
corresponding to the two-stage dependence of the crystallinity degree. The determination of these characteristics for films P3HT: ICBA showed that the intensity of peak (100) and the current increase linearly with the film deposition before achieving the maximum in the solid state. The highest conductivity values are observed in the solid state. This is due to better compatibility of P3HT and ICBA, whereby ICBA generates smaller domains which inhibit crystallization of the polymer during the solvent evaporation to a lesser degree. The structure of polymer films of a different chemical composition in the initial state (after casting) has a low degree of crystallinity, and its value does not exceed few percent, and the sizes of the crystalline domains do not exceed an average of several tens of nanometers. So, the studies have revealed the optimal mode of preparation of the active material for solar cells to increase their efficiency.

![Graph a) and b)](image)

**Figure 2.** The time dependence of the current (a blue curve) and peak intensity (100) (a red line) in the films of P3HT: PCBM (a) and P3HT: ICBA (b) during crystallization.

3.2. The mode of assessment of the structural state of the film material by the current-voltage characteristic

During production the designed equipment may be used for controlling the properties of the final product – the nanostructured composite film, and in the mode of measuring of the current-voltage characteristics of the film material, made at various stages of the process. Deviation of current-voltage dependence obtained at this step from the one obtained at the step of researching the relationship between structure and current-voltage characteristics indicates deviation from the required structure. The level of current values may thus differ insignificantly in case of controlling the films, sensitive to X-rays, for example, in assessing the current-voltage characteristics of the semiconductor films.
4. Conclusions
The developed equipment is designed to assess the structural state of nanostructured thin films of conductive and semiconductor materials with minimization of the sample preparation operations. The use of the equipment in production provides the ability to embed the process of nondestructive testing of film material into it.

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