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Method for Recording and Reproducing an Experimental Research into the Field Emitter Properties Based on Carbon Nanotubes

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Abstract

A method for recording and emulating a complex experiment in field emission was developed. The method includes processing of three types of data streams: data on the electrical characteristics of the field cathode (voltage and current pulses), data on the field emission projector (glow patterns) and time-of-flight mass spectrometer data (mass spectra of volatile products in the measuring chamber). The LabView software environment implements an algorithm for synchronous reproduction of multichannel experiment data with the possibility of processing it in real time. The program has a built-in set of software tools that implement the functionality and repeat the experiment many times, pause at specified points in time, as well as change the time flow rate in the emulation. The capabilities of the method are demonstrated by the example of studying field emission from a nanocomposite field cathode based on carbon nanotubes.

Keywords

Carbon nanotubes; field emission; multi-channel data collection; online processing; experiment emulation.

Introduction

Methods for obtaining, storing and processing experimental data are one of the integral parts of experimental physics. These methods are continuously developing along with the development of computer and measuring equipment. Means of high-speed registration and data recording today allow receiving huge amounts of information. So, for example, high-speed recording of indicators of radioactive particles sensors is used to study the processes occurring in plasma of thermonuclear reactors (**ITER**) [1].

On the other hand, modern computing systems allow for online data processing, decreasing the amount of recorded information by orders of magnitude. Online processing also allows controlling the behavior of the experimental system in time and when changing experimental conditions, for example, registering the amplitude of the field emitter (current pulses) response with a sharp increase in the amplitude of the voltage pulses [2].

The field emission experiment described in this work is a special class of experiments, the implementation of which requires creating vacuum conditions with a residual pressure of no higher than 10^{-5} Torr and supplying a high voltage *field emitter* (conducting structure with a high aspect ratio) to the sample under study, i.e. creation of an electric field of high intensity on the surface of the emitter (cathode). High tension and vacuum are necessary for the occurrence of field emission on the cathode surface. In this phenomenon, electrons leave the cathode surface and exit into vacuum passing under a potential barrier due to the quantum tunneling effect [3].

Modern field emitters have a complex surface that has many separate emission centers (for example, carbon nanotube forest) [4]. The study of field emitters is associated with the study of the influence of many factors on their work: the mutual influence of emission centers, heating of the longest centers up to their thermal explosion, adsorption processes on the surface of the emitter, etc. [5]. Due to this multifactorial nature, modern research complexes are increasingly moving from classical recording of current-voltage characteristics [6], to multi-channel recording of related processes: the level of vacuum in the measuring chamber and its composition, cathode and anode temperature, glow patterns arising on a special phosphor anode, as well as infrared radiation of emission-heated emission centers [7]. Methods for scanning the surface of a sample [8] and obtaining the energy spectrum of electrons [9] are also widely used.

In the framework of technological optimization, registration of the current-voltage characteristics and their corresponding glow patterns is most often used [10]. Another priority is the use of mass spectrometric analysis of the residual atmosphere composition in the chamber [11].

In the present work, we will focus specifically on these three streams of experimental information (current-voltage characteristics, glow patterns, and mass spectra). In general terms the analysis algorithms presented in the study can be applied not only to the experiments of field emission, but also to other types of physical experiments with high density of information.

The recording speed and sampling rate of data from a field emission experiment for different information channels differ by orders of magnitude. Thus, the current-voltage characteristics can be obtained both in the slow mode (one current-voltage characteristic for a time of the order of a minute [12, 13]) and in the fast mode (one current-voltage characteristic in a fraction of a second [2]). In this case, the number of measured voltage and emission current values in the current-voltage characteristic itself depends on the speed of the signal-recording equipment. The fast mode (alternating voltage) is less destructive for the field emitter, i.e. it allows studying the emission properties at higher currents [14].

The frequency of registration of glow patterns depends on the data transmission channel, as well as on the resolution of the recorded picture, for example, a Nikon Coolpix P950 CCD camera with a resolution of ~20 μm was used in the dissertation [15]. The speed of continuous photographing is 7 frames per second. In [2], an in situ analyzer of emission characteristics and a high-speed camera for recording glow patterns were used. The camera recorded pictures at a frequency of 60 frames per second, and the current-voltage characteristic was recorded and processed every 50 ms. About 10,000 current-voltage characteristics were processed and the SK diagram of fluctuations in the emission properties of the tungsten cathode was constructed.

Mass spectrometric analysis is perhaps the most capacious in terms of data density. For example, in [16], an SRS RGA100 residual atmosphere analyzer was used with a measurement range from 0 to 100 amu and a spectrum resolution of ~10 points/amu. The temporal resolution of the spectrum was 20 ms/amu, so that the full mass spectrum was recorded in a few milliseconds, however, its transmission to a computer via an Ethernet channel can be delayed up to 1 spectrum per second. The amount of data transmitted to the computer can be reduced by using hardware gating. This method was used in the study of field emitters in [17].

Field emission scientists have increasingly used the LabVIEW platform in recent years. This software shell is used to simultaneously register data from several channels for online processing of experimental data and controlling the experiment itself.

In [16], a special LabVIEW program was used to simultaneously record the emission current and voltage levels (using a Keithley 6485 picoammeter), mass spectra (SRS RGA100) and the vacuum level in the chamber (UHV ion gauge), together with the control of the high-voltage unit power supply (Spellman 1200).

The LabVIEW is also used to control a video camera that registers glow patterns, for example, in case of a stepwise change in the voltage level [18].

The LabVIEW control of high-voltage power supplies of the Keithley type in the study of field emission is widespread [11, 13, 15, 16, 18, 19].

Working with emission data online is quite rare. As a rule, only the dependence of the recorded values (current and voltage) on time is carried out [19]. In [20], it was shown that processing current-voltage characteristics online has several advantages over postprocessing. Online processing of the current-voltage characteristics can also be used to test current experimental data for compliance with the cold field emission mode, i.e. using the well-known Fowler–Nordheim equation [21]. The authors of this article have developed a methodology for synchronous online processing of glow patterns and current-voltage characteristics which allows obtaining detailed information about the operation of individual emission centers [22–26].

Currently, scientists are actively studying and optimizing multi-tip field emitters (arrays of microscopic tips, nanocrystals, nanoparticles, etc. [5]). An experiment studying the properties of these emitters encounters a number of difficulties, some of which could solve multiple and exact repetition of an experiment.

First, there is a need for a detailed study of the field emitter behavior which is complicated by an unpredictable or irreversible change in its structure (for example, a change in the emitter surface under the influence of high voltages when registering its current-voltage characteristic [27]).

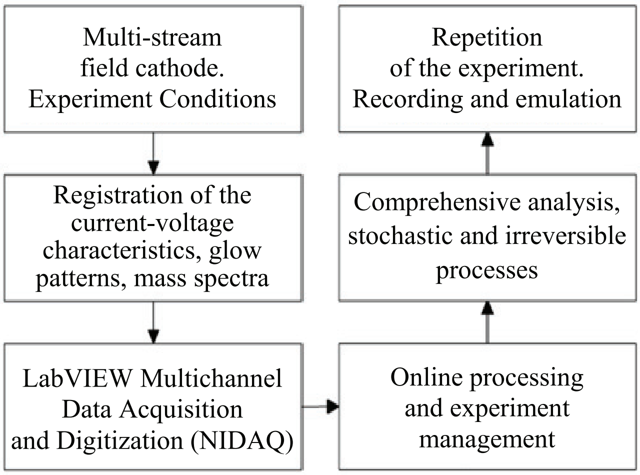


Fig. 1. Diagram of computerized recording and analysis of field emission data using a high voltage vacuum unit

Secondly, it is the need to implement a complex data analysis algorithm, for example, searching for the position of emission centers, and then observing their behavior. The continuous appearance of new emission centers on the cathode surface complicates this procedure [28].

Thirdly, the need to analyze fast and unpredictable processes during field emission, such as current surges in the system caused, for example, by short-term vacuum discharges [29].

Fourthly, the use of online data analysis sometimes significantly slows down the operation of the control program, which leads to a decrease in the frequency of data recording, i.e. to the loss of some information about the field experiment.

Fifthly, when developing and testing in practice new programs for online data analysis, it becomes necessary to repeat the experiment repeatedly.

Similar problems arise when studying classical single-axis emitters. Thus, despite significant technological progress in the methods of recording various physical quantities, the experiment with field emitters continues to experience difficulties in reproducibility and processing of a large flow of experimental data (Fig. 1). One solution might be to emulate an experiment.

The objective of this work was developing and testing the method for recording and emulating a complex experiment in field emission.

Methods and materials

Installation of multi-channel data collection with online analysis

A unique method for studying multi-edge field cathodes was developed in the laboratory at the Ioffe Physical-Technical Institute [30]. It is based on a vacuum unit for multichannel collection and computer processing of experimental data on the field emission process and its accompanying processes. The setup allows to register the current-voltage characteristics of the test sample, its glow patterns, and also to obtain mass spectra of the residual atmosphere composition in the measuring chamber.

The measuring chamber is equipped with a high-voltage input in a standard diode configuration with flat electrodes, on one of which the studied field cathode is fixed. The turbomolecular pump maintains a pressure of $\sim 10^{-7}$ Torr in the working volume. High voltage bushings and a system of voltage dividers make it possible to register the voltage applied to the sample and the corresponding emission current. The voltage is applied in the "fast" scanning mode: half-sine pulses with a frequency of 50 Hz and an amplitude of up to 10 kV (the source is an AII-70 X-ray transformer with a load controlled by a manual LATR). It is also possible to use a slow mode (constant high voltage source of arbitrary shape specified from a computer).

Fig. 2 shows a simplified scheme of multichannel data collection, including registration of the current-voltage characteristics, mass spectra, and glow patterns.

Registration of electrical signals is carried out by a collection board NI DAQ 6351 built into the computer and equipped with an external system of protection against voltage surges in the measuring circuit. The glow patterns are recorded using a long-focus USB microscope eScope DP-M12. The mass spectra are recorded using a time-of-flight reflective mass spectrometer [31] which transmits the signal to a computer via a four-channel Tektronix DPO2024B high-speed oscilloscope equipped with an Ethernet unit.

Information from the listed recording channels is sent to the computer in the form of separate data packets: current-voltage characteristics – 1000 voltage and current values recorded at a frequency of 50 kHz, glow patterns – color camera frames with a resolution of 1280 × 960, mass spectra – 100,000 ion flux density values recorded at a frequency of 1 GHz. The frequency of recording and downloading this data to a computer is different. The current-voltage characteristics arrive at a speed of one packet in 20–40 ms, glow patterns – one frame in ~ 0.1 s, mass spectra – one spectrum in 1 s.

To process experimental data packets, a special program written in the LabView 2016 is used. It consists of three independent modules that communicate through a common global variable.

The first is a module for recording and processing the current-voltage characteristics (code name “Zeus”). The functions of this module include calculation and real-time monitoring: 1) the shape of the voltage

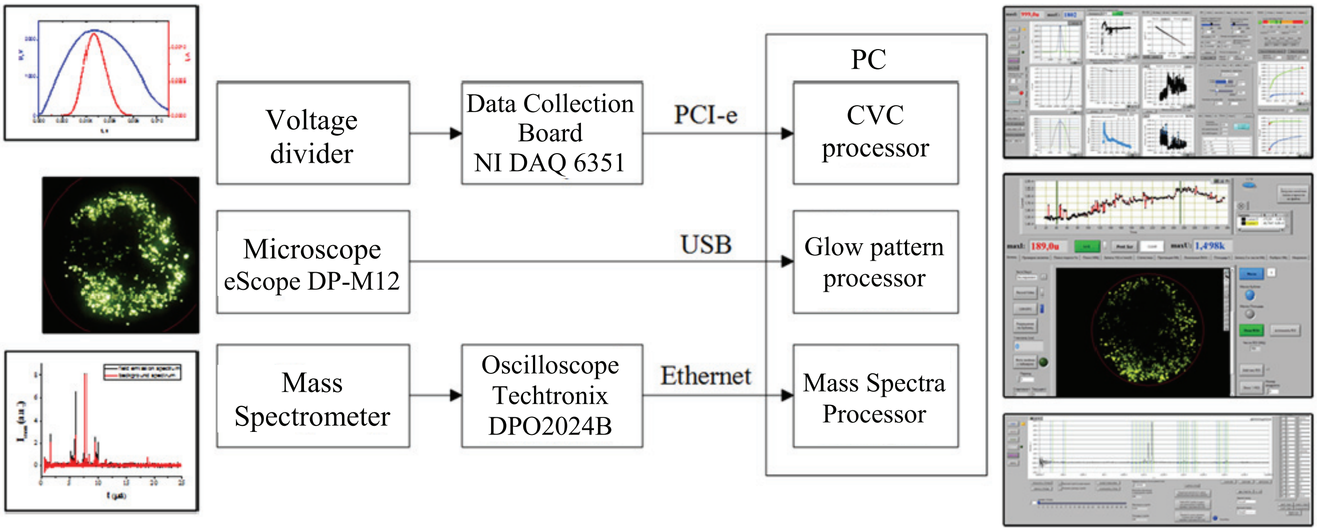


Fig. 2. Scheme of multichannel registration and online processing of field emission data while studying a cathode based on multi-walled carbon nanotubes

pulses $U(t)$ and current $I(t)$; 2) current-voltage characteristics in standard coordinates $I(U)$, in Fowler-Nordheim coordinates (CVC–FN) – $X = 1/U$, $Y = \ln(I/U^2)$ and in modified Fowler–Nordheim coordinates (CVC–FNη), more true from the point of theory, $X = 1/U$, $Y = \ln(I/U^{2-\eta})$ [32]; 3) a diagram of the change in the current-voltage characteristics in the coordinates of the cutoff – slope, where the cutoff and the slope are along the trend line plotted for the CVC–FN (the so called SK diagram [2]); 4) the time dependence of the vacuum level $P(t)$, as well as the amplitudes of the voltage $U_m(t)$ and current $I_m(t)$; the time dependence of the effective parameters of the emitter obtained as a result of online calculations according to the SK diagram – the electric field gain $\beta(t)$ and the emission area $S(t)$; 5) fluctuation histograms of β and S at a stable current level.

The second is a module for recording and processing glow patterns (code name "Argus"). The functions of this module include: 1) determining the position of emission centers by the brightness peaks of the luminescence pattern and accumulating the found centers with time, which is accompanied by the construction of a "collection curve" – the distribution of the centers by the time they appear "in the frame" [23]; 2) calculating the local current load of each of the found emission centers as a function of time $I_{local}(t)$ (this tool is interesting for analyzing the noise characteristics of the cathode [24]); 3) plotting a histogram of the distribution of centers by current load $N(I_{local})$ (average and maximum), as well as a histogram of the distribution of centers by the effective field gain $N(\beta_0)$ found by numerically solving the Fowler–Nordheim equation for each emission center, taking into account the experimenter emission area of one center S_0 [25]; 4) constructing local current-voltage characteristics for each emission center when scanning a sample with high voltage $I_{local}(U)$, as well as the regression analysis of the obtained dependences with the calculation of the local parameters of each emission center – diagram $S_0(\beta_0)$ [26]; 5) assessing the uniformity of the distribution of emission centers on the cathode surface along the radius, angle, brightness, current load on the segment [23].

The third is a module for recording and processing mass spectra (code name "Ether"). Its functions include: 1) building a mass spectrum – the dependence of the ion flux density on time / mass; 2) calculating the positions of the peaks of the given masses from the two peaks indicated on the spectrum with known masses; 3) constructing the time dependence of the partial pressures of the masses $P_i(t)$ chosen by the experimenter; 4) subtracting the previously saved background spectrum online from the current spectrum.

All three modules allow saving the obtained dependences and the results of online calculations in text files, as well as making multiple "photographing" of the current indicators, so that either a dot (marked value, for example, current amplitude) or an additional graph (for example, marked as CVC).

In addition, the modules are equipped with a tool for recording the corresponding signal into a text file (SIGNAL IVC.txt, SIGNAL GLOW.txt, SIGNAL MASS.txt). The recording is accompanied by the formation of a sequence of t_{signal} numbers – the registration times of each signal implementation (data packet), which is also recorded in a separate text file (times – IVC.txt, times – GLOW.txt, times – MASS.txt). These six files can accommodate a many-hour experiment on the study of field emission and form the basis for subsequent emulation of the experiment.

Experiment Emulation Program

Let us formulate the requirements for the experiment emulation program that were put forward during its creation:

– parallel reading of data from several files recorded during a real experiment for different data streams;

– reproduction of the relative speed of data streams according to the recorded times of their registration;

– processing of data packets in the corresponding modules of the main processor program written in the LabVIEW (modules “Zeus”, “Argus”, “Ether”);

– the possibility of changing the experiment speed during emulation;

– eliminating omissions (loss) of reproducible data that may occur due to delays in processing packets.

The main idea of the developed program is presented in Fig. 3*a* in the form of a diagram. The scheme is a control cycle for loading and processing data packets of one information channel. It consists of three blocks that control each other through two Boolean variables.

The Timer module is responsible for the duration of the emulation experiment. When the time is right for processing the relevant data, the Timer module transmits a signal “processing = true”. The Download module is responsible for preloading data. After loading the corresponding data packet, it sends a “load = false” signal, which indicates that the data has been successfully downloaded and is ready for processing. The Processing module after receiving two signals, namely “processing = true” and “loading = = false”, starts processing the corresponding data. When processing is completed, the Processing module changes the values of two Boolean variables to the opposite, giving a start signal to the previous two modules. Then the process is repeated.

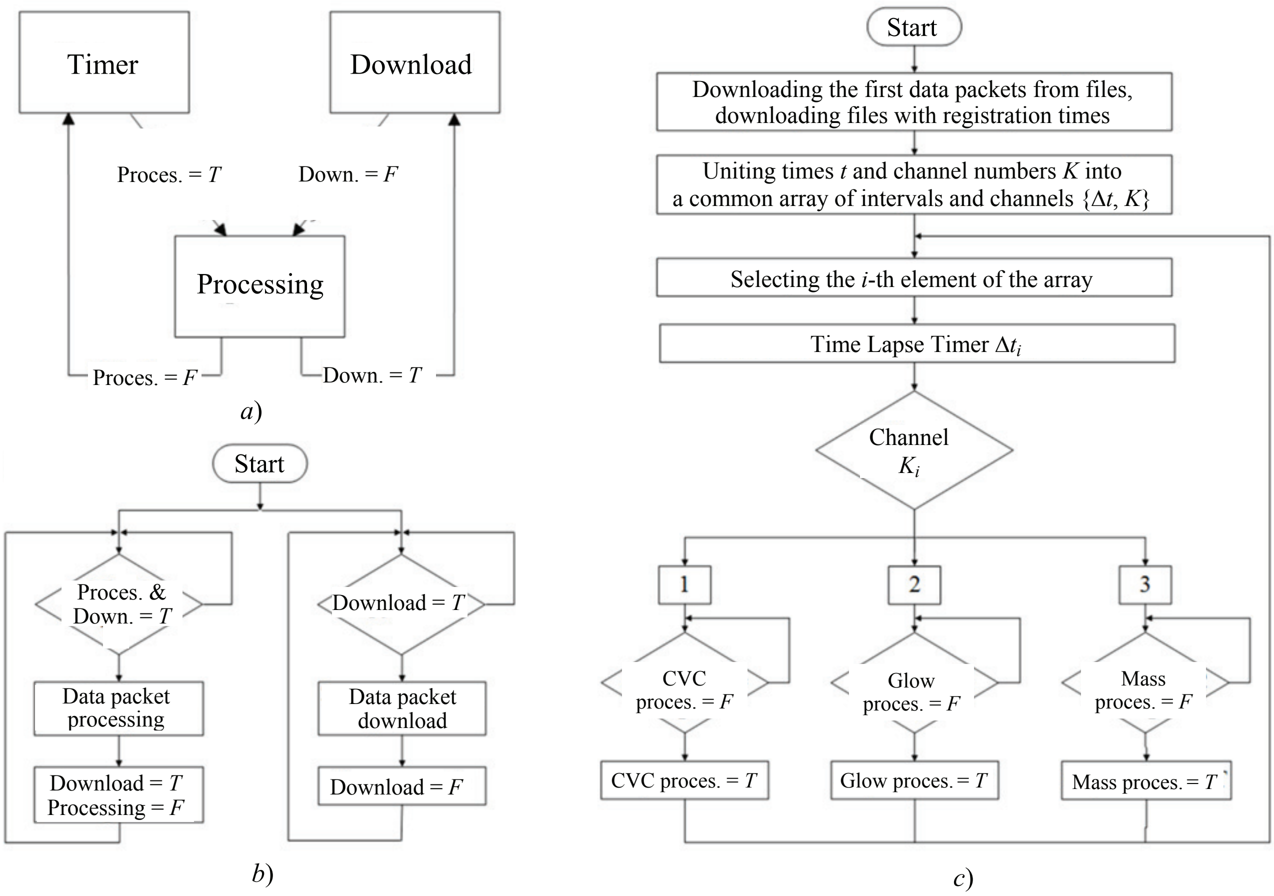


Fig. 3. Algorithm for the emulation program:
a – a simplified diagram of the basic idea of the emulation process experiment;
b – an organization scheme of the “Processing” – “Download” processes for one data stream;
c – an operation scheme of the module managing time intervals

The Download cycle was built to implement the presented scheme in each of the three main modules (“Zeus”, “Argus” and “Ether”) next to the main cycle (Processing). This double cycle “Processing” – “Download” is shown in Fig. 3*b*. By the signal of the global variable “download = true”, the Download module loads the next data packet from the file specified in it into the program’s memory, and then sends the signal “download = false”. The Processor module has a built-in delay element that waits for a signal both from the Download module and from another global variable “processing = true” (external start). After starting, the Processor module processes the last packet loaded into the memory and sends a signal “processing = false”.

The algorithm for organizing the work of three independent modules can be implemented in a direct way: each module reads and processes data packets maintaining known time intervals. However, the probability of one of the modules lagging behind the others (due to the lengthy processing of packets that the experimenter may require), as well as a number of additional functions (for example, general suspension of emulation) require the interaction of the modules and significantly complicates the algorithm. Moreover, the emulation of the described experiment does not require direct synchronization of flows. Synchronization is conditional and consists in reproducing the relative speed of registrating data packets of different channels. In this regard, a separate program (code name “Morpheus”) was written to control and conditionally synchronize three independent modules. Its main function is to organize the mutual delay of the start signals and the processing sequence of heterogeneous data packets.

The block diagram of the Timer algorithm is shown in Fig. 3*c*. At the very beginning of the entire emulation program, Morpheus checks the specified paths to the files with experimental data (SIGNAL-IVC.txt, SIGNAL-GLOW.txt and SIGNAL MASS.txt). Then a signal is sent to the loader modules (“load = = true”) to preload the first data packets. Next, the signal registration times (times-ivc.txt, times-glow.txt and times-mass.txt files) are downloaded and a common sequence of clusters $\{t, K\}$ is created from them. Each of its clusters consists of two elements – time t and an indicator digit K, which shows which of the three data streams this timestamp belongs to ($K = 1, 2, 3$). Then this array is sorted by time (ascending) and the times in the clusters are converted into time intervals: waiting intervals between events – the moments of arrival (registration) of data packets. Thus, the sequence $\{\Delta t, K\}$ is obtained, which contains information on how much time after the previous packet the next one should be submitted, as well as which of the three data streams it refers to. After one waiting interval, the Timer sends a signal to the corresponding global variable ("processing = true" for the desired module), after which it immediately starts counting down the next waiting interval.

The device of the presented algorithm is such that if the processing of a data packet of one of the three streams exceeds the time interval between registration of this packet and the next one, the Timer module creates the necessary delay for all channels. Thus, no data packet will be skipped, although an emulation experiment may be longer than its real prototype.

On the other hand, all time intervals in the sequence $\{\Delta t, K\}$ are multiplied by the coefficient ε specified by the experimenter, which allows both to slow down the experiment ($\varepsilon > 1$) and to accelerate it ($\varepsilon < 1$), up to maximum acceleration ($\varepsilon = 0$). Acceleration is possible due to the fact that emulation does not use the ADC of the reading equipment. The maximum emulation speed is limited by the speed of reading data from files.

The interface of the Morpheus program is shown in Fig. 4.

In addition to solving the initial problems, the developed program includes the following features:

– suspension of the experiment at predetermined points in time with the ability to enable the necessary functions in the data processing modules (for example, “photography”);

– looping with automatic extension of the timeline;

– synchronous launch of independent software modules in the emulation mode.

Model sample

A field cathode based on single-walled carbon nanotubes and polystyrene was used as a model sample for the experiment and subsequent testing of the developed emulation tool. This is a promising form of field cathodes which is quite often found in the scientific literature [33].

Nanotubes were manufactured by OCSiAl (Novosibirsk). Their outer diameter is ~ 2 nm and their length is ~ 10 μm. The emitter was created by applying a suspension of “nanotube – polystyrene – orthoxylene” to a metal substrate using a rotating table method. After the evaporation of orthoxylene on the substrate, a solid nanocomposite film was formed where a non-conductive polymer plays the role of a binder that increases the resistance of nanotubes to the pulling forces of an electric field.

Fig. 4. Interface of the program controlling the emulation process

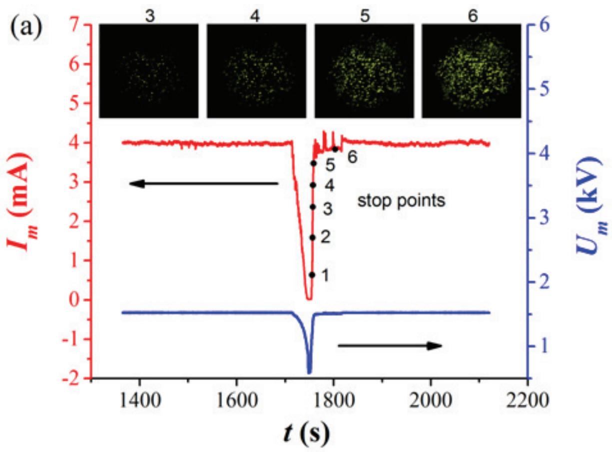
Results: application of the repeated experiment method

The experiment consisted of long-term (~ 10 min) maintaining a constant voltage level at the field cathode with a single rapid decrease in voltage to zero and subsequent rapid (~ 5 s) increase to the previous level. The interelectrode distance was 370 µm, the amplitude of the voltage pulses was ~ 1.5 kV, and the corresponding field emission current was ~ 4 mA.

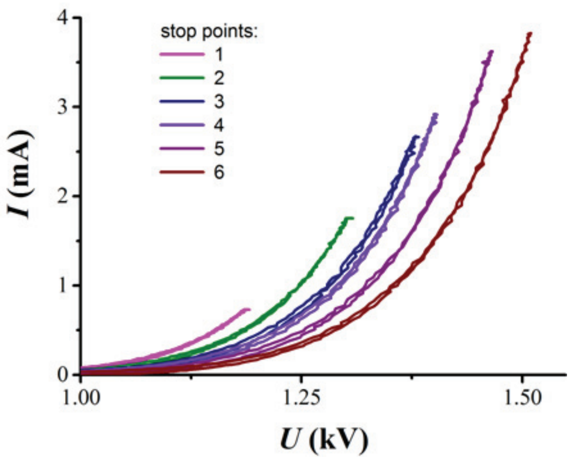
The experiment was recorded using the "Record" blocks in each of the three independent software modules. We show the capabilities of the emulation program using the example of three studies.

The CVC evolution with a rapid change in the voltage level

The first viewing of the experiment in the emulation mode made it possible to construct the dependence $I_m(t)$, at which time moments with different voltage levels were noted. The second scan was accompanied by predetermined stops at these points in time which made it possible to "photograph" the state of the cathode: its current-voltage characteristics and the corresponding glow patterns. The result is presented in Fig. 5. A change in the shape of the current-voltage characteristics with an increase in the amplitude of voltage pulses is associated with a restructuring of the adsorption state of the cathode surface, as it was shown in [25].

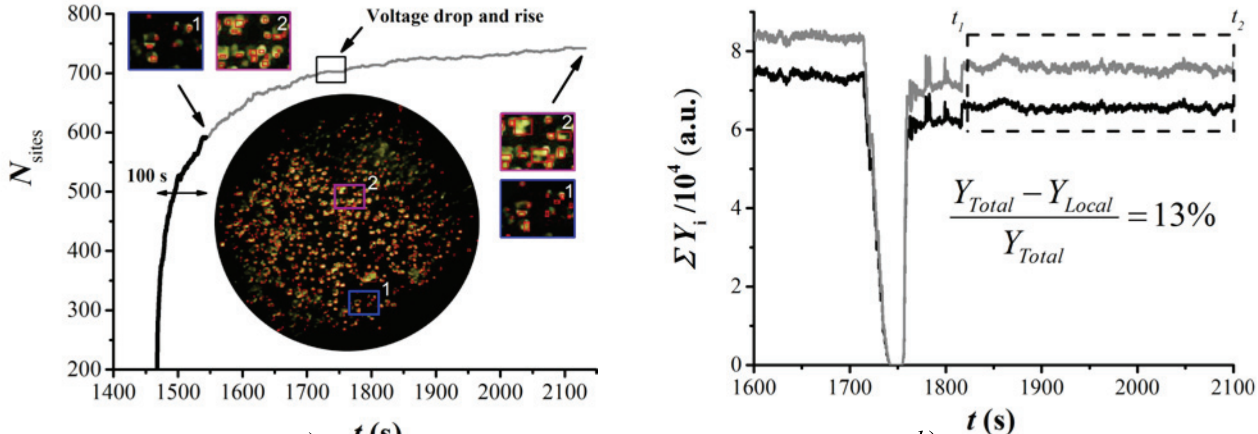


a)



b)

Fig. 5. Study of the evolution of the current-voltage characteristics with a rapid change in the voltage level:
a – Time dependences of the current and voltage levels, the stopping points of the experiment and the corresponding distribution patterns of emission centers marked on them;
b – the family of current-voltage characteristics "photographed" at the indicated time moments



a) *b*)

Fig. 6. Studying the influence of the collection time of emission centers:
a – curves of collection centers for 100 s (black line) and for the duration of the entire experiment (gray line);
b – time dependence of the total brightness of the centers found
(The insets show the glow pattern and its element with the found emission centers (small rectangles))

Studying the impact of the collection time of emission centers

The study consisted in analyzing the behavior of groups of emission centers collected over various time intervals: over 100 s and during the entire experiment, including the decay and voltage rise time shown in Fig. 5*a*. The first viewing of the experiment in emulation mode was used to collect the centers (determining their positions). Fig. 6*a* shows the obtained collection curves: a shortened group (~ 600 centers) and a complete group (~ 740 centers). It should be noted that along with the registration of new centers (fragments No. 1 in the glow patterns of Fig. 6*a*), the previously noted ones (fragments No. 2) merge with time.

The following scan was used to track the brightness of the found groups of centers. Fig. 6*b* shows the time dependences of the total brightness of the centers (a shortened group of 600 centers and a complete group of 740 centers), which repeat in shape the dependence on the total current time (see Fig. 5*a*). In the region with relative signal stability (dashed in Fig. 6*a*), the relative loss of the overall brightness of the picture was determined when using the reduced group instead of the full one:

$$Y_{\text{total}} = \text{mean}_t \left(\sum Y\right)_{\text{total collection}} = 75820; \qquad (1)$$

$$Y_{\text{local}} = \text{mean}_t \left(\sum Y\right)_{\text{local collection}} = 65700; \qquad (2)$$

$$\frac{Y_{\text{total}} - Y_{\text{local}}}{Y_{\text{total}}} = 0.13\,. \qquad (3)$$

The loss of the total brightness of the centers (averaged over time) was 13 %. This is a possible error in determining local currents when using the reduced group. On the other hand, it can be seen from the constructed dependences that the total brightness of the reduced group is just as stable in time as the full group. This suggests the possibility of reducing the time of collection centers.

Studying the behavior of different groups of emission centers

In the framework of this experiment, the threshold brightness Y_{th} of the collection of emission centers, which cuts off the dim centers, was varied. The obtained "spectrum" of collection curves (see Fig. 7) can be used for frequency analysis of various groups

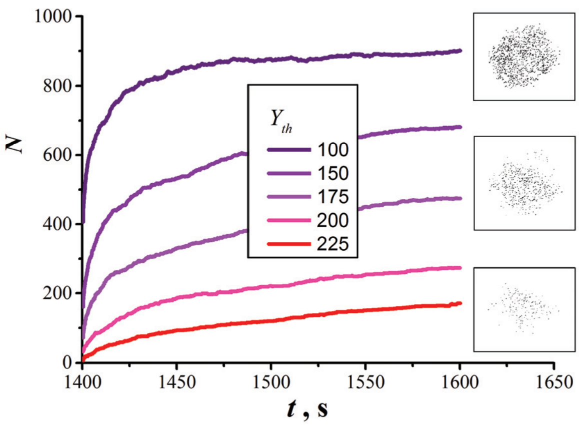


Fig. 7. Collection curves obtained for different threshold brightnesses Y_{th}: 225, 200, 175, 150, 100
(The insets show the collection diagrams of the positions of emission centers for Y_{th} = 225, 175, 100 (inverse image))

of emission centers on the surface of a multi-edge field cathode, since the collection curve is, in essence, the frequency response of the scintillation of emission centers in the glow pattern under the influence of adsorption processes.

The construction of such a spectrum in a real experiment requires writing a special program that would repeatedly process each frame. This will significantly slow down the processing and increase the time interval between the registration of signals. This problem is removed in cyclic emulation mode.

Conclusion

An experimental data analysis method based on software tools that allow recording and reproducing a field emission experiment with multi-channel data recording in the emulation mode (streams: current-voltage characteristics, glow patterns and mass spectra) was developed. The emulation program carries out parallel reading of data from several files corresponding to different measurement channels, provides reproduction of their relative speed (including the possibility of accelerating and slowing down the experiment), and provides the ability to process this data in the main program-processor without skipping information packets. The ability to order the suspension of the experiment and photograph the state of the field cathode, including saving the results of the experiment in text files and automatically plotting the selected graphs in the Origin software package, was also built in.

An emulation tool was tested using an example of an experiment with a nanocomposite field cathode. The possibilities of the new method are shown on the example of studies that are difficult to conduct in a real experiment.

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