The technique of visualization and evaluation of the emission site distribution for large area field emitters

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Abstract. This paper presents a method for online treatment of the distribution pattern of emission sites on the emitter surface, combined with a multichannel registration and processing system of the IVC. Using computerized field emission projector we have studied nanocomposite emitter based on carbon nanotubes "Taunit M" and polystyrene. The distribution pattern of emission sites over the brightness level was obtained as well as the emission area of the sample was determined. Time dependences of the current level, the total brightness and the emission area were registered.

1. Introduction
The development of methods for creating atomic-size directional structures such as carbon nanotubes, graphene sheets and other nanoscaled materials - which have unique physical properties - has led to the production of these materials in industrial scales. Nanocomposite based on abovementioned structures are widely and intensively studied as multi-tip field emitters, which have a potential to be applied for a wide range of electronic devices (compact X-ray machines, display facilities, mass spectrometers [1-3]).

The uniformity of the emitting surface is a necessary condition for nanocomposite field emitters to operate efficiently. This uniformity can be improved by the technological optimization using widely employed control methods of emissivity such as the evaluation of current-voltage characteristics and registration of the distribution patterns of emission sites [4-6].

At the present time there are several methods for evaluating the surface uniformity of the field cathodes. Using field emission scanning electron microscopy (FE-SEM), high-resolution transmission electron microscope (HRTEM) and scanning electron microscopy (SEM) it is possible to directly observe the surface morphology of the emitter on a nanometer scale [7, 8].

The information about the uniformity of the emission properties is obtained using field emission microscope with a scanning anode (SAFEM) [9], which allows the mapping its emission properties.
However, during the scanning poorly fixed emission sites - for example strongly protruding nanotubes - might follow the scanning electrode that results in an inaccurate pattern of their distribution.

Another method for determining emission uniformity is exposure to the emitter on the resist layer sensitive to electron beam [10]. In this case, however, it is not possible to determine values of the threshold fields of individual regions of the emitter surface. Perhaps the most common method for determining the emission sites density on the field emitter surface is a method of comparison of luminous sites images on a phosphor screen of the field emission projector (FEP). [9, 11, 12]

In some studies, the number of emission sites is directly counted [13-15]. Usually this calculation is performed for nanocomposite structures, which are made in the form of regular arrays of isolated dots. In the case of a thin film field emitter, there are thousands of emission sites and their location on the surface is not ordered, thus the process of emission site counting is highly laborious. Additionally, at a regime of high voltages the entire projection screen glows evenly, thus there are some problems with the resolution of the neighboring sites. Online treatment of images could help solving the abovementioned problems. In the present work we described method of recording and analysis of spatial distribution of emission sites, coupled with the method of registration and online IVC processing.

The nanocomposite MWCNTs / polystyrene was used as a sample for studying field emission properties. Such materials are very promising for the development of the emission electronics [16].

2. Experimental
The nanocomposite was fabricated from a suspension of carbon nanotubes and polystyrene. The suspension was prepared in three steps: 1) polystyrene dissolution in o-xylene; 2) creating a suspension of nanotubes in o-xylene with ultrasonic treatment; 3) mixing of the obtained materials and ultrasonic treatment during 8 hours to increase the uniformity of nanotubes distribution in volume. Thereafter, the suspension was deposited on a polished stainless steel substrate (diameter 10 mm) using a spin coating technique. The concentration of nanotubes in the polymer matrix of the nanocomposite was 10 wt. %.

MWCNT Taunit-M ("NanoTechCentr", TSTU Tambov, Russia) were used as a filling material. They had the following parameters: length is more than 2 µm, an outer diameter is 8 to 15 nm. The production of carbon nanotubes Taunit-M is described in [17] in details. SEM image of MWCNT powder is shown in figure 1.

![SEM image of MWCNT Taunit-M.](image)

IVC measurements were carried out in a plane-parallel construction, the anode was ITO coated with a phosphor layer as shown in figure 2. The residual pressure in the chamber was ~5·10^-7 torr. Recording and processing of experimental data was carried out by a special program written in LabView 2013 environment using NI-IMAQ for USB Cameras driver software and Vision Development Module 2015.
This experimental setup was assembled in the Ioffe Institute for multi-tip emitter research. Detailed description of the implemented methods for studying the properties of the nanomaterial’s field characteristics is described in [18].

Figure 2. Experimental chamber:
1 - high-voltage input,
2 - inputs of vacuum measurement channels,
3 - emitter (MWCNT / PS),
4 - thick vacuum glass,
5 - ITO glass with a phosphor layer,
6 - chuck for ITO setting,
7 - telephoto USB-microscope (DP-M12 USB eScope),
8 - anode system,
9 - a metal cathode holder,
10 - vacuum pipe to turbomolecular pump.

The capabilities of the system includes: obtaining IVC in the mode of fast scanning half-sine voltage, plotting IVC in the Fowler-Nordheim coordinates, the calculation of the effective parameters of the emitter, such as field enhancement factor $\beta_{\text{eff}}$ and emission area $S_{\text{eff}}$. Further development of a complex research method has led to appearing new features: the registration of distribution pattern of emission sites (DPES), which is displaying on the luminescent projector screen, the calculation of the total brightness of the picture $Y$ and the estimation of its emission area $S_Y$.

3. Results
The results of the conducted experiments are considered in this section. Figure 3 shows the time dependence of the voltage level applied to the sample and the corresponding emission current in the sample stable operation with $I_{\text{max}} \sim 100 \, \mu A$. Registered DPES show that the emission site distribution is constantly changing over time.

Figure 3. Time dependence of the emission current and voltage for stable current level 100 $\mu A$. In the inserts: DPES registered with intervals of 200 s.
The maximum current – which was possible to be received using this measuring system was about 500 µA. Increase of the current led to vacuum discharges, which could damage the luminophore. It is to be noted that this instability is due to insufficient ITO conductance and the quality of the phosphor film, because similar system with a metallic anode and same emitter gave current level that exceed 20 mA. Obviously, the electron emitting streams heat the phosphor coating and lead to its destruction and deterioration of local vacuum in the interelectrode gap that increases the discharge probability.

The emitter operation at three current levels has been studied: 100, 250 and 400 µA.

We carried out determination of the emission area $S_Y$ using online treatment of the DPES in automatic mode with a given level of brightness $Y_0$ (all pixels of the recorded picture – which brighter than this level - are considered as a part of the emitting surface). For the brightness distribution analysis, the field emission image was converted into gray scales (pixel brightness from 0 to 255). The shape of the emitting surface was in the form of black-and-white pictures.

The setup of a brightness level $Y_0$ was executed from the analysis of dependence of the number of emission sites $N$ (separate regions of the emitting surface) from the brightness level of their peaks $Y_p$ for an instant DPES at emission current of 400 µA (see fig. 4a).

During the scanning of value $Y_p$ a number of emission sites continuously grew up, but starting from a certain level of brightness ($\approx 185$), there was a sharp drop in the value of $N$, due to merger of the most lackluster centers into a single luminous spots. This value we have taken as a threshold for determining the emission area and for further study of its evolution ($Y_0 = 185$).

![Figure 4. a) Dependence of the number of emission sites $N$ from the brightness level of its peaks $Y_p$; b) Dependence of the number of emission sites from the brightness level for the instant DPES at three different current levels (400, 250 and 100 µA).](image)

In order to remove emission sites with low brightness from the consideration and avoid impact of the exposure effect on the calculations, a mask was superimposed on the processed image: a brightness of all areas of the surface expelled from the emission area was taken to be equal 0. The formation of the mask was made taking account in the fluctuating nature of the site distribution pattern: surface area - which corresponds to the brightness level $Y > Y_0$ has been accumulated into a single "landscape" by collecting all registered luminescent regions on the picture during 5 minutes with the current level of 400 µA. The result is shown in figure 5.

The luminescent area observed on the projector screen was about 8 % of the total emitter area. The resulting value is the numerical assessment of the degree of emission sites uniformity on the surface of the emitter, as well as degree of its smoothness.

Figure 6a represents measured level of emission current time dependence at three voltage levels. The insets show examples of the recorded IVCs. The achieved stability of the emission current on each step was determined according to the formula $\left( I_{\text{max}} - I_{\text{min}} \right) / I_{\text{mean}} \cdot 100\%$ and amounted to: 5,5 % - to 100 µA, 7,6 % - to 250 µA and 12,4 % - for 400 µA. It can be seen that with increasing...
current level sample stability is falling. It should be related to the inclusion in the emission process new sites and corresponding increase in the adsorption stochastic fluctuations. A change in the brightness level of field emission image and a change in the value of emission area (fig. 6b) – which is determined by the predetermined level $Y_0$ - correlate with changes in the fig. 6a. It confirms that it is possible to use the brightness of the individual regions of the cathode in order to determine their own current loading. Figure 4b shows results of scanning of instant DPES using brightness level $Y_p$ (similar to figure 4a) for each of the steps in the figure 6.

Figure 5. DPES on the cathode surface: a) instant DPES before the treatment, b) black-and-white picture, where the white spots correspond to the brightness above the predetermined $Y > Y_0$, c) DPES obtained by accumulating the instant picture during 5 minutes with the current level of 400 $\mu$A, d) instant DPES received after applying the mask.

Figure 6. a) Time dependence of the voltage level and the corresponding level of the emission current (steps: 400 $\mu$A, 250 $\mu$A, 100 $\mu$A). The inset shows the instant IVCs for each step; b) Time dependences of the brightness level of image (after applying the mask) and the emission area of this pattern determined for a given brightness level $Y_0 = 185$. The insets show the instant field emission images, which were used for the calculation of the emission area.
The obtained graphs show that at the emission current level of 250 and of 400 µA distribution of emission sites brightness consists of two sections, which are close to linear form (before and after \( Y_0 = 240 \)). Possibly that some districts of emitter surface related to high brightness regions have higher temperatures and have no influence of adsorbates, whereas regions with low brightness are influenced by adsorbates on its work function and are responsible for the observed fluctuations of DPES during field emission (see figure 3).

4. Conclusion

The above described technique of multi-channel recording and online data processing of field emission of multi-tip nanocomposite cathodes is a powerful tool for the experimental analysis of the emitting cathode structure and for the defining its relationship with the macroscopic parameters such as total emission current and applied voltage. It allows obtaining the emission sites distribution on the cathode surface for emission brightness levels (which is actually the current loading levels), to estimate the total emission area over luminescent districts of the cathode.

Such numerical analysis of DPES obtained using a field emission projector, still was not carried out. The proposed approach gives great possibilities for further research of large area field emitters.

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