Field Emission Microscopy, Spectroscopy, Scanning Electron Microscopy and Field Emission Phenomena from Graphene-like Structures

Georgy Fursey, Mikhail Polyakov

Abstract— The presented paper gives a general overview of studies of field emission from carbon nanoclusters. The special attention has been given to the study of emission processes in the strong and super strong electric fields from graphene-like structures. The direct experiments have confirmed the phenomena of the low threshold field emission and explosive emission from carbon nanoclusters. Experimental data exhibiting the peculiarities of the energy spectrum of the field emission electrons emitted by graphene-like structures are presented. The new emission phenomenon connected to the high current electron emission from carbon nanoclusters (flash emission) has been discovered and reported. The application of the field emission from carbon nanoclusters cathodes are exemplified by the series of the portable x-ray devices.

Index Terms— field emission, explosive emission, low-threshold electron emission, carbon nanoclasters, nanotubes, graphene-like structure, x-ray sources

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1 Introduction

THE present report outlines the results of the study of the emission processes from the carbon nano-clusters (graphene, nanotubes and like) in the electric field, which have been carried out in our laboratory.

The special emphasis has been given to the recently noticed fact that the emission mechanism from graphene and nanotubes differs radically from the traditional mechanism of field emission (FE) from metals and semiconductors [1], [2], [3], [4]. In a number of studies [5], [6], [7], [8], [9], [10] it has been pointed out that the electron emission from nano-clusters is revealed at low voltages. The estimates of the electric fields at which the field emission is initiated show that these threshold fields fall in the range 10^4 -10^5 V/cm, that is 2 -3 orders of magnitude lower than that for traditional materials. This effect we denote as the low threshold electron emission (LTE) in the electric field. The acquisition of the reliable data on the LTE mechanism is hampered by a lack of adequate control for the micro-geometry of the surface under study. It relates to the studies of the FE in nanoclearances and also in macroclearances with the undetermined macrosurface.

This uncertainty in the knowledge of the surface topology resulted in the attempt to explain trivially the emission initiation at low voltages at the sacrifice of a primitive amplification of the electric field on the microroughnesses of the cathode surface. Such a view point has been shared by many researchers including [11]. For this reason the special attempts have been undertaken to form controllably the pointed microroughnesses on the cathode surface, or to produce the multiemitting systems in the form of vertically positioned nanotube.

The more thorough study of the surface microgeometry, applying sufficiently high resolution, has shown that the emission processes from graphene and nanotubes can't be explained in such a simplest way. In particular, it was demonstrated in the series of studies undertaken in our laboratory [2], [3], [12]. Below we present the results of these studies conducted with the help of the high resolution scanning electron microscopy (SEM).

The most informative method for studying of surfaces in situ, i.e. practically during the emission process, is the field emission projection microscopy (FEM). However, this method is applied for the study of the LTE quite seldom due to the following reasons:

1. For the nanostructures which were the main subject of many experiments, the application of the above method is troubled or even impossible because of small distances between the cathode and anode-screen.
2. The traditional FE microscopy practically excludes the opportunity to observe the emission processes on flat macroscopic surfaces.

In our studies we aimed to combine the FE microscopy of the surface with the high resolution SEM, analyzing the sample before and after the measurements. This is just the main peculiar feature of all of our studies. The FE microscopy has been carried out directly (in situ) in the high vacuum experimental volume.

A special material discovered and studied recently became graphene. This material represents itself the flat crystal with thickness of only one atom and possesses the unique nonequilibrium properties [13], [14]. One of the most important char-

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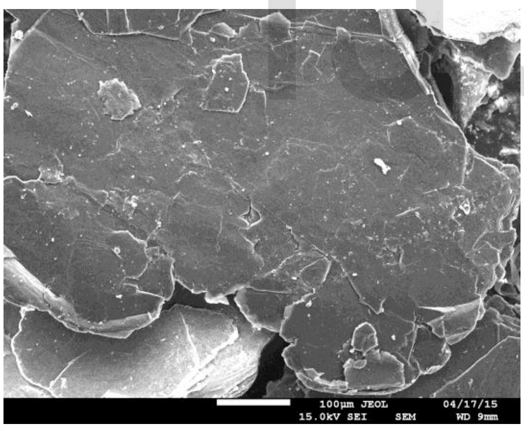
acteristics of this flat crystal is the topology of its surface. Namely this fact fostered us for the detailed study of the cathode surface using the SEM and FEM methods.

Investigation of emission processes from such structures as graphene and nanotubes (rolling-up graphene) is stimulated by their essentially nonlinear properties and is of principal interest for fundamental and applied studies. Fundamental interest stems from the fact that the emission processes carry on the important information on physical of graphene as being the essentially nonequilibrium object, while the applied significance proceeds from the opportunity to us the LTE excitation of the emission process in various nanoelectronics devices (displays, x-ray technique, microwave apparatus, etc).

The given study, besides the research of the surface microgeometry, contains the data on the current –voltage characteristics and on the energy spectra of emitted electrons as well as the results of the pulsed studies under extreme strong electric fields.

2 Subject of Study

The main studies have been carried out with carbon materials of two types. In our previous experiments we used the nanotubes obtained at formation of the cathode deposit [12]. All of the recent experiments have been fulfilled on the graphene-like structures. These structures were produced by the detonation synthesis method [2], [3]. The specimen consisted of several carbon layers (flakes), Fig.1



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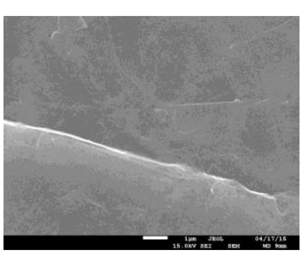
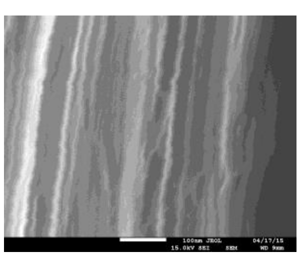


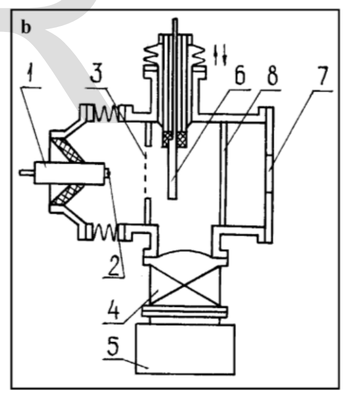


(b) (c)

Fig. 1. SEM micrographs of graphen-like flake (a), local element of flake (b), cross section of flake (c).

3 Experimental Methods

Some of the experiments were performed in the sealed glass chambers type of the Muller projector (vacuum 10^{-9} Torr) For the most part the experiments were carried out in the tailor-made vacuum chamber (Fig.2 a, b). Provision was made to perform the experiments both in stationary and pulse regimes. The field emission images were detected on the luminescence screen. The input and output channels were matched on wave impedance, resulting in possibility to measure the currents and voltages in nanosecond range. The experimental set up was provided with a special-purpose movable collector allowing for measuring the emission current. When detecting the field emission image, the collector was moved out from the detection zone with the help of the special-purpose bellows element. It gave the opportunity to obtain the emission image of the cathode-emitter surface at high resolution and large magnification on the luminescence screen. The intermediate grid placed near the cathode, provided the sufficient field intensity between the anode and cathode and served as a separation screen for cutting out of capacity currents in the course of pulse measurements. The distance between the anode-grid and the cathode could be varied in the range from fractions of mm up to few cm with the help of the special bellows. The stationary voltage was varied in the range 0 -15 kV, the pulse voltage in the range 15 -150 kV. A vacuum in the chamber during the experiment was maintained 10^{-8} -10^{-9} Torr.



(a) (b)

Fig. 2. General view (a) and construction of the experimental chamber (b). 1, high-voltage bushing; 2, emitter; 3, anode-grid; 4, vacuum pumping; 5, sorption pump; 6, electron collector; 7, window for photography; 8, fluorescent screen.

The spectrum of the field emission electrons was studied by two methods, which are the dispersion analysis and retarding potential method [15], [16]. A vacuum in the chamber was not less than 10^{-9} Torr. The basic schemes are shown in Figs. 3a,b.

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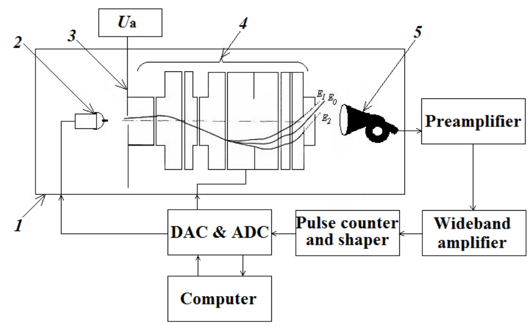

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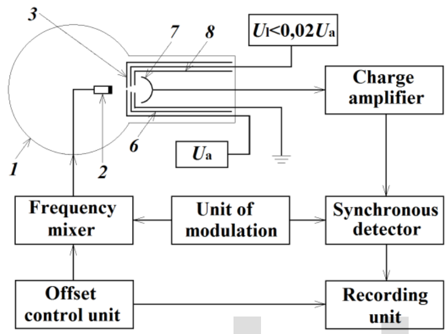


(b)

Fig. 3. Schematic diagrams of the experimental sets for dispersion analyzer (a) and retarding potential methods (b). (1- high vacuum experimental apparatus, 2- cathode, 3- fluorescent screen (anode), 4- dispersion energy analyzer, 5- secondary electron multiplier, 6- electron lens, 7- hemispherical collector, 8- protective cylinder).

4 Microscopy

The experiments were carried out with the scanning electron microscope JEOL 7001 F. The electron field images were analyzed in the field emission projection microscope (type of the Muller projector). The scheme of the projection microscopy is shown in Fig. 4. The FEM images of the emitting nanotubes are shown in Fig. 5a. The photograph obtained in the SEM is shown in Fig. 5b.

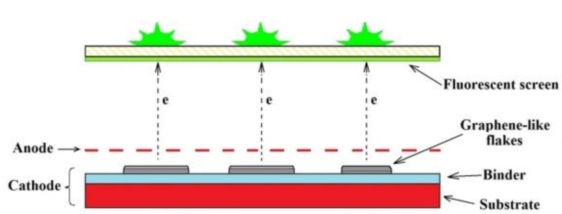
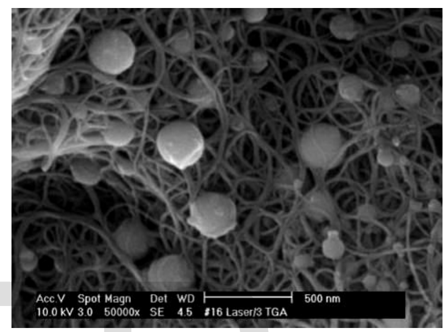


Fig. 4. FEM of multi emitter cathode.

As one can see from Fig.5a, the emission images illustrate rather uniform distribution of the emitting centers of the cathode surface.

(a)



(b)

Fig. 5. Field emission picture from nanotubes (a), SEM micrograph of nanotubes (b).

The SEM image of the cathode surface demonstrates the absence of acute angles, micropoints and protruding nanotubes. The study of different cathode fragments has shown that the nanotubes are located horizontally at the surface of the cathode-deposit. This fact allowed us already in our early works to conclude that the excitation of the emission process at low voltages does not connect with the trivial amplification of the field at local nonuniformities of the surface [12]

The direct evidence that the microninuniformities do not play a significant role in a phenomenon of the low-threshold field emission, was obtained in our studies using graphene-like structures.. We assume that the most prominent is the fact that the emission occurs practically uniformly from the whole surface of the flaky cathode. Some emission images obtained for different specimens of a graphene-like structures are shown in Figs. 6a, b. All emission images clearly demonstrate that the emission is distributed uniformly over the whole surface of a flake. Every separate flake is noticeably pronounced (Fig.6). It is important that at the emission image one can observe 2-3 elements of the overlapping flakes with clearly seen boundaries. This fact indicates that we observe the emission through one-two layers of graphene, that means that we visualize an individual graphene surface.

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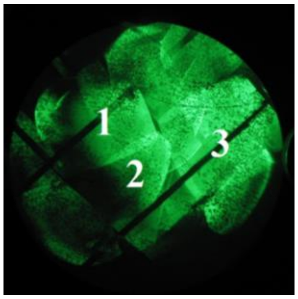
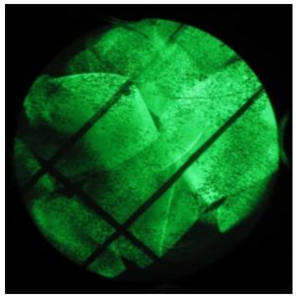
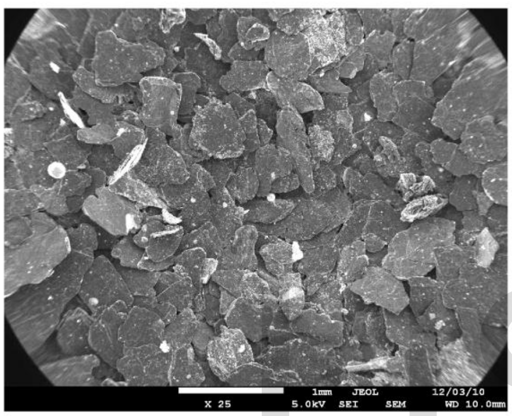




(a) (b)

Fig. 6. Field emission pictures from graphene-like structures. (1, 2, 3 – images of different flakes)



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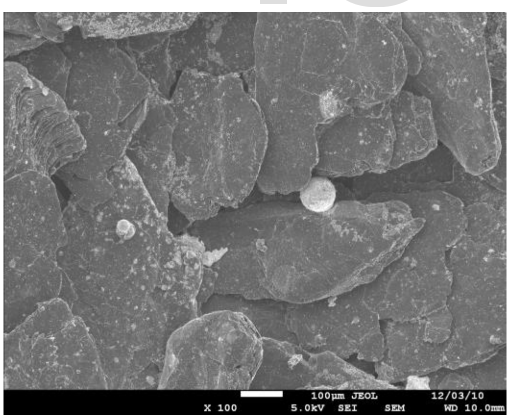

(b)

Fig. 7. General view of multi flake cathode surface in SEM (a), local fragment of multi flake cathode surface SEM (b).

After the experiment each specimen has been removed from the chamber and analyzed using the SEM. Fig.7a shows a general view of the cathode surface tightly covered with flakes. As one can see, at the surface both of the whole cathode and of separate elements the noticeable microroughnesses are not observed. The magnified images of the cathode surface are shown in Fig.7b.

5 Emission Features

As it was noted by us in a number of papers [2], [3], [12], [17] and is confirmed by the present study, the field intensity at which the emission process occurs, turned out to be several orders of magnitude lower than in the case of the traditional FEM from metals and semiconductors. Below (Table 1) given are the comparative values of the fields for the field emission, the average values of the fields for a macroclearance (Fmacro) and of the local fields at the maximal admissible field amplification on the suggested microroughnesses on the surface of carbon nanoclusters (Fmicro). F_{FEE} is threshold field strength in case of metal.

We would like to outline that the phenomenon of the low threshold is the non trivial, *paramount* effect of fundamental importance.

The current–voltage characteristics plotted in Fowler- Nordheim coordinates exhibit a linear behavior (Fig.8). This lead many researchers to the wrong interpretation, since such a

TABLE 1
Threshold Fields

F_{macro}	$\approx 10^3$-10^4 V/cm
F_{micro}	$\approx 10^5$-10^6 V/cm
F_{FEE}	$\approx 3*10^7$ V/cm

Threshold fields is the fields which is necessary for initiation emission current approximately near of 10^{-9}A.

behavior qualitatively corresponds to the traditional theory of the field emission from metals. The latter stimulated a number of corrections to the Fowler-Nordheim theory, including those prescribing the low threshold phenomenon to the microgeometry of the surface. However, the most thorough analysis of the emission dependences shows that the slope of the current-voltage characteristics in Fowler-Nordheim coordinates appeared to be very small compared with that of metals. The attempt to determine the work function by the slope of the current-voltage characteristics, at the suggested most largest geometrical factors 10, 100 times, results in physically absurd small values of work functions (10^{-2}-10^{-1} eV) [2], [3], [5].

Inapplicability of the Fowler-Nordheim theory to the interpretation of the low-threshold effect follows also from the fact that the effective electron mass (m*), determined by other methods [2] (the field effect in electrolyte, etc) turns out significantly less than unity and consists only 10^{-2}-10^{-1} m_0, where m_0 is the free electron mass. Such a small m_0 points to the essentially different dispersion low. The dispersion law is not quadratic ($E \sim p^2$) but linear ($E \sim p$) that corresponds to the relativistic case. A number of ideas were put forth, which were connected with effect of the size quantization and resonance tunneling [18], [19]. At present an unambiguous interpretation is absent. There are attempts of the theoretical analysis based upon the idea of the size quantization and resonance tunneling [2], [4]. The preliminary results of calculations, according to this mod-

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el, do not contradict to the idea of size quantization, but do not allow researchers to obtain the accurate quantitative estimates and analytical dependence of the emission current on the electric field.

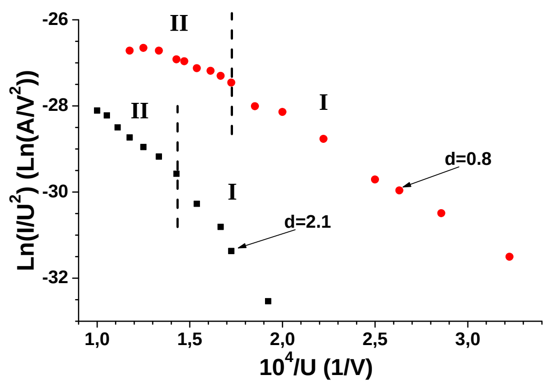

Fig. 8. Field emission current-voltage characteristics from graphene-like structures (d – vacuum gap).

A peculiarity of the experimental current-voltage characteristics is the presence of a region of a weak current growth with increasing field (the region II in Fig.8). Such characteristics are superficially similar to those observed in the case of high resistance semiconductors of n- and p-types (look ch.5 in [1]) [20]. The behavior of the current-voltage characteristics can indicate that in the case of graphene we also meet the limitation in a number of carriers (electrons) and the low density of energy states at the surface.

The important information on the origin of the low-threshold emission could be obtained via a study of the *energy spectrum of emitted electrons*. The results of the study of the field emission electron distribution over total energies (TED) with the help of dispersion analysis were presented in our paper [16]. The results of more detailed study of the TED in a wide range of electric fields [15], using the retarded potential method (look in p. 80 in [21]).

The TED curves are shown in Fig.9. The curves 1 – 5 were plotted for our graphene-like structure at the consequently increasing anode voltage. The curve 6 is the reference one, obtained for tungsten. The energy of a free electron in vacuum is assumed to be a zero energy.

The peculiar features of the TED which were found in our experiment are:

1. The significant shift of the energy distribution maximum to the region of low energies in respect to the tungsten maximum.
2. The detection of the second maximum. The maximums are displaced from each other by 0.7 – 1.7 eV.
3. The study of the local regions of the emitting surface with the help of the probing and emission microscopy has shown that a shape of the TED curve is different for different local regions of the emitting surface [15].

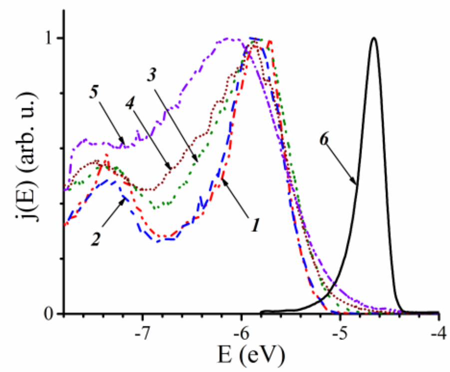

Fig. 9. TED curves of the field emission electrons by retarding potential method [15].

6 Explosive electron emission (EEE)

The EEE is a special type of emission discovered on metals in super strong electric fields (10^8 -10^9 eV). Initially it was established that the electron emission appears at the cathode surface if the current density of the conventional field emission exceeds the critical values of the order 10^9 A/cm^2. It was shown that the EEE currents can be 10^2, 10^3 and even 10^6 A. In the course of direct experiments [17], [22], [23] it was found that the EEE is excited when the local regions of the cathode, through which the high density current is flowing, are exploded by this current.It was also shown that at the moment of explosion the current increases by many orders of magnitude.

This powerful emission process occurs at the conversion of a condensed cathode material (solid or liquid) into plasma [23], [24]. It turned out that this act of emission is not unique. In pulse regimes it reproduces with high accuracy. It was shown that the initial plasma, originated at the explosion of the local center, propagates along the cathode surface and results in appearance of new emission centers.

A phenomenon of the EEE is stationary until the plasma close the vacuum clearance, which provides the high intensity electron emission. The analysis of the experimental results concerning the explosion of pointed field emitters in 1963 [25], 1965 [26] (see in [23]) allowed in due time the author of the given paper to understand the mechanism and construct the model of this process. The model of the EEE development is schematically shown in Fig.10a. Mesyats and coworkers obtained, with the help of nanosecond photography, the consequent frames of the plasma luminescence at the initial stages of a vacuum breakdown (Fig. 10b). The obtained results confirmed the generalization made on the base of experiments with the pointed emitters [23]. The combination of these results permitted to conclude that in the extremely strong fields the principally new kind of electron emission is excited. This phenomenon, according to the principle of excitation, has been called as the explosive electron emission and was recognized as the joint Discovery [27] with priority from 1966.

Later on the EEE phenomenon was observed on different materials, liquid metals [28] and semiconductors [20].

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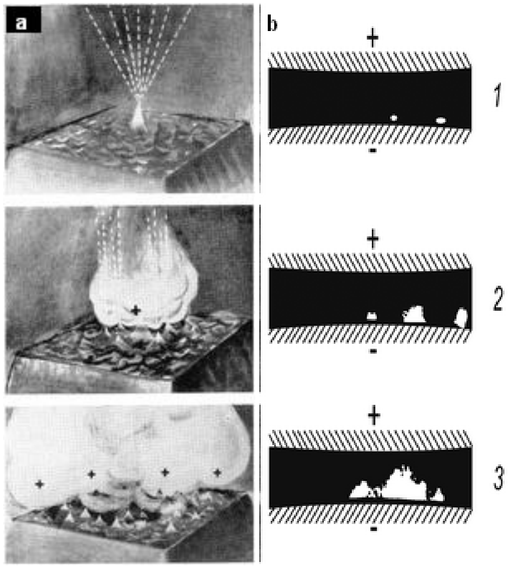
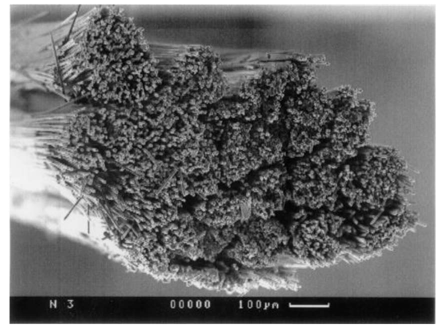


(a) (b)

Fig. 10. Initiation and development of explosive emission process in model [23] (a), statistical photos of the plasma propagation [24] (b) (1, 2, 3 -sequence of stages).



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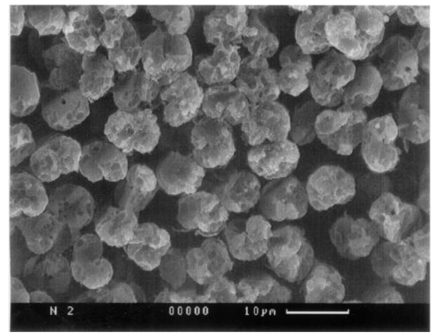


(b)

Fig. 11. Fragment of the cathode from PAN fiber (a), local part of PAN fiber surface (b)

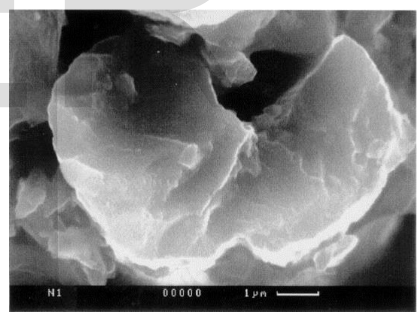
7 THE EEE FROM CARBON NANOCLUSTERS.

The EEE exhibits the peculiar features in the case of carbon materials, namely, carbon nanoclusters. This is due to the fact that carbon possesses a very high melting temperature and at the conventional pressures it is practically impossible to transfer it to a liquid state. Nevertheless, it was found that in the EEE process there exists a liquid phase on the carbon cathode surface [17], [29]. It has been established that on the liquid carbon surface the nanocapillary waves were generated, whose apexes served as new emission centers. These nanoasperities were found to be essentially smaller than that formed on the metal surfaces. The latter, as it was shown, results in the small erosion and mass transfer in the EEE process, and provides the significant increase of the stability and duty cycle of the EEE cathodes manufactured from carbon nanoclusters. We studied several types of carbon nanoclusters: polyacrylonitrile fibers (PAN fibers), nanotubes and graphene-like structures.

8 THE EEE FROM PAN FIBERS

A fragment of the cathode specimen manufactured from PAN fiber is shown in Fig.11a, b.

Fig. 12 shows the comparison of the fiber surfaces before and after the EEE excitation. As is clearly seen, the surface of the carbon cathode is melting in the process of the EEE. The traces of the melting are seen in Fig 12b. We have shown that in the EEE process the nano-asperities of only 20-30 Å are formed at the carbon cathode surface [17]. Namely, the generation of such nanoasperities at the surface of the EE cathode, leads to the high uniform emission and stability of cathode functioning.



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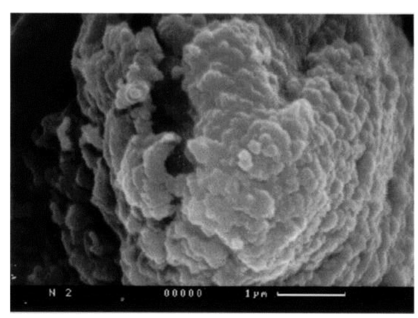

(b)

Fig. 12. PAN fiber surfaces before (a) and after the EEE excitation (b)

The PAN-fiber cathodes were used for the development of our x-ray tubes with a non-incandescent cathode [30] (Fig. 13)

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in the portable x-ray apparatus (Fig 14).

Fig. 13. X-ray tube.

Fig. 14. The portable x-ray apparatus.

The functional scheme of device is shown in Fig. 15.

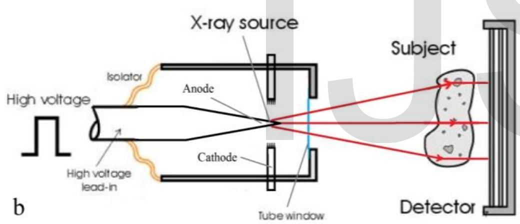
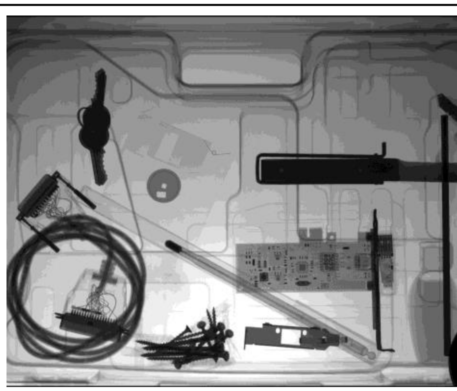


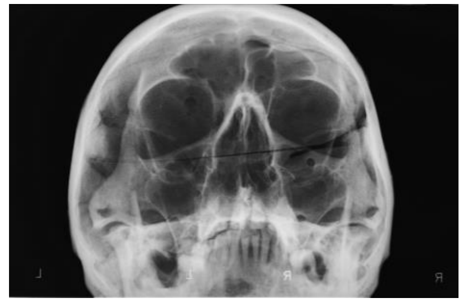
Fig. 15. The functional scheme of X-ray device.

Some of x-ray images are presented in Fig 16a, b, c.



(a)

Fig. 16. X-ray images: different subjects: inside the bag (a)



(b)

(c)

Fig. 16. X-ray images: different subjects: human skull (b), birdie (c)

7 The Emission from the Graphene-like Structures in the Extremely Strong Electric Fields. Flash Emission.

In most recent experiments we observed the peculiar behavior of the emission process. It has been found that at the switching on of the nanosecond pulse the emission current acquires the character of the “flash”. The current is emitted only during the short period of time compared to the pulse length of the applied voltage (Fig 17).

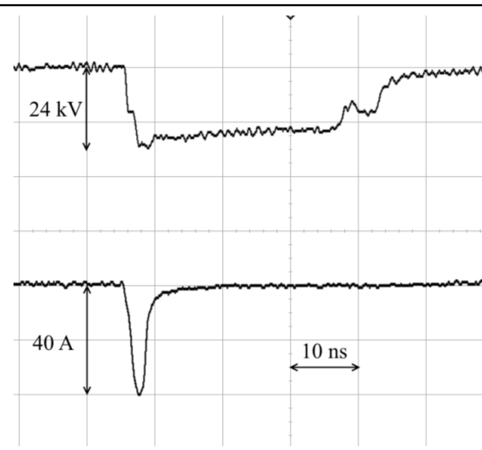


Fig. 17. Flash emission oscillogram.

The voltage was varied from 20 to 150 kV. The current were from 20 to 100 A, depending on the voltage. While generating of a current, we didn’t observe an appearance of the luminous plasma, as it usually takes place at the EEE event. It was noticed that with increasing voltage the current also rises, but the pulse duration decreases with retention of the total area under the oscillogram trace (Fig. 18a, b). This fact permits to con-

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clude that in strong pulsed electric fields it is possible to realize the powerful high current emission process. Along with that, the value of the electron charge that could be taken away by EEE at the different values of field intensity remains constant (qe= It = const). The emission patterns before and after the "flash"- event remain unchanged.

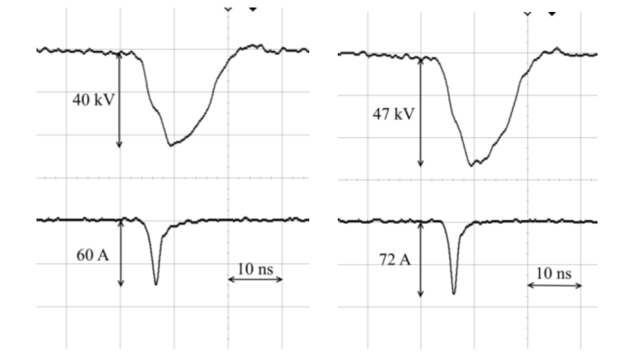

Fig. 18. Flash emission oscillogram for different voltages.

At present there doesn't exist any unambiguous interpretation of this new effect. One can only assume that the limitation of the emission current in time is connected with the fact that in the graphene-like structures takes place the restricted density of electron states in near- surface regions of the specimen and the accumulation of the electron charge up to the limit on them. It indicates also that the inflow of the electrons from the inner areas to the surface is partially blocked. It is possible that in this particular case we deal with, so called, Coulomb blocking [31].

8 Conclusion

1. The direct experiments confirm the existence of the specific phenomenon which is the low threshold emission from graphene-like structures. It was shown that the fields, at which the electron emission is initiated, are 100-1000 times lower than those necessary for the excitation of the traditional field emission from metals and semiconductors. On the base of the obtained results we can conclude that the emission from carbon nanoclusters and, specifically, from graphene-like structures is the particular kind of emission whose mechanism cannot be explained by the Fowler-Nordheim theory.
2. It has been convincingly shown that the uniform emission can be realized over the surface of a separate fragment of the graphene-like structure as well as of whole surface of the field emission cathode (Fig. 6a, b)
3. The specific features in the energy spectrum of electrons emitted from graphene-like structures have been found. They are the presence of two maximums on the TED curves, the broadening of the TED as opposed to metals. It has been shown that electrons from graphene-like structures were emitted from the energy levels which are lower than the Fermi level of metal.
4. It has been demonstrated that the formation of the low-threshold high-current EEE from PAN fibers and graphene-like structures is viable.
5. The effect of the Flash emission has been discovered. The perspectives of using of the low-threshold high-current emission in the x-ray technology of the new generation are proposed.

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