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Emission Characteristics of Pressed Pd-Ba Cathodes with an Increased Homogeneity of Distribution of the Emission Active Phase on the Surface

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Abstract: The universal parameter that characterizes the emission capacity of a cathode material is its electron work function which has a definitive impact on the emission current. Reducing the work function is a top-priority task for the designers of all types of cathode materials. The emission properties of composite cathode materials are determined primarily by the properties of their work surface. The investigation of the connection between the structure and physical and chemical properties of the cathode surface and its emission properties is a relevant problem. The understanding of these properties not only gives a mode in-depth perception of the physics behind the processes that occur on the surface of the cathode but also enables purposefully adjusting its properties by reasonably choosing the parameters of the primary components and improving the cathode manufacturing technology. An important parameter of the composite material is the emission homogeneity of its surface which is determined primarily by the homogeneous distribution of the emission active phase and the chemical and structural homogeneity of the work surface of the cathode. This research used methods of scanning electron microscopy and electron probe microanalysis to study the effect of the grain-size composition of primary powder components of a pressed Pd-Ba cathode on the evenness and density of distribution of powder particles of the emission active phase Pd_5Ba on its surface. The optimal particle size distribution of powder components was determined. An experimental technique for manufacturing pressed Pd-Ba cathodes was proposed, it enables increasing the evenness of the distribution of the emission active phase across the surface of the cathode. The increase in emission homogeneity reduced the work function of the electron from 2.5-2.4 eV which resulted in a more than 2.5 fold increase in emission current density.

Key words: Grain-size, composition, emission active phase, electron, microscopy, research, pressed Pd-Ba cathode, emission homogeneity

INTRODUCTION

The main type of effective cathode that is used in modern vacuum-tube microwave devices is the metal-allow cathode in which the active metal is present in the form of an intermetallic compound (Lopin, 2014; Yesaulov, 2016; Pashkov *et al*., 2011). Magnetrons with selfheated actuation (with a "field emission cathode") which are of interest due to the instantaneous transition of the device into generation mode (not more than 0.5 s) (Li and Bondarenko, 2012; Djubua and Polivnikova, 2015; Li *et al*., 2016), use thermal secondary emission cathodes made out of a Pd-Ba alloy. In recent times, Pd-Ba cathodes made through the powder metallurgy technique out of a mixture of Pd powders and an emission active component the intermetallic compound Pd_5Ba have become widespread (Polivnikova and Li, 2013). However, despite the longstanding use of metal-alloy cathodes, many issues related to the physical and chemical processes that occur on the work surface during their actuation and operation remain understudied.

Most researchers believe that the high emission capacity of Pd-Ba cathodes is caused by the formation of a monoatomic Ba film on the emission surface, the film forms during the thermal dissociation of phase Pd_5Ba particles:

$$Pd_5Ba \rightarrow 5Pd+Ba$$

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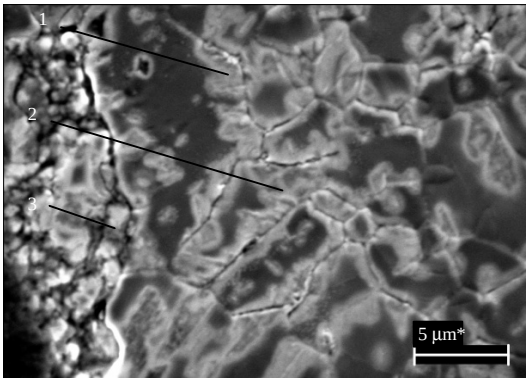

Fig. 1: Micrograph of a Pd-Ba cathode; 1: (Pd matrix); 2: (BaO film) and 3: (Pd_5Ba phase)

Despite the losses from evaporation and the destructive effect of ion and electron bombardment, residual gas poisoning and sputtering from other electrodes of the device, the Ba film is reproduced on the surface of the cathode through diffusion and migration of the active metal from the emission body throughout the service life of the device (Djubua *et al*., 2008; Lozovan, *et al*., 2014). On the surface of the cathode, the Ba film is catalyzed by oxygen that is present in the atmosphere of the residual gases of the device (Djubua and Korolev, 2011).

However, the contribution of these processes and that of Knudsen flows of Ba from open pores and phase Pd_5Ba particles onto the surfaces during the formation of the Ba film surface remains understudied. Figure 1 shows an electron microscopic image of a Pd-Ba cathode.

X-ray microanalysis has found that the darkest area (1) corresponds to the Pd matrix, the lighter area (2) is a thin BaP film that forms due to the diffusion of Ba from the volume along the edges of grains and area (3) is the Pd_5Ba phase particle.

Thus, the emission surface of the Pd-Ba cathode is a mosaic structure that comprises of areas ("spots") with a varying elemental composition and work function: Pd matrix-4.8 eV; Pd_5Ba phase particles-3.7 eV; BaO oxide-2.3 eV (Baiburin *et al*., 2002; Komai *et al*., 1999) in other words, the surface is emission-nonhomogeneous.

The emission in homogeneity of the cathode surface destabilizes the operating parameters of devices: causes fluctuations and misfire, reduces the power of generated fluctuations and significantly affects high-frequency noises (Kislitsyn, 2015). In order to search for ways to increase the emission homogeneity, it is necessary to study the connection between the emission properties of the cathode and the composition and state of its surface (Swartzentruber *et al*., 2014; Djubua *et al*., 2012). The main parameter that affects the elemental-phase composition of the surface of Pd-Ba cathodes that are manufactured via. the powder metallurgy technique is the particle size of primary powder components.

This research examined the effect of dispersity of primary powder components and regimes of manufacturing of pressed Pd-Ba cathodes on their emission characteristics.

MATERIALS AND METHODS

The morphology and elemental composition of the surface of samples of pressed Pd-Ba cathodes was studied through scanning electron microscopy and X-ray microanalysis using a Zeiss EVO 40 scanning electron microscope (Germany) equipped with an SDD X Flash 1106 energy-dispersive silicon drift chamber detector.

Cathode samples were placed in niches on a special holding table which enabled conducting studies at various stages of their manufacturing without any contamination or damage of the sample surface. The studies were conducted at $p \leq 6 \times 10^{-4}$ Pa residual gas Pressure in the microscope chamber, 20-25 kV accelerating voltage, 25-12 mm working distance, 70-150 pA electron probe current in high-definition mode and 0.9-1.5 nA in elemental microanalysis mode.

The Pd_5Ba phase particle count and the percentage of the cathode surface corresponding to the emission active phase was determined by analyzing micrographs using ESPRIT Feature Software and the ImageJ Software package.

Current-voltage characteristics were measured using a laboratory rig for studying the emission properties of cathode materials at T = 1000°C and $p \leq 1 \times 10^{-6}$ Pa residual gas Pressure. Samples of Pd-Ba cathodes were manufactured using PdAP-1 Pd powder (Russia, GOST R 52244-2004 state standard).

The Pd_5Ba intermetallic compound with a barium content of 18-19% was obtained through argon arc melting of pure primary components (Li and Bondarenko, 2012; Pashkov *et al*., 2016). The precise concentration of Ba in the intermetallic compound was determined through gravimetric analysis. In order to obtain emission active phase powder ingots were ground in a Retsch MM 200 vibrating mill (Germany). The necessary fractions of Pd and Pd_5Ba powders were isolated using the Retsch AS 200 digit analytical sieve shaker (Germany).

Samples of pressed Pd-Ba cathodes were made according to the standard manufacturing technique (Li and Bondarenko, 2012) that consists of the following main operations:

- Preliminary preparation of primary powder components
- Calculation of the amounts of Pd powder and Pd_5Ba phase powder necessary to ensure the content of Ba in the cathode material at 1.8-2.0% of weight
- Production of a homogeneous physical mixture of powder components (press powder) in the vibrating mill
- Pressing of cathode workpieces through single-ended cold pressing in an enclosed metallic mold using a VANEOX PR-25A-HD semi-automatic press at R_{sp} = 8.5-9.5 t/cm^2 specific pressing pressure
- Sintering of pressed cathode workpieces in a vacuum on a ceramic base for 90 min at T = 1050°C and $p \leq 5 \times 10^{-3}$ Pa residual gas pressure
- Repressing secondary pressure treatment of sintered cathode workpieces with subsequent vacuum annealing for 30 min at T = 750°C and $p \leq 5 \times 10^{-3}$ Pa residual gas pressure. This operation is carried out until the required thickness of the item is achieved
- Lathing of the side surface of the cathode until the necessary diameter of the item is achieved followed by vacuum annealing for 30 min at T = 750°C and $p \leq 5 \times 10^{-3}$ Pa residual gas pressure

Permissible deviations of cathode dimensions should not exceed ±0.01 mm.

RESULTS AND DISCUSSION

Samples of Pd-Ba cathodes were studied in three stages. During the first stage, we studied the effect of dispersity of the primary powder components on the particle count of the Pd_5Ba emission active phase on the surface of cathode samples, the evenness of their distribution and the percentage of the emission surface area that was occupied by phase particles. To that end three types of Pd-Ba cathode samples were manufactured which had different particle sizes of powder components. Table 1 shows the powder fraction particle size of Pd and Pd_5Ba powders that were used to manufacture samples of type 1-3 cathodes.

Each press powder composition was used to manufacture five samples in the shape of flat pellets 0.8 mm thick. In order to obtain the full picture of distribution of Pd_5Ba phase particles across the surface of cathodes, electron-microscopic examinations were conducted before the lathing operations.

The micrographs of the surfaces of these three cathode samples are presented in Fig. 2. Elemental analysis showed that that the dark areas on images corresponded to the shape-forming material Pd while light areas corresponded to Pd_5Ba phase particles.

We discovered an uneven distribution of Pd_5Ba phase particles which was typical for samples of all grain-size compositions. The emission surface of samples features a circular area ~300-400 μm thick with an increased concentration of Pd_5Ba phase particles while in the central part of the surface of all samples, the distribution of phase particles was even. The uneven distribution of Pd_5Ba phase particles manifested itself to a greater extent on the surface of type 1 and 2 samples (Fig. 2a and b). On the surface of type 3 samples, edge rings with an increased concentration of phase particles either were absent or faint (Fig. 2c):

- Area enriched by phase particles
- Area depleted by phase particles

Figure 3 shows a fragment of the surface of a type 1 cathode from which one can see that phase particles in

Table 1: Powder fraction particle size of Pd and Pd_5Ba powders that were used to manufacture samples of type 1, 2 and 3 cathodes

	Powder fraction particle size (μm) (sample)		
Press powder component	Type 1	Type 2	Type 3
Pd_5Ba	20-45	20-45	45-63
Pd	20-45	45-63	45-63

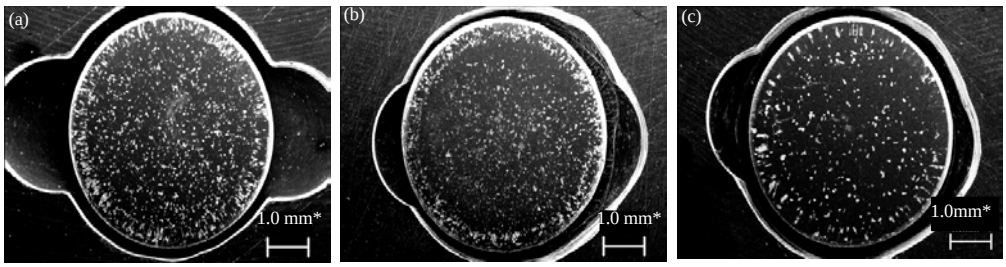

Fig. 2: Micrographs of cathode sample surfaces: a) Type 1; b) Type 2 and c) Type 3

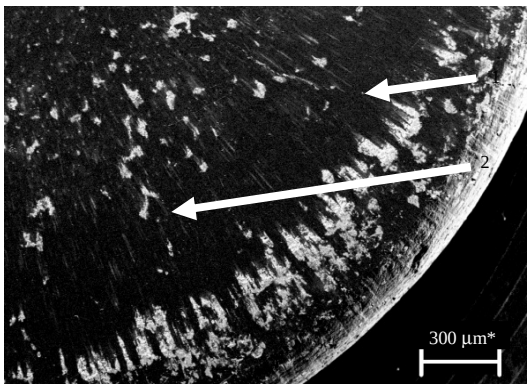

Fig. 3: Micrograph of a surface fragment of a cathode sample

Table 2: Count, mean diameter of phase Pd_5Ba particles and percentage of area occupied by these particles in samples of type 1, 2 and 3 cathodes

Samples	Particle count units on the surface	Mean particle diameter (μm)	Total particle area (%)
Type 1	1898	23	3.88
Type 2	1243	33	3.91
Type 3	352	59	3.86

the edge area have an elongated shape, strictly oriented in the radial direction, while in front of the area with an increased concentration of the Pd_5Ba phase (1) there is a circular area with a reduced concentration of phase particles (2).

We analyzed the micrographs of sample surfaces to study the effect of dispersity of primary powder components on the amount of emission active substance on the surface of cathode samples with a view to choosing the optimal grain-size composition of the press powder.

We chose the following parameters of objects for measurement: Pd_5Ba phase particle count in the analyzed area; mean particle diameter; percentage of surface area occupied by phase particles. Table 2 shows the results for three types of cathodes with an averaging of the dimensions of 5 samples of each type.

Table 2 shows that the percentage of the area occupied by Pd_5Ba phase particles on the emission surface of type 1, 2 and 3 samples is virtually identical and is 3.88, 3.91 and 3.86% of the total area of the emission surface, respectively. The maximum phase particle count was discovered on the surface of type 1 samples. Despite the fact that type1 and 2 samples used the same Pd_5Ba powder fraction (20-45 μm) the phase particle count on the surface of these samples differed by ~30%, while the mean Pd_5Ba phase particle size in type 1 samples was 10 μm smaller than that in type 2 samples. This is probably related to the difference in the particle sizes of the shape-forming Pd powder (Table 1).

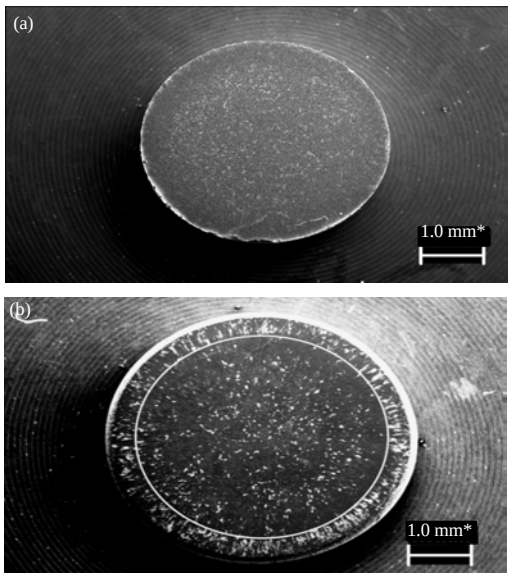

Fig. 4: Micrograph of a cathode sample surface after sintering; a) After repressing and b) The circle marks the diameter of the sample before repressing

Thus, the particle size of Pd and Pd_5Ba powders had practically no effect on the total area of the emission active Pd_5Ba phase on the surface of cathode samples. However, the emission active phase particle count is affected by not only the size of Pd_5Ba phase particles but also the particle size of the shape-forming Pd powder.

The goal of the second stage of the experiment was to figure out the reasons behind the formation of edge rings with increased Pd_5Ba particle concentration on the surface. To that end, 5 samples of type 1 cathodes were manufactured according to the standard technique. Electron-microscopic examinations and X-ray microanalysis of the surface were conducted during each stage of manufacturing.

The studies showed that after cold pressing, the distribution of Pd_5Ba phase particles across the surface was even and had no radial dependency. After solid-phase sintering, we detected an insignificant shrinkage of the material the diameters of samples shrunk by ≤1%. The comparison of micrographs of the surface after pressing and after sintering showed that no displacement of Pd_5Ba phase particles was present during the sintering process. After repressing, the diameters of samples grew by 0.6-0.8 mm. This is explained by the fact that during repressing, the punch motion occurs until it hits the distance ring that sets the precise thickness of the product. The diameter of the distance ring exceeds the diameter of the sintered sample which enables the cylindrical cathode workpiece to "spread" in the radial direction under the impact of punch pressure. Figure 4

Table 3: Difference in the manufacturing regimes for Pd-Ba cathode samples according to standard and experimental techniques. Pressed sample sizes

				Size (mm)			
		Specific pressure (t/cm^2)		After sintering		After repressing	
Techniques	Press powder amount (g)	Pressing	Repressing	d_1	h_1	d_2	h_2
Standard	0.250	8.5-9.50	9.5-10.5	4.06	1.74	4.79	0.80
Experimental	0.190	10.5-11.5	1.0-1.50	4.02	1.05	4.29	0.80

shows micrographs of the cathode sample surface after sintering and after repressing. The circle marks the diameter of the sample before repressing.

The micrograph shows that the concentration of Pd_5Ba phase particles increased specifically in the edge area. At the same time, the concentration of particles of this phase dropped significantly in the circular area that was located closer to the center.

The third stage of the study was aimed at eliminating the causes of displacement of surface Pd_5Ba phase particles towards the edge of the sample. To that end, the standard manufacturing technique was adjusted. The primary Pd powder underwent vacuum annealing at a temperature of T = 800°C for 30 min at $p \leq 1 \times 10^{-4}$ Pa residual gas pressure. The amount of press powder that was used to manufacture one sample was reduced from 0.250-0.190 g. The specific pressing pressure $P_{sp.}$ was increased from 8.5-9.5 to 10.5-11.5 t/cm^2. Five type 1 cathode samples were manufactured according to this technique (Table 1).

Previously, it was shown that thermal treatment of primary Pd powders stabilized the particle size distribution and increased the evenness of component distribution in pressed Pd-Ba cathodes (Li and Bondarenko, 2012). Powder annealing increases the plasticity of particles and improves its compressibility while powder compaction due to particle deformation occurs at lower pressures.

Table 3 shows data on the difference in the manufacturing regimes for Pd-Ba cathode samples according to standard and experimental techniques as well as sample diameters and thicknesses after sintering (d_1 and h_1) and after repressing (d_2 and h_2).

The data show that the adjustment that were made to the sample manufacturing technique allow drawing closer to the required thickness of the cathode after pressing. The mean thickness of pressed and sintered samples was 1.05 mm as opposed to 1.74 mm of samples that were manufactured according to the standard technique. As a direct result of this, repressing required considerably lower pressure: 1.0-1.5 t/cm^2 instead of 9.5-10.5 t/cm^2. After repressing, the diameter of the sample increased by 6.7% as opposed to 17.9% when manufactured according to the standard technique.

The distribution of Pd_5Ba phase particles across the surface of experimental cathode samples was even without increased concentrations in the edge areas (Fig. 5). The mean percentage of the surface area that was

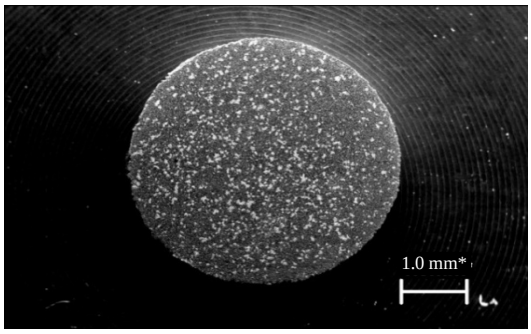

Fig. 5: Micrograph of the surface of a sample that was manufactured according to the experimental technique, after repressing

occupied by the emission active material was 3.85% while the mean count of Pd_5Ba phase particles on the surface was 1590 particles. The reduction of the area of emission active material on the surface and the decrease in the Pd_5Ba phase particle count is related to the reduction of the amount of press powder that was used to manufacture one sample.

In order to correctly compare the emission characteristics of cathode samples that were manufactured according to the standard and experimental techniques, their diameters were brought to the required size of 4.2 mm by lathing the side surface. At that the area of the emission surface of the samples that were manufactured according to the standard technique reduced by 23.2%, while that of samples that were manufactured according to the experimental technique reduced by 4.1%. The analysis of micrographs showed that after lathing, an average of 1507 Pd_5Ba phase particles were located on the surface of experimental samples and 1176 particles on the surface of standard samples. Thus, the manufacturing of samples according to the experimental technique increased not only the evenness of distribution of Pd_5Ba emission active phase particles across the surface but also the density of their placement which increased the emission homogeneity of the surface.

The universal parameter that characterizes the emission capacity of a cathode material is its electron work function. Even a slight reduction of the work function increases the thermal emission current magnitude significantly. The current-voltage characteristics of cathode samples were measured to determine the work

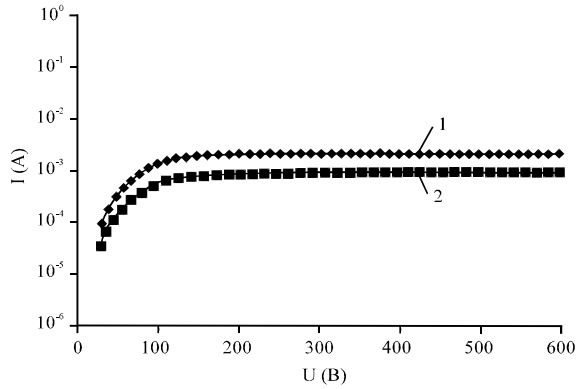

Fig. 6: Current-voltage characteristics of the experimental; 1: Standard and 2: cathodes

function. The work function was determined according to the net current method (Dobretsov and Gomoyunova, 1966).

Figure 6 shows the current-voltage characteristics that were measured for the standard and experimental samples at a temperature of 1273 K. The obtained diagrams were used to determine the levels of saturation currents with a zero field on the cathode (I_0).

For the experimental sample, $I_{0e.} = 8.0\times10^{-4}$ A; for the standard sample, $I_{0st.} = 3.7\times10^{-4}$ A; the saturation current density which equals the saturation current per unit of emitter area was $j_{0e.} = 6.3\times10^{-2}$ A/cm^2 and $j_{0st.} = 2.5\times0^2$ A/cm^2, respectively.

The averaged work function of thermoelectrons was calculated according to the Richardson-Dushman equation:

$$j_o = A_o T^2 \exp\left(-\frac{e\varphi}{kT}\right)$$

Where:

j_o = The saturation current density
A_o = The Sommerfeld's universal constant = 120.4×10^4 A/K^2·cm^2
T = The cathode temperature (K)
e = The electron charge = 1.602×10^{-19} K
φ = The electron work function
k = The Boltzmann constant = 1.38×10^{-23} J/L

The average work function was 2.4 eV for the experimental sample and 2.5 eV for the standard sample. Apparently, emission characteristics were improved by the emission homogeneity of the surface of the cathode.

The presence of areas with radically different concentrations of the emission active substance on the surface inevitably causes a macro-emission inhomogeneity of the cathode surface. The detected cluster of Pd_5Ba phase particles on the edges of samples will cause not only emission inhomogeneity of their surface but also an unpredictable alteration of the phase concentration during lathing which is carried out to achieve the necessary diameter of the cathode.

It was obvious that said inhomogeneity of Pd_5Ba particle distribution emerged during the manufacturing of the cathode. The results of electron-microscopic examinations of the cathode surface during various stages of its manufacturing showed (Fig. 4) that the inhomogeneous distribution of the phase emerged after repressing. Apparently, the impact of the punch force displaces surface phase particles towards the edge area. The formation of radially extended particle clusters can be explained by their easier displacement along trajectories in the Pd matrix. Weak manifestation of edge rings on the surface of type 3 samples is apparently determined by larger Pd_5Ba phase particle sizes in these samples. By using a larger powder fraction, it is possible to reduce considerably the number of Pd_5Ba particles on the surface of samples (Table 2). It is also worth noting that due to the greater area of adhesion to the Pd matrix, larger particles are better fixated in the material (Li *et al*., 2015) and more effort is required to displace them.

The adjustments that were made in the cathode manufacturing technique significantly increased the evenness of the distribution of Pd_5Ba phase particles. However in order to achieve homogeneous emission properties of the cathode, it is important to ensure not only the even distribution of emission active phase particles across the surface but also the density of their placement. The analysis of micrographs (Table 2) showed that with an identical area occupied by Pd_5Ba phase particles on the surface of the cathode, the greatest density of their placement was found in type 1 cathode samples.

The measurement of current-voltage characteristics showed that cathode samples that were made out of the press powder with an optimal grain-size composition according to the experimental technique had better emission characteristics than samples that were manufactured according to the standard technique had.

CONCLUSION

The optimal ratio of the percentage of the area occupied by the emission active material to the density of placement of Pd_5Ba phase particles on the surface was discovered in type 1 cathode samples: 20-45 μm Pd powder particle size; 20-45 μm Pd_5Ba powder particle size.

Edge areas with an increased concentration of Pd_5Ba phase particles were discovered on the surface of the cathodes; the mechanism of their formation was set forth. An experimental cathode manufacturing technique was developed which enabled increasing the emission homogeneity of their surface.

The experimental technique of manufacturing of pressed Pd-Ba cathode workpieces reduced the average work function of the thermoelectron from 2.5-2.4 eV which resulted in a more than 2.5 fold increase in emission current density.

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