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改进碳纳米管电接触及粘贴性能的一体式 冷阴极制作

李玉魁1,王凤歌1,刘兴辉2,卢文科3,曾凡光4

(1 中原工学院 电子信息学院,郑州 450007)
(2 辽宁大学 物理学院,沈阳 110036)
(3 东华大学 信息科学与技术学院,上海 201620)
(4 郑州航空工业管理学院 数理系,郑州 450015)

摘 要:在阴极玻璃面板上研发了一种新型的一体式冷阴极.印刷的银浆被烧结后用于形成银底电极.制备了薄层底电极浆料,其中含有大量碳纳米管.将薄层底电极浆料印刷在银底电极表面,然后 再将普通碳纳米管浆料制作在烘烤的薄层底电极浆料上.利用高纯度氩气作为保护气体,在烧结炉 中对这两种浆料同时进行烧结.烧结后的薄层底电极将和银底电极相互融合在一起,碳纳米管层则 覆盖于薄层底电极的表面.同一阴极像素中制作了两个碳纳米管发射极.备用碳纳米管发射极的存 在,有利于延长整体显示器的使用寿命.利用薄层底电极作为碳纳米管层和银底电极之间的中间 层,能够有效改善碳纳米管的粘贴性能,同时增强二者之间的可靠欧姆接触.利用碳纳米管作为阴 极制作了一体式冷阴极场发射显示器.该显示器具有良好的发光图像质量以及更好的场发射特性. 与普通碳纳米管阴极场发射显示器.该显示器具有良好的发光图像质量以及更好的场发射特性. 与普通碳纳米管阴极场发射显示器.该显示器具有良好的发光图像质量以及更好的场发射特性. 与普通碳纳米管阴极场发射显示器.该显示器具有良好的发光图像质量以及更好的场发射转性. 与普通碳纳米管阴极场发射显示器.该显示器具有良好的发光图像质量以及更好的场发射特性. 与普通碳纳米管阴极场发射显示器相比,一体式冷阴极场发射显示器能够将开启场强从 2.11 V/ μ m 减小到 1.68 V/ μ m;将最大场发射电流从 905 μ A 提高到 1 866.2 μ A;数值为 367 μ A 场发射电 流的电流波动不超过 4.5%.该一体式冷阴极场发射显示器已经以稳定的发光亮度而连续运行 10 余天.

关键词:冷阴极;银底电极;烧结;丝网印刷;场致发射 中图分类号:TB383;O462 文献标识码:A

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Fabrication of Integral Type Cold Cathode with Carbon Nanotube for Improving the Electrical Contact and Adhesion Performance

LI Yu-kui¹, WANG Feng-ge¹, LIU Xing-hui², LU Wen-ke³, ZENG Fan-guang⁴

(1 School of Electronic Information, Zhongyuan Institute of Technology, Zhengzhou 450007, China)
 (2 School of Physics, Liaoning University, Shenyang 110036, China)

(3 School of Information Science and Technology, Donghua University, Shanghai 201620, China)

(4 Department of Mathematics and Physics, Zhengzhou Institute of Aeronautical Industry Management,

Zhengzhou 450015, China)

Abstract: On cathode glass faceplate the new integral type cold cathode was developed. The printed silver slurry was sintered to form the silver bottom electrode. The thin-layer bottom electrode paste including a great deal of carbon nanotube was prepared, and was screen-printed on the silver bottom electrode surface. The ordinary carbon nanotube paste was fabricated on the

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First author: LI Yu-kui(1973 -), male, associate professor, Ph. D. degree, mainly focuses on display technology, electron device fabrication, vacuum technique, nano-materials preparation and applications. Email:lyksound@sina.com

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baked thin-layer bottom electrode paste. The two types of pastes were simultaneous sintered in the sintering furnace with the protection of high purity argon atmosphere. The sintered thin-layer bottom electrode would combine with the silver bottom electrode, which the carbon nanotube layer was covered on its surface. In one cathode pixel there were two carbon nanotube field emitters. Due to the spare carbon nanotube field emitter, the service life of field emission display could be prolonged greatly. Using thin-layer bottom electrode as interface layer between the silver bottom electrode and the carbon nanotube layer, the adhesion property of carbon nanotube was improved and the reliable ohmic contact had also been confirmed. With carbon nanotube as cathode material, the integral type cold cathode field emission display was fabricated, which exhibited good luminescence image quality and better field emission characteristics. Comparing with the ordinary carbon nanotube cathode field emission display, the turn-on electric-field could be reduced from 2. 11 V/ μ m to 1. 68 V/ μ m, the maximum field emission current could be enhanced from 905. 7 μ A to 1 866. 2 μ A, and the field emission display has subsequently been operated for more than 10 days with stable luminance brightness.

Key words: Cold cathode; Silver bottom electrode; Sintering; Screen-printing; Field emission

0 Introduction

Carbon nanotube (CNT), due to its unique physical properties of high aspect ratios and superior mechanical strength, was considered as an excellent field emission material^[1-3]. In triode field emission display (FED), the CNTs were prepared on the bottom electrode surface to form the field emitters, which were used to supply lots of environment. electrons in vacuum So the fabrication of CNT field emitters was all the way the key technology of triode FED. In cathode fabrication process, the high effective screenprinting technique had been extensively adopted, which was a low cost process for manufacturing the large-area field emitters[4-6]. However, the screen-printed CNT was easy to fall off from the bottom electrode surface due to the poor adhesion property, which the result was the FED could not be operated normally owing to the ineffective CNT field emitters. The formed electric field was reduced usually because of the poor electrical contact between the CNT and the bottom electrode, which would increase the FED driving circuit cost on account of high operation voltage. The above-mentioned drawbacks had obstructed the development of triode FED with CNT field emitters^[7-10]. For the bottom electrode fabricated with silicon wafer, the good electrical contact had been confirmed and the adhesion property could be improved for CNT field emitters, but the manufacture cost was too high and the large-area cathode fabrication was impracticable. That the ZnO material was added in the CNT paste was an effective way to enhance the adhesion performance

of CNT field emitters, but the electron-emission ability was not satisfactory and the electrical contact problem could not solved. For the bottom electrode formed with electric dielectric layer, although the field emission uniformity was enhanced due to the good electrical contact, the adhesion performance of CNT field emitter was poor, and the complex fabrication process was also its disadvantage. For solving the technical difficult problems, the research on fabrication of CNT cold cathode had been in progress^[11-16]. In this article, the novel integral type cold cathode was first developed with screen-printing technique, and the detailed fabrication process was also given. On the silver bottom electrode, the screen-printed thinlayer bottom electrode paste and the baked ordinary CNT paste were simultaneous sintered, so the integral type cold cathode was formed, which the adhesion property of CNT was improved and the electrical contact performance was also enhanced. The integral type cold cathode FED was fabricated, which possessed better field emission characteristics and good luminescence image quality.

1 Experiments

1.1 Structure of integral type cold cathode

The soda-lime flat-panel glass which the thickness was 2.8 mm was used as the cathode glass faceplate, and the integral type cold cathode was fabricated on the cathode glass faceplate surface. A structure schematic diagram of integral type cold cathode was illustrated in Fig. 1. The CNT field emitter was used to supply lots of electrons. The different cathode pixels would be separated by the cathode insulation layer. In one cathode pixel, there were two CNT field emitters, which were independent of each other. The applied cathode potential could be conducted to the CNT field emitter with the corresponding silver bottom electrode and thin-layer bottom electrode.



Fig. 1 Structure schematic diagram of integral type cold cathode on cathode glass faceplate

1.2 Fabrication of integral type cold cathode

Firstly, the original silver slurry was screenprinted on the cathode glass faceplate, which the printing thickness was about 75 μ m. After the baking and sintering process in proper sequence the silver bottom electrodes were fabricated. For the baking process, the baking temperature was 255 °C and the baking time was about 35 minutes. For the the maximum sintering process, sintering temperature was 535° C and the holding time in the maximum sintering temperature was 8 minutes. The whole sintering time was about 65 minutes. The bar silver bottom electrodes were arranged in order on cathode glass faceplate surface, in which the electrical insulation should be performed among the different bar silver bottom electrodes. In one cathode pixel, the width of one silver bottom electrode was 300 µm, and the distance between the adjacent silver bottom electrodes was 400 μ m.

Secondly, the purified double-walled CNT was used in our experiments, which the diameter was greater than 20 nm and the length was approximate $10 \sim 20 \ \mu m$. At the macroscopic level, the CNT was black powder. The original silver slurry, the CNT powder and small amount other organic solvent were mixed to form the mixture slurry, in which the CNT content was about 7.5%(in weight). The mixture slurry was heated to 65°C, and was blended with terpineol solvent. After sufficient stirring process the thin-layer bottom electrode paste was obtained. The thinlayer bottom electrode paste was printed on the sintered silver bottom electrode, which the printing thickness was approximate 20 μ m. The baking process was conducted for the printed thinlayer bottom electrode paste in automatic electric oven. The constant baking temperature was 285°C and the baking time was about 20 minutes.

Thirdly, the ordinary CNT paste was

prepared, which mainly consisted of the CNT, the ethyl cellulose and the terpineol. The ordinary CNT paste was printed on the baked thin-layer bottom electrode paste surface. The similar baking process was also carried out in automatic electric oven, in which the baking temperature was changed to 220°C and the total baking time was about 25 minutes.

Finally, the baked thin-layer bottom electrode paste and the baked ordinary CNT paste on cathode glass faceplate were sintered simultaneously in the special CNT sintering furnace. The maximum sintering temperature was 535°C, which was identical with that of silver bottom electrode. The holding time of 10 minutes was adopted at the maximum sintering temperature of 535°C in order to sinter fully the fabricated thinlayer bottom electrode paste and the ordinary CNT paste. The total sintering time was approximate 70 minutes. For avoiding the CNTs were oxidized in the high temperature environment, the protective gas of high purity argon (with 99. 99% purity) was used in the sintering course. After the cathode glass faceplate was cooled to room temperature, it could be seen that the thin-layer bottom electrode had combined with the silver bottom electrode, and the black CNT layer was covered on its surface. A proper post-treatment process was performed for the prepared CNT field emitters for improving their field emission capacity. So the fabrication of integral type cold cathode was completed.

1. 3 Encapsulation of integral type cold cathode FED

A structure schematic diagram of FED was showed in Fig. 2. The phosphor paste was screenprinted on the anode electrodes to form the luminous layer on anode glass faceplate. The grid fabricated on cathode glass faceplate was used to control the electron-emitting of CNT field emitters. The cathode and anode glass faceplates were separated by the glass frame, and combined to make a vacuum room, in which the getters would reside on the cathode glass faceplate. The gas in the vacuum room would be exhausted by a vacuum system with pressure on the order of 10^{-4} Pa. After the activation process of getters, the triode integral type cold cathode FED was fabricated. For comparison, the ordinary CNT paste was screen-printed on the silver bottom electrode surface to form the electron-emitting source. With similar manufacture process, the ordinary CNT cathode FED was also fabricated.



Fig. 2 Structure schematic diagram of integral type cold cathode FED

2 Results and discussion

2.1 SEM photo of surface morphology for CNT

The SEM photos of surface morphology for CNT in integral type cold cathode were presented in Fig. 3. Thereinto, Fig. 3(a) showed the photo of baked CNT. Seen from the photo, the sticky CNT paste had been dried due to the proper baking process. Because the baking temperature of 220°C was not enough high, a great deal of organic solvent still existed. In addition, a lot of CNTs adhered well on the baked thin-layer bottom electrode paste surface, which would demonstrate that the CNT had been transplanted successfully with the screen-printing technique. The SEM photo of CNT after post-treatment process was given in Fig. 3(b). Seen from the photo, some results could be achieved.





1) Due to the high temperature of 535°C, the organic solvent on the CNT layer surface had disappeared. The organic solvent, whether in the ordinary CNT paste or in the thin-layer bottom electrode paste, would obstruct the normal electron emission of CNT, and must be removed. The high temperature sintering process was an effective method, in which the CNT could be preserved and other organic solvent would be sintered completely. Besides, the proper posttreatment process was conducted for the CNT field emitters in the integral type cold cathode. So in Fig. 3(b), the residual organic solvent on the CNT layer surface would not be observed.

2) Owing to the protection action of argon atmosphere, the CNT field emitter would not be

damaged, although they had experienced the high temperature sintering course of 535°C. A large amount of CNTs would still reside on the thinlayer bottom electrode surface. Furthermore, much CNT ends were exposed on the post-treated CNT layer. Obviously, it was beneficial to further increase the field emission current.

3) In the integral type cold cathode, due to the high CNT content in the thin-layer bottom electrode paste, much CNTs would exist on the sintered thin-layer bottom electrode surface. On the one hand, many CNT had twined each other, and part of CNTs would be buried in the sintered thin-layer bottom electrode. That is to say, these CNTs would be fixed firmly on the surface of thinlayer bottom electrode. So the adhesion property of CNT could be effective enhanced. On the other hand, in the fabrication course the ordinary CNT paste was prepared on the baked thin-layer bottom electrode paste. So in the CNT layer, one part of CNTs would derive from the printed ordinary CNT paste; and another part of CNTs should come from the thin-layer bottom electrode paste. As shown in Fig. 3(b), these CNTs were mixed each other, and could not be distinguished. But, due to the printing of ordinary CNT paste, the number of CNT would be remarkable increased.

2.2 Real photograph of integral type cold cathode FED prototype

The real photograph of integral type cold cathode FED prototype was showed in Fig. 4. Seen from the photograph, several results were as follows.



Fig. 4 Real photograph of integral type cold cathode FED prototype

1) The integral type cold cathode FED prototype was rectangular shape, and the outer dimension was 200 mm(length) \times 50 mm(width). On anode glass faceplate, the one anode pixel area was 0.6 \times 0.6 mm². Of course, the size of all the anode pixels was identical. There were 273 anode pixels with green phosphor in total on anode glass faceplate, which were arranged in the form of 7 (low) \times 39(column) pixel matrix.

2) The whole FED had been vacuum-sealed by the sintered glass frit. For the low-melting glass frit, the maximum sintering temperature was 453 °C in the nitrogen atmosphere, and the total sintering time was approximate 55 minutes. Near the edge of anode glass faceplate (seen in Fig. 4) the glass frit had been reacted completely with the soda-lime glass. The fabrication material of glass frame was the same with the one of cathode and anode glass faceplate, so the cracking phenomenon of FED prototype in the sintering course would be avoided. The good air-tightness could ensure that the triode FED would run for a long time.

3) The gas in the vacuum room was evacuated out through the exhaust tube (seen the left side of FED prototype photograph), which the exhaust tube had been protected with a section of plastic pipe. The prototype cathode and grid parts were fabricated on the cathode glass faceplate, and the prototype anode part was formed on the anode glass faceplate, which all were packaged in the vacuum room of FED. Due to the covering of anode insulation layer, only the anode pixels were visible.

2.3 Field emission characteristics

The measured field emission curves were illustrated in Fig. 5. The two curves were included, in which the curve A_1 revealed the field emission curve for the integral type cold cathode FED and the curve A_2 demonstrated the one for ordinary CNT cathode FED. In the measuring course, the CNT cathode was grounded (0 V), and the grid fabricated above the CNT field emitter was biased with a positive voltage to extract the electrons. The anode voltage was fixed with 1.73 kV. Seen from the curves, some results could be gained.



Fig. 5 Measured field emission curve for (A_1) the integral type cold cathode FED and (A_2) the ordinary CNT cathode FED

1) The two types of triode FED possessed

typical field emission characteristic, whether for the integral type cold cathode FED or for the ordinary CNT cathode FED. As the enhancement of electric field, the field emission current was first increased slowly and then would be raised drastically. The result showed that both the silver bottom electrode in integral type cold cathode FED and the one in ordinary CNT cathode FED could conduct the cathode potential smoothly to the CNT field emitters, and a stronger electric field would also be formed easily. It would also mean that the integral type cold cathode was eligible electronemitting source in FED.

2) The turn-on electric-field for the integral type cold cathode FED was approximate 1.68 V/ μ m, which was obvious less than the one for ordinary CNT cathode FED (was about 2.11 V/ μ m). For the same field emission current of 398 μA , the required electric field for integral type cold cathode FED was 2. 29 V/ μ m, while the one for ordinary CNT cathode FED was about 2. 67 V/ μ m. Nevertheless, with the same electric field of 2.71 V/ μ m, the field emission current of integral type cold cathode was 1 086. 3 μ A, which was higher than the one of ordinary CNT cathode FED (was 426.5 μ A). These results would indicate that the silver bottom electrode (including the thinlayer bottom electrode) in integral type cold cathode FED possessed better electrical conduction performance and the CNT field emitters in integral type cold cathode FED had superior electron emission capability.

3) The maximum field emission current of integral type cold cathode FED was 1 866. 2 μ A, and the one of ordinary CNT cathode FED only was 905. 7 μ A. During the field emission current enhancing phase, the increasing range of field emission current was 877. 4 μ A when the electric field was raised from 2. 32 V/ μ m to 2. 83 V/ μ m for the integral type cold cathode FED. However, the one of field emission current only was 310 μ A with the same electric field increment for the ordinary CNT cathode FED. The above results would imply that the integral type cold cathode FED obviously possessed better field emission characteristic.

2.4 Luminance brightness curves

The luminance brightness curves were given in Fig. 6. Thereinto, the curve X_1 showed the functional relation of luminance brightness vs. electric field for the integral type cold cathode FED, while the curve X_2 demonstrated the one for the ordinary CNT cathode FED. It was obvious found that the integral type cold cathode FED had higher luminance brightness, in which the maximum luminance brightness had reached 2 576 cd/m². With the same electric field of 2.77 V/ μ m, the luminance brightness of integral type cold cathode FED (curve X_1) was 1 269 cd/m², while the one of ordinary CNT cathode FED (curve X_2) only was 573 cd/m^2 . These results would indicate that the fabricated integral type cold cathode could effective improve the electron emission performance of CNT field emitters due to the better electrical contact and adhesion property.



Fig. 6 Luminance brightness curve for (X_1) the integral type cold cathode FED and (X_2) the ordinary CNT cathode FED

In the integral type cold cathode, both the CNT and the sintered silver bottom electrode possessed good electrical conductivity. So the cathode potential could be conducted smoothly to the CNT field emitters by the silver bottom electrode and the thin-layer bottom electrode.

Firstly, the thin-layer bottom electrode paste was used. Due to the small amount of organic solvent in the thin-layer bottom electrode paste, the harmful effect on CNT field emitter would be reduced. The baking temperature (was 285(C) was proper increased, which the organic solvent in the thin-layer bottom electrode paste would be more thorough removed.

Secondly, in the thin-layer bottom electrode paste, a large amount of CNTs were included, which was helpful to form a better electrical contact between the CNT and the silver bottom electrode. After the thin-layer bottom electrode paste was sintered, the thin-layer bottom electrode would combine with the fabricated silver bottom electrode entirely. In other words, the sintered thin-layer bottom electrode including a great deal of CNTs had been successful inserted between the silver bottom electrode and the CNT field emitter. Using the thin-layer bottom electrode as interface layer, not only the good electrical conduction performance of integral type cold cathode would not be affected at all because both the sintered thin-layer bottom electrode and the CNT could conduct electricity well, but also the electrical contact between the CNT and the silver bottom electrode would be effective improved due to the included a large amount of CNTs in the thin-layer bottom electrode.

Thirdly, the thin-layer bottom electrode paste and the baked ordinary CNT paste were simultaneous sintered. In the sintering course, the silver particles and the CNT would permeate each other, which the adhesion property of CNT field emitter could be obvious improved and the electrical contact between the CNT field emitter and the silver bottom electrode would be modified greatly.

Fourthly, in one cathode pixel the two CNT field emitters were fabricated. Behind one anode pixel there was one cathode pixel in FED with the "matrix addressing" mode. In the FED operation course, only one CNT field emitter would supply electrons, the corresponding anode pixel could generate visible light. So the other CNT field emitter in the same cathode pixel was a spare electron-emission source. In other words, if one CNT field emitter became invalid, the FED could also run normally because lots of electrons would be emitted from the other CNT field emitter, which could prolong the service life greatly of whole FED prototype. Based on the abovementioned improving measure, it was confirmed that the field emission performance of CNT field emitter would be enhanced significantly.

2.5 Emission current stability

The emission current stability testing curves were plotted in Fig. 7. Thereinto, the curve Y_1 showed the stability functional relationship of integral type cold cathode FED, while the curve Y_2 demonstrated the one of ordinary CNT cathode FED. For two kinds of triode FED the same electric field of 2. 26 V/ μ m was applied. The emission current for integral type cold cathode FED was 367 μ A, and that for ordinary CNT cathode FED was 172 μ A. Under the condition that the applied electric field remained was constant, the two kinds of FED were operating simultaneously for a long time, and the emission current fluctuation would be monitored. At the initial stage, the emission currents for two kinds of FED prototype all increased firstly and then

decreased. However, after the field emission testing of 150 minutes, the current fluctuation for integral type cold cathode FED was less than 4.5%. For the ordinary CNT cathode FED, the constant emission current was maintained about 65 minutes and then would begin to decrease. The decreased range had exceeded 12.8%. This result would indicate that the integral type cold cathode FED prototype possessed better emission current stability. In addition, it was worth noting that the integral type cold cathode FED has subsequently been operated for more than 10 days with stable luminance brightness.



Fig. 7 Emission current stability curve for (Y_1) the integral type cold cathode FED and (Y_2) the ordinary CNT cathode FED

2.6 Luminescence image

The emission images of triode FED were exhibited in Fig. 8. Thereinto, the Fig. 8(a) was the dot matrix image of ordinary CNT cathode FED, and the Fig. 8(b) was that of integral type cold cathode FED. Seen from the presented images, some results could be obtained.



Fig. 8 The emission image of (a) the ordinary CNT cathode FED and (b) the integral type cold cathode FED

1) For the integral type cold cathode FED, all the anode pixels could be normal lightened, and the luminescence image brightness of different anode pixels was very uniform. On the one hand, with the same electric field, the luminescence image brightness for all the anode pixels almost

was identical. On the other hand, as the increasing of formed electric field, the luminescence image brightness for all the anode pixels would also be enhanced simultaneously. It was surprised that the higher luminescence image brightness and better luminescence image brightness uniformity had been observed. For the ordinary CNT cathode FED, although most of the anode pixels could be lightened, the luminescence image brightness of different anode pixels was obvious uneven. Some of anode pixels were very bright, and the other parts of anode pixels were relative dark. Even few anode pixels could not generate the visible light. The result implied that the integral type cold cathode would play a significant role for improving the luminescence image quality of FED.

2) With the applied operating voltage, the integral type cold cathode FED could run properly, and the luminescence image could also be displayed. It was demonstrated that not only the fabrication of integral type cold cathode for triode FED was feasible, but also the manufacture process developed here possessed considerable potential for practical applications. Moreover, the fabrication process was simple and the total manufacture cost was also low.

3 Conclusions

On cathode glass faceplate, a new integral type cold cathode was proposed firstly with high temperature process. The silver bottom electrodes were fabricated with the sintered silver slurry. The thin-layer bottom electrode paste was prepared, in which much CNTs were included. The thin-layer bottom electrode paste was printed on the silver bottom electrode surface, and the ordinary CNT paste would be fabricated on the baked thin-layer bottom electrode paste. The two types of pastes were sintered simultaneous in the special CNT sintering furnace. After the sintering process, the thin-layer bottom electrode had combined with the silver bottom electrode, and the black CNT layer was covered on its surface. In one cathode pixel two CNT field emitters were fabricated. Using the thin-layer bottom electrode as interface layer, the electrical contact between the CNT and the silver bottom electrode would be effective improved and the adhesion performance of CNT would also be remarkable enhanced. The integral type cold cathode FED had been fabricated firstly, which possessed better field emission characteristics and good luminescence image quality. Comparing with

the ordinary CNT cathode FED, the turn-on electric-field could be reduced from 2.11 V/ μ m to 1.68 V/ μ m; the maximum field emission current could be enhanced from 905.7 μ A to 1 866.2 μ A. The fabrication process was simple and the manufacture cost was also low. The fabrication process possessed considerable potential for practical applications.

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