SURFACE TREATMENT ON SILICON FIELD-EMISSION CATHODES

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ABSTRACT

Arrays and single-tip p-type silicon micro-emitters have been formed using a subtractive tip fabrication technique. Following fabrication, several different surface treatments have been attempted for comparison. We utilized ion and electron bombardment at elevated pressures (with interrupted pumping), and also hydrogen seasoning during field emission operation. The objectives of these treatments include stabilization of the emission, lowering the effective workfunction, and reducing low-frequency noise. The tips were evaluated using I-V measurements in the diode configuration. A flat Si anode, spaced nominally 6 μ m and 150 μ m from the cathode, was used. For the purpose of treatment, the field emission characteristics are measured in a high vacuum chamber at a pressure range between 10⁻⁵ and 10⁻⁸ Torr. The results suggest that the emitters benefit from seasoning or conditioning, for optimal performance, low noise, minimum work function and maximum reproducibility and reliability over the lifetime of the cathode.

INTRODUCTION

Silicon field emitters have demonstrated their viability as electron sources for various vacuum microelectronics applications. In recent years, the attention is drawn towards post-fabrication seasoning processes aiming to improve the performance of the field emitters. In the current work, we attempt various methods of surface treatment of single-crystal silicon emitter arrays fabricated by the well-developed subtractive manufacturing process [1]. The arrays of 50x50 tip emitters were fabricated from p-type (4-6 Ω cm) Si (100) substrate by the above process. Firstly, thermal grown oxide of 2000 Å thick and a 1000 Å thick chromium layer on the Si were patterned into a 1.8 µm-diameter disk. Using the chromium and the SiO₂ disk as a mask, the outline of the emitter tip was formed by means of ion reactive etching with SF₆ as shown in figure 1 (a). The tips were then sharpened using the method of oxidation sharpening as we have previously described elsewhere [2]. Tip caps were subsequently removed by wet etching of the silicon dioxide. The final silicon emission tip is shown in Fig.1 (b). The typical tip curvature radius is estimated using microscopy, to be on the order of 15 nm.

We investigate three methods of seasoning during the operation of the field emitters – conditioning of the emission surface using low-energy electron-stimulated desorption, surface treatment by residual gas ions, and surface cleaning using hydrogen-enhanced residual gas atmosphere.





Figure 1. a) SEM picture showing silicon tip before oxidation sharpening. Oxide cap is on top of the tip. b) After oxidation sharpening

RESULTS AND DISCUSSION

We tested the electrical and emission properties of the cathodes in a diode configuration as shown in figure 2. A flat Si anode, spaced nominally 150 μ m or 6 μ m from the cathode by using a quartz spacer of a proper thickness, was used. Field emission properties of the cathodes were measured in a vacuum chamber under a residual gas pressure of 10⁻⁸ Torr. Characterization was performed without bakeout of the vacuum system. The Hewlett-Packard 4142B modular DC source/monitor was used to acquire the emission data. A positive potential (up to 100 V) was applied to anode, and the cathode had a negative bias. The field emission properties of the cathodes were measured after the tips were conditioned for 3 days.



Fig. 2. Test structure. The Cathode is mounted on to a TO₃ Transistor package



Fig. 3. I-V characteristics (a) and the *F-N* characteristics (b) before (lower curve) and after (upper curve) the surface conditioning by low-energy electron stimulated desorption under low current loading.

First method of conditioning consists of cleaning of the surface by low-energy electron stimulated desorption under low-current loading. The first treatment method was performed in a slightly different configuration compared to one shown in Fig.2: instead of the flat silicon anode we have used the same tip structure as the cathode to be treated. The reverse bias voltage was in the region from 16 to 22 V, which allowed us to obtain the emission from the anode tip array to the cathode tip array. We have observed the initial I/V characteristics and the short-term current behavior of the "fresh" silicon cathodes (Fig. 3a, lower curve), then we reversed the polarity of the electric field by applying negative voltage to a silicon anode and using the silicon field emitter as an electron collector without changing the geometry of the cathode-anode "sandwich". In this configuration we ran the diode for a period up to 16 hours. The current density during the treatment was maintained on the level of 10^{-4} A/cm². The electron energy was in the range between 16 to 22 eV. The Coulomb dosage applied to the cathode surface was estimated to be on the level of $6*10^{-2}$ C/cm². No changes in the emission from the anode were observed during the entire treatment. After the treatment, an increase of the emission current by a factor of 5 (Fig. 3a, upper curve), along with stabilization of the I/V characteristics were observed.



Fig. 4. The effect of hydrogen treatment on silicon field-emission array cathodes under (a) high (1500 V) and (b) low (22V) anode voltage.

We believe that the electron bombardment using low-energy electrons helps to clean the surface of the emissive tips.

The second method consists of the surface cleaning using hydrogen-enhanced residual gas atmosphere during the operation of the cathode [3]. The getter ST-172 was heated up to 700°C to produce the hydrogen enriched atmosphere [4]. The treatment using a getter as a hydrogen source was suggested as a simple and cost effective method to use in the laboratory vacuum system. This method also can be easily used in industrial manufacturing of sealed vacuum devices equipped with modern getters. The experiment was performed for high voltage (1500V, 150 µm gap) and low voltage (22V, 6 µm gap) diodes as shown in figure 4a. and 4b. In the figures the solid line indicate the change in hydrogen pressure from 4×10^{-8} to 10^{-5} Torr, the curve indicate the average emission current over time and the dots indicate the actual emission current. It is seen in figure 4a that for high voltage diode there is a considerable increase in the emission current for about a factor of 5 after approximately 1 hr of treatment in hydrogen atmosphere. The same effect is observed for the low voltage diode during the time range of 20 min to 30 min. After the surface was cleaned using hydrogen-enhanced residual gas atmosphere, the tips were tested for a day at a pressure of 4×10^{-8} Torr. It was observed that the emission current remained approximately at the value immediately following the surface treatment. We suggest the following mechanism for the FEA performance improvement: that the electrons emitted from the cathode help to break the hydrogen molecules and/or ionize the hydrogen atoms from the hydrogen enhanced atmosphere. These ions and atoms react with the surface contamination like oxide, carbon residue etc. of the tips and clean the surface lowering the work function

The third method, accelerated "natural" surface conditioning was performed by increasing the pressure of residual gases. This was done by interrupting the pumping in the vacuum chamber during prolonged operation of silicon emitters from several hours to several days. The I-V characteristic seen in figure 5a corresponds to the two curves measured before (lower curve) and after (upper curve) the experiment. It is seen from the two graphs that there is a considerable increase in the emission current.



Fig 5. I-V characteristic and the F-N characteristic before (lower curve) and after (upper curve) the "natural" surface conditioning was performed by increasing the pressure of residual gases.

We believe that the short-term exposure to the enhanced residual gases atmosphere helps to remove an initial film of contaminants and adsorbents by ion sputtering. The two main disadvantages of this method are the following: first it requires a prolong treatment to achieve a desirable value for the emission properties, and second this method is not very advantageous to achieve the highest emission properties as obtained in the above two processes.

It should be noted that all the above methods resulted in comparable improvement in reproducibility of I-V characteristics taken after the treatment.

CONCLUSION

Three methods of surface treatment were applied to silicon field emitter arrays fabricated by subtractive process. All three methods – low energy electron-stimulated desorption, hydrogen seasoning, and residual gas ion conditioning, were performed in a simple small-volume stainless vacuum system. The methods clearly show improvement of emission characteristics, increase of emission current and stabilization of cathode performance. The hydrogen treatment seems to be suitable for improving the properties of the gated field-emission cathodes.

ACKNOWLEDGEMENTS

The Authors acknowledge the helpful input of Dr. Si-Ty Lam, and his colleagues, of Hewlett Packard Labs. This work was funded through the DARPA-MTO MEMS program under contract agreement F30602-98-3-0232, administered by the US Air Force Material Command.

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