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Field Emission from As-Grown and Ion-Beam-Sharpened Diamond Particles Deposited on Silicon Tips

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Abstract. Ion-beam bombardment/milling was used for sharpening of diamond particles deposited on ends of silicon tips. Radii curvature of diamond coating down to about 20 nm have been formed in such a way. Field emission experiments with sharpened diamond coated emitters have shown that the ion beam treatment effects a considerable shift of current-voltage characteristics of in the lower voltage region.

1. INTRODUCTION

The discovery of possibility to realize the negative electron affinity (NEA) for the (111) - face of diamond [1] stimulated strongly research in field emission of diamond and diamond-like materials [2-4]. The obtaining of relatively large field-emission currents from tipped diamond surfaces in comparison with flat ones [5] was interpreted as a sequence of the NEA phenomenon, although a mechanism of such an emission remains unclear.

From practical point of view, the use of the tip emitters prepared from single-crystalline diamond [5] seems to be unrealistic due to extremely high cost of the material. Another approach to solving of the problem based on using of diamond particles or films deposited onto high-aspect-ratio silicon tips has been proposed [6]. In the approach, hot-filament CVD process is preferable for the deposition of diamond, because such a process enables to prepare high-quality (poly-crystalline) material [7,8] with reproducible electrophysical properties. However, radii of curvature of the diamond coated emitters are too large for obtaining of sufficient currents at moderate electric fields.

In this paper, a process of ion milling is used in order to prepare sharp, reproducible diamond emitters.

2. EXPERIMENTAL

The diamond-coated silicon-tip emitters were prepared according to the procedures described in papers [7,9]. The emitters were formed on the butt-ends of [111]-oriented silicon rods and, then, all of them, except one, were removed so that characteristics of single emitters could be measured.

Then, the samples were treated by beams of single-charged argon ions at energies 30 keV, ion currents 0.2 to 0.3 mA/cm², and fluences $2 - 5 \cdot 10^{18}$ ion/cm². The ions were directed along axes of the emitters. The procedure was performed at 60°C. In more detail, the sharpening procedure was described in papers [10,11].

After the sharpening, some samples were annealed in atomic and/or molecular hydrogen at 600-700°C during time periods 15 to 30 min.

Field-emission characteristics of the samples before and after the treatments were taken according to the technique described in [12]. Both I-V and FN characteristics were plotted.

3. RESULTS AND DISCUSSIONS

3.1. Ion-milling

The ion beam had a gaussian density distribution. In the center of the beam where ion densities were maximal, at sufficiently-large ion fluences diamond coating was sometimes etched off completely so that silicon tip itself became exposed, while at a periphery of the beam a partial diamond sharpening occurred. Practically, with our post-mounted tips, ion fluences were chosen so that to reach an optimal sharpening and avoid the exposure of silicon.

An example of the ultrasharp diamond tip prepared by the technique is given in Fig. 1.

Figure 1: Diamond-coated silicon-tip emitter: a) - as-grown (before sharpening); b) - sharpened one.

3.2. Field emission from sharpened diamond tips

In Fig. 2 are shown emission characteristics obtained from the same sample before and after the sharpening. As it is seen, the voltage corresponding to the onset of the emission, at measurable (in our experiments) 10^{-9} A, decreased strongly (from about 1300 V down to about 150 V) when the emitter was sharpened. This decrease we connect with at least two factors:

- (a) decrease of the curvature radius of the emitter from about 0.5 mm to about 20 nm;
- (b) tentative formation of defects in the emitter during the ion-beam bombardment with corresponding improvement of the field emission, as it was supposed by Zhu et al [13].

The latter effect is confirmed by our results with annealings of the emitters. In Fig. 3, the curve 1 corresponds to a sharpened emitter before the heat treatment, while the curve 2 - after its annealing (650°C, 15 min) in atomic hydrogen (the treatment was held inside an equipment for hot-filament CVD

process, at the temperature of the filament 2000°C). The curve 3 was obtained after an annealing of the same emitter in molecular hydrogen at 650 - 700°C, 15 min.

Figure 2: I-V characteristics and FN plots of the same diamond emitter before and after sharpening.

From comparison of the curves it is seen that after the annealings the currents decreased strongly, for several orders of magnitude.

Figure 3: Changes of I-V characteristics of a sharpened diamond emitter as a result of annealing of the emitter in atomic hydrogen and, then, in molecular hydrogen

One of the explanation of the effects is that the annealings remove defects formed at the sharpening and, in such a way, deteriorate emission properties of the diamond. In particular, the action of atomic hydrogen can be more effective because, as is known, it is able to dissolve effectively atomic carbon. Such a carbon can be formed preferentially at the surface. Another possibility is that annealing may change the silicon/diamond interface structure as well as diamond surface. Additional experiments are needed to investigate the ion milling/annealing effects on crystal structure and surface/interface properties of diamond coating.

The more weak action of molecular hydrogen is shown in Fig. 4. Here, the procedure of annealing in atomic hydrogen was excluded, and the annealing effect was markedly decreased.

In general, the annealing effects need in more detailed (including microstructural) investigations.

Figure 4: A change of I-V characteristics of a sharpened diamond emitter as a result of annealing in molecular hydrogen

4. CONCLUSIONS

1. Ion-beam bombardment/milling was effectively used for ultra-sharpening of diamond particles deposited on ends of silicon tips. Radii curvature down to about 20 nm can be formed in such a way.
2. Ion beam treatment of diamond coated field emitters effects a considerable shift of current-voltage characteristics of in the lower voltage region.

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