## <sup>13.2</sup> Iridium field emitter as a source of direct current of electrons and ions

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Emission properties and shape transformation of the iridium field emitter under the impact of strong electric field and temperature were studied by field emission microscopy. Creation of an emitter with a stable surface and sufficiently large number of emitting nanoprotrusions for obtaining a direct ion current is impossible in the case of Ir, the current always appears to be pulsed. However, obtaining direct current of Ir ions is none-the-less possible, but only by using separate nanoscale protrusions growing on the  $\{012\}$  and  $\{023\}$  corners of the emitter built up in the emitter electric field. Ion currents are low in this case, but each protrusion is an almost ideal point source of ions.

Keywords: iridium, nanoscale protrusions, field ion emission.

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Point field emitters of charged particles are widely used as sources of both electrons and ions [1]. However, the most efficient way of employing such emitters is using them after exposure to strong electric fields F and high temperatures T (thermal-field treatment). Iridium is a material quite interesting for many purposes; however, almost no detailed investigation of its thermal-field shape changes has been performed. Concerning Ir, there are known only studies devoted to determining the self-diffusion activation energy [2] and using Ir-emitters as substrates for depositing graphene layers with subsequent field desorption of Ba atoms [3]. The only publication devoted to thermalfield impact on Ir is [4]; however, this paper describes only the "ring collapse effect" discovered there, namely, growth and evaporation of crystalline outgrowths on various emitter faces.

The main goal of this work was to study the main types of changes in an Ir-based field emitter shape and clarify what are the characters of ion currents able to be generated by such emitters at different T and F. The studies were performed by using field electron microscopy. Here the main factors are the emitter temperature T and impinging electric field F. The temperature was measured with a conventional micropyrometer, while the field strength was determined by the known Fowler–Nordheim method [5] under the assumption that the mean Ir work function is  $\varphi = 5.0 \text{ eV}$  [6].

Nowadays, there are known the following types of thermal-field shape changes of field emitters (the emitters almost always appear to be single-crystals because of a small (below a micrometer) size of the tip apex, i. e. emitter) [7]. First, the so-called field-induced build up of the emitter is observed at the lowest T and F; thereat, the initially smoothed emitter transforms to a polyhedron. Second, nanoscale (as small as a few nanometers) sharp protrusions arise on the corners (where three edges meet) and edges of the built-up emitter. And, finally, at the highest T and

F macro-outgrowths comparable in size with the original emitter grow primarily on the close-packed faces. All these processes are characterized as field-induced crystal growth. Some stages of this growth are demonstrated in Fig. 1 which presents field-emission images of the Iremitter with the most common facet orientation  $\{100\}$ to the observer and device screen at different F and T. Fig. 1, *a* presents the initial image of the pure Ir surface. The first noticeable changes in the emitter surface are observed even at the lowest tip temperature T = 900 K only under impinging field F = 3.2 V/nm; thereat, the emitter initially smoothed by heating gets rebuilt into a polyhedron with formation of inter-facet edges and corners around the  $\{100\}$  and  $\{110\}$  facets in the  $\{023\}$  regions (Fig. 1, b). Increasing the treatment temperature to 1500 K induces some variations in the process. The brightly emitting corners of the emitter built-up in the field shift from the  $\{023\}$ regions to the  $\{012\}$  ones; formation of such corners occurs even at F = 1-2 V/nm. Then, at F = 3-4 V/nm, nanoscale protrusions also grow on the same corners (Fig. 1, c). And, though the emission images of the corners in Fig. 1, band nanoprotrusions in Fig. 1, c are quite similar, the nanoprotrusions are much sharper, and the  $U_{10}$  values in these cases are absolutely different:  $U_{10} = 7300 \text{ V}$  for the corners in Fig. 1, b and  $U_{10} = 3870$  V for nanoprotrusions in Fig. 1, c ( $U_{10}$  is the applied voltage necessary for obtaining one and the same electron emission current I = 10 nA). At higher F = 4.5 - 5.2 V/nm, nanoprotrusions grow already over almost the entire emitter surface (Fig. 1, d).

Fig. 2 demonstrates only two cases of thermal-field shape changes of the iridium {111} facet because the publication volume is limited and does not allow more images to be presented. This orientation is less common, but the process of deformation proceeds exactly in the same way. Fig. 2, *a* shows the initial {111}-oriented Ir with  $U_{10} = 6440$  V; Fig. 2, *b* illustrates formation of six emitting nanoprotrusions located at the corners in the {012} crystallographic regions



**Figure 1.** Field-emission images of the Ir {100}-oriented surface. a — original Ir surface; b — after exposure to T = 900 K and F = 3.2 V/nm, four corners of the built-up tip in the {023} regions; c — after exposure to T = 1500 K and F = 3.2 V/nm, four nanoprotrusions in the {012} regions, d — after exposure to T = 1500 K and F = 5.0 V/nm, nanoprotrusions over the entire Ir surface.

at  $U_{10} = 2561$  V, i.e. the emitter becomes almost 3 times "sharper".

Thus, the Ir shape changes under the thermal-field impact consist primarily in the extension of close-packed facets {111} and {100} with formation of sharp trihedral corners in the {023} regions at T = 900-1400 K; when T = 1500-1900 K, the corners shift to the {012} region. Large outgrowths, namely, macro-outgrowths, also grow first of all on close-packed facets {111} and {100} at T = 1300-1500 K, while at higher T(1700-1900 K) these outgrowths grow also on loose emitter facets, such as {110} and {012}, and their growth is caused only by the presence of nanoscale protrusions playing the role of growth initiators. Hence, it is possible to obtain on the surface of such an emitter four or six point sources of electrons and ions in the form of corners or nanoscale protrusions in the {012} or {023} regions. Obtaining a single emitting protrusion is

also possible. For this purpose, it is necessary either to use the method of stepwise reduction in applied voltage U, and, therefore, in F, at constant temperature T [8] (which does not guarantee a success), or to use in fabricating the point emitter wires of appropriate orientation ({012} and {023}).

The character of the temperature dependence of F necessary for changing the surface shape is typical for metals: the higher is T, the lower is F at which a certain shape change takes place. For instance, if formation of nanoscale protrusions at T = 900 K needs F = 4.8 V/nm (at F = 3.2 V/nm, the field-induced emitter build-up just begins), then at T = 1900 K F is already equal to 2.8 V/nm. The use of T above 1900 K is impractical, since at such T rapid emitter blunting takes place.

Since iridium is not oxidizable in air and, what is more, is diffusion-resistant, it is interesting first of all as a material for nanotechnology; this means that the Ir-based emitter is



**Figure 2.** Field-emission images of the Ir {111}-oriented surface. a — original Ir surface; b — after exposure to T = 1500 K and F = 4.7 V/nm, six nanoprotrusions in the {012} regions.

interesting mainly as a point source of ion current. Evaporation at cryogenic temperatures is not applicable in this case because the emitter gets blunt quickly, and obtaining a continuous ion flux becomes impossible. Here is applicable only high-temperature field evaporation capable of creating a continuous ion flux due to constant diffusion influx of atoms to the emitter apex with subsequent evaporation of the atoms in the form of ions. Compared to other refractory metals (W, Ta and Re), iridium evaporates relatively easily, which is mainly due to its lower evaporation heat. In the case of using as an ion source an emitter covered with a great number of nanoprotrusions, the ion current is maximal: in the case of iridium it can reach  $I \sim 10-15 \text{ nA}$ ; this value is quite high and fairly sufficient for the purposes of nanotechnology. However, similarly to the cases of W and Ta, the current has the character of individual bursts with a variable duty ratio. In contrast to the case of Ta [9], here it is impossible to form a surface with a sufficiently large number of nanoprotrusions that would produce an almost constant ion current. However, in this case, a stable and even almost constant current source can be an Ir surface with four corners in the vicinity of the  $\{012\}$  facets or, better yet, with nanoprotrusions grown on them. This is a very stable form able to produce ion currents  $T \sim 10^{-12}$  A, which are even somewhat higher than for Ta. Here the number of sources is lower (only four nanoprotrusions), but the current may be somewhat higher because of an easier evaporation of Ir. In addition, in this case the current is produced by four or six point sources, since there are only a few atoms at the apexes of nanoprotrusions which have grown on the corners. If an  $\{012\}$ -oriented emitter is used, it is possible to create a source with a single nanoprotrusion in the center of the tip surface. The current from this single protrusion will, of course, be small  $(\sim 10^{-13} \,\mathrm{A})$ , but the emission angle will be minimal (about 0.05-0.07 sr).

Thus, the main result of this work is that appropriate thermal-field treatment of the iridium emitter makes it possible to obtain a stationary Ir-ion current source producing an almost constant ion current. This source has the form of either four-six corners of a tip built-up in the field or of four-six nanoprotrusions grown on those corners. Notice that this source will be of an almost point character. The current it produces is, of course, quite low; however, the source is characterized by a very high local brightness and very small emission angle.

## **Conflict of interests**

The author declares that he has no conflict of interests.

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