THE FORMATION OF In AND Sn SMALL-SIZED NARROW STRIPS BY MEANS OF LIQUID METAL ION SOURCE TECHNIQUE

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In this paper the processes of applying the nanodroplet phase from a sharp emitter to a nearby moving surface by means of a finely dispersed phase of a liquid metal ion source (LMIS) using In and Sn as metal source and both tungsten and copper plates as a substrate are considered. To apply narrow strips, the emitting needle was located at a close distance from the moving surface. At a needle-surface distance of the order of 80 μm, massive continuous tracks a few microns wide were obtained on the axis of a wide and thin trace of ions (In^+, Sn^+). The structure of the deposited strips with a length of more than 10 mm is granular. As the needle approaches the surface further, the track melts due to the high density of the ion current, and the inhomogeneous profile of its cross section is smoothed out. For the deposition of narrower structures, efficient cooling of the conductive movable substrate is required.

Keywords: liquid metal ion source, LMIS, field emission, nanoparticle, ion beam.
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1. INTRODUCTION

For the creation of various surface structures, the method of depositinq of nanodroplets on a conductinq surface is very promisinq. For this, liquid metal ion sources with an emitter tip are used. Point emitters have the highest current density, a very small emission zone and the ability to generate charged drops of the working substance with nanometer dimenzions. The possibility of focusing the resulting beams to submicron sizes is used in microtechnologies, and the generation of nanoparticles of various compositions is of great interest for the production of thin films and nanotechnologies. In liquid metal ion sources, along with the field emission of ions, under certain conditions, the generation of charged drops occurs [1, 3]. If the ion beam divergence angle reaches 90º, then the droplet flow divergence is (3–4)º [2]. The size of the ion emission zone is about 5 nm, which determines the high initial density of the ion current and the small size of the generated droplets.

The histogram of the size of the deposited particles is a sharply decreasing exponential function in the range of (2-40) nm [2]. The number of small particles exceeds the number of the largest ones by 3 orders of magnitude. Individual droplets with a size of about 100 nm are observed. It should be added that the generation of nanoparticles occurs in a threshold manner and is accompanied by oscillations of the ion current with a frequency of tens of MHz. The reason for the oscillations is the excitation of capillary instability on the surface of a liquid emitter [5].

The recorded particle sizes determine the possibility of the formation of small-sized structures on the surface through the dispersed phase of the source, which was the subject of this study.

2. EXPERIMENTAL

In order to receive a beam of ions from liquid-metal ion sources, the needle must be wetted with a working substance. For this purpose, we have prepared a container-type structure from graphite. The needle and the workinq substance are placed in a closed volume inside the container, which minimizes the evaporation of the active substance. The container is made as small as possible to reduce the energy consumption required when heating the working substance to the melting temperature. Needles with tip sizes of several micrometers can be made from various materials (W, Ni, Fe, etc.) by chemical etching or mechanical methods. Thus container-type liquid metal source was used, by means of which could be obtain beams of In, Sn, Au, Ni, Ge, B ions.

Material for a refractory needle was selected for each working substance in order to reliably wet its surface. A graphite container with a needle and a working substance was heated from the back by electron bombardment to the melting temperature of the working substance. To achieve the emission of ions, an extractor was used, located at a distance of 1 mm from the needle with a voltage of several kilovolts. At an ion current of about 40 μA (In, Sn), its oscillations occur with a frequency of tens of MHz, simultaneously charged nanoparticles are generated [5,7]. If necessary, the composition of the beam was determined using a mass analyzer with crossed electromagnetic fields such as a Wines velocity filter [6]. Taking into account the low divergence of the nanoparticle beam, narrow trajectories can be obtained with horizontal displacement of the substrate located at a close distance from the needle. That is, complex ion optics is not used in this case. As a conductive substrate, polished plates of copper, tungsten, molybdenum, and silicon were used. For precise movement of the substrate in three

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coordinates, a piezoelectric table of the PZU 2300 brand was used, controlled by a computer, which allowed vertical movement of up to 300 μm with an accuracy of 1 nm and horizontal movement of up to 100 mm (Fig. 1, b). The speed of horizontal movement varied within 0.5–2 mm/s. The piezotable was placed in a vacuum chamber and showed reliable operation at a residual pressure of $p \lesssim 10^{-5}$ Torr. The system was mounted on the basis of the A-700 Q Leybold-Heraeus vacuum unit with turbomolecular pumping. The deposited strips were analyzed using optical, scanning electron and atomic force microscopes.

3. RESULTS AND DISCUSSION

It is known that at low ion currents the radiation of liquid metal sources is stable. At a certain threshold current (about 40 μA) high-frequency oscillations are excited in the beam, which are accompanied by the generation of nanodroplets of size (2–20) nm and with specific charge $\frac{q}{m} = 5 \cdot 10^4 \frac{C}{kg}$ (In, Sn) [6]. The oscillations of the beam current are caused by the development of capillary instability on the surface of the Taylor cone which consists of a system of standing waves.

The ion energy spectra were determined using a velocity filter (Wien filter) with crossed static electromagnetic fields. Ions are emitted from a small spot on the top of the Taylor cone. The divergence angle of the ion beam reaches 90°. At a sufficient value of the electric field intensity the separation of nanodroplets occurs from the upper part of the cone. In the ion current stream, the divergence angle is approximately (2-3)°.

It was established in experiments that, due to excitation instability in the emitter, the ion current density in the center of the beam is slightly lower compared to its periphery. The ion energy spectra, which were repeatedly reproduced while maintaining the experimental conditions, were recorded in the center of the beam (Fig.1, a) and outside its axis (Fig. 1, b). The latter were obtained by moving the mass analyzer along the beam axis. The spectra were obtained in two modes: in the absence of nanodroplet generation (curve 1, $I_b = 30\ \mu A,\ U_b = 6\ kV$) and during their generation (curve 2, $I_b = 50 \mu A,\ U_b = 6{,}2 kV$). Ions with an energy of 6 keV correspond to an electric field intensity in the analyzer of about 140.5 V/cm. It should be noted that the input slit of the analyzer was achieved with the calculated value of the potential change between the plates.

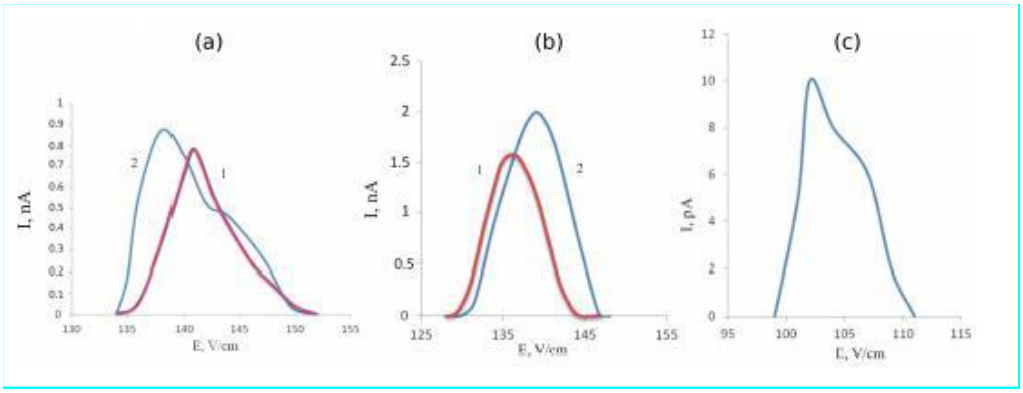


Fig. 1. Ion current through the filter of velocities as function an electric field intensity for In_1^+ (curve 1: I_b = 30 μA, U = 6 kV; curve 2: I_b = 50 μA, U = 6,2 kV) (a) in the beam center, and (b) outside the beam axis. Ion current for In_2^+ I_b = 50 μA, U = 6,2 kV, in the beam center (c)

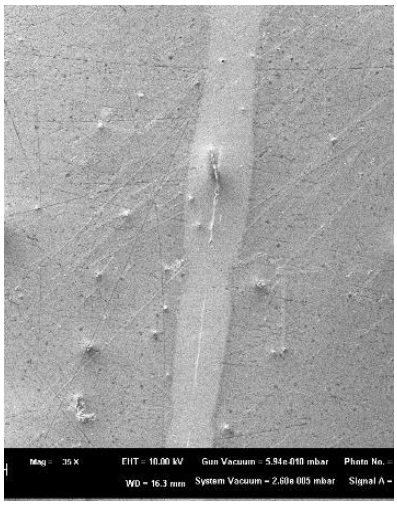


Fig.2 SEM - image of the indium band on tungsten plate surface.

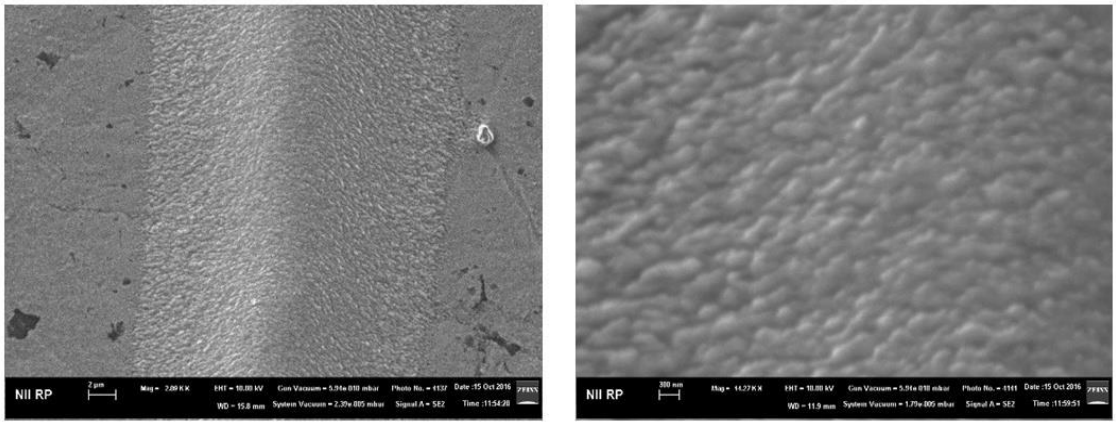
The position of the analyzer did not change further when registering nanodroplets. The energy spectrum of a diatomic ion In_2^+ was also recorded in the nanodroplet generation mode (Fig. 1, c), when generating nanodroplets the maximum of the spectrum in the center of the beam is shifted toward lower energies (250 eV), but off-axis it is shifted toward higher energies.The latter shows that in order to increase the beam current, the extractor voltage must be increased.

In the usual extraction of ions by means of an extractor (needle-extractor distance (0.5 - 1 mm)), the emission threshold voltage is (5 ÷ 6) kV. When the extractor was replaced with a flat substrate and the needle approached, the emission voltage decreased markedly.

Fig. 2 shows a strip of indium ions and nanoparticles deposited on a tungsten plate. The extraction voltage was 4.5 kV, the needle-plate distance was ~ 200 μm.

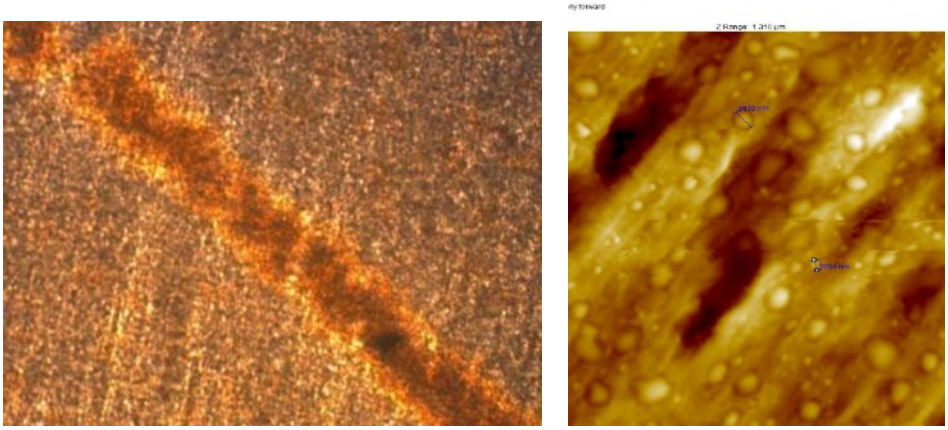
The strip has a width of about 200 μm, and it is determined by the diameter of the ion beam. A narrow path is clearly visible in the middle of the strip due to the deposition of nanodroplets. The width of this path is about 20 μm (Fig. 3, a), the central region is significantly elevated compared to neighboring regions, and it has a coarse-grained structure (Fig. 3, b). The characteristic diameter of elongated grains is (50-60) nm, and the length is (100-200) nm. These sizes significantly exceed the sizes of generated nanodroplets [8,9]. Apparently, due to the high density of the ion current, the substrate heats up significantly, the deposited particles do not have time to condense, which leads to their coagulation and the formation of large grains. The calculation shows that at a distance of 10 cm from the needle, the ion current density is about 10 A/cm^2, and the beam power is $3 \cdot 10^4$ W/cm^2. In order for the nanodroplets to condense without adhering to each other, effective dissipation of energy from the substrate is necessary.

Further approaching the needle to surface leads to smoothing of the transverse profile of the applied strip: it becomes more uniform (Fig. 4, a). It is likely that the deposited substance is now heated almost to its melting point, and the axial massive path does not condense. The structure of the strip is no longer granular, but in it the dispersed phase is evenly distributed (Fig. 4, b). Large particles with a size of several microns are easily distinguishable, but, apparently, the number of small particles significantly exceeds the number of large ones.



a) b)

Fig. 3. a) SEM image of a trace of deposited nanoparticles, b) trace structure.



a) b)

Fig. 4. a) AFM image of the indium band on the surface of the molybdenum plate. The needle-plate distance is 50 μm, U = 3.5 kV b) Dispersed strip structure.

4. CONCLUSION

When a finely dispersed phase of the liquid metal source is deposited on a nearby uncooled surface, long In and Sn strips several micrometers wide are formed. The strip structure is due to submicron sizes particles. There are no complex elements of ion-optical devices in the system. Proper cooling of the substrate allows the formation of narrower surface structures of practical interest. If the tracks are deposited on an efficiently cooled thin metal film, then, if necessary, it can be removed by etching. This method can be used in microelectronics to perform various technological operations.

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