MAIN ELEMENTS AND CHARACTERISTICS OF ELECTROHYDRODYNAMIC ION SOURCES BASED ON InSb

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For the first time, the method of preparation of the main constructive elements of the ion source used for obtaining ion beams with high current density based on InSb and $InSb_{0.98}Bi_{0.02}$ was developed and the volt-ampere characteristic of its ion current was measured. The material of the needle, which is the main element of the source, was selected according to the working substance (InSb and $InSb_{0.98}Bi_{0.02}$), the needle was sharpened by mechanical and chemical methods, and then wetted in the melt of the working substance at $550°C$ in a vacuum. The sharpened and wetted needles are placed in a closed graphite container, which is another element of the ion source, with the working substance inside. During the process, we ensured that the tip of the needle extended into the ion emission zone. Batches of InSb and $InSb_{0.98}Bi_{0.02}$ ions with different mass and charge were obtained in the prepared ion source.

INTRODUCTION

Electrohydrodynamic (EGD) or liquid metal ion sources based on alloys (LMAIS) allow for the generation of atomic and molecular (cluster) ion currents of various elements and their chemical compounds with densities of up to hundreds of millions $\sim10^8 A/sm^2$ and low energy spread. The main characteristics of EGD sources and their differences from a wide range of gas, plasma, and other types of ion sources are analyzed in detail in [1,2]. In the field of micro and nanotechnology, electrohydrodynamic ion sources are indispensable for sub-micrometer lithography, ion-stimulated chemical synthesis, implantation, and microanalysis [3]. Today, it is impossible to conceive of the formation, analysis, and processing of a desired surface relief with sub-micrometer resolution without a focused ion beam [4,5]. Initially, pure metals with low melting temperatures, such as gallium, indium, etc., were used as the working substances for EGD emitters [6]. However, the solution to many modern technological challenges, such as localized ion implantation, ion beam mixing, direct non-resistive ion lithography, and the fabrication of micro and nanoelectromechanical systems, is impossible without the utilization of ion beams with varying masses, charge states, and chemical properties [7]. This has stimulated the development of liquid metal ion sources based on alloys, enabling the generation of a broad spectrum of different ions [8]. The utilization of eutectic alloys with different compositions has allowed for a more predictable and controllable alteration of the electrical, optical, magnetic, and mechanical properties of the processed materials. LMAIS and subsequently LMAIS based on alloys are also extensively employed in secondary ion mass spectrometry and secondary neutral particle analysis [9], as well as in space technology and ion microscopy [10].

In recent times, surface processing with ion beams is almost exclusively conducted using electrohydrodynamic ion sources. The fact that the emission zone of liquid metal ion sources is very small creates favorable opportunities for the application of these sources in technologies aimed at producing various surface structures and thin films. Considering that the types of ions used significantly influence the physicochemical properties of the obtained microscale structures, including electrical, optical, magnetic, and mechanical properties, it can be asserted that a large potential application area has emerged, which can be regulated through appropriate selection. As a result of research conducted in this field over the past half-century, almost half of the chemical elements from the periodic table have been found to be potentially suitable or already utilized in modern LMAIS [2, 4].

The presented article elucidates the peculiarities of manufacturing elements for electrohydrodynamic ion sources using InSb as the working substance. It provides the results of scientific research on certain specific characteristics of ion beams generated from these sources.

EXPERIMENT

One of the main components of liquid metal ion sources is a sharp-tipped needle. The needle is wetted with the working substance, and upon subsequent heating, the working substance melts and flows along the surface of the needle towards its pointed end. Depending on the working substance, the material of the needle should be chosen to be refractory, wettable by the working substance, resistant to dissolution in it, and chemically inert towards it. It should also maintain the shape of its tip for a sufficient duration without undergoing erosion. Moreover, the melting temperature of the needle material should be higher than that of the working substance. The voltage at which ion emission occurs and the longevity of the needle directly depend on its sharpness. Needles can be fabricated through both mechanical and electrochemical means. However, since mechanically sharpened needles require a relatively high threshold voltage for ion emission, electrochemical sharpening is preferable.
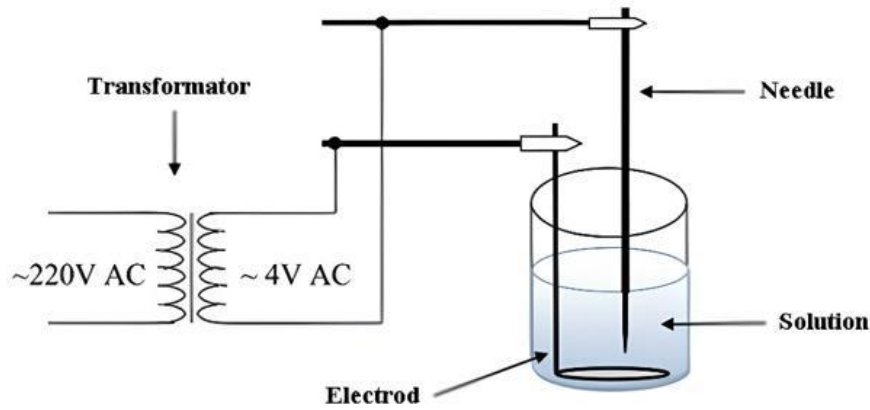
Fig. 1. Process of chemical preparation of the needle.

Figure 1 illustrates the process of chemical preparation of the needle. Prior to the procedure, the choice of chemical solution is determined based on the needle material. In our experiments, we used nickel-chromium (NiCr) needles and selected a 6% solution of HCl. The solution is poured into a specialized container containing an electrode. A constant voltage of approximately 4V is applied between the needle and the electrode. Subsequently, the needle is quickly immersed in the solution for several minutes and then removed. The tip of the needle is observed through a magnifying glass during the procedure. When using a constant voltage in the experiments, the sharp end of the needle becomes smooth, creating favorable conditions for the melted liquid to flow towards the tip of the needle.

In our experiments, tungsten or nichrome needles with a diameter of (0.5-0.8) mm were sharpened by mechanical or electrochemical means to a tip radius of several (30-50) μm. Diamond files and the finest sandpaper were used for mechanical wire processing. To ensure continuous delivery of the working substance to the needle tip and its further emission, the needle needs to be sharpened along its axis with the creation of longitudinal grooves. This facilitates the flow of the working substance along the grooves. The needle can also be sharpened by electrochemical means. In this case, for each needle material, the electrochemical treatment requires the selection of an appropriate electrolyte composition.

To wet the NiCr needle with the working substance, it is placed in a narrow graphite boat, and InSb is poured onto the sharp end. The boat is covered with a graphite lid and heated under vacuum conditions until the InSb melts and wets the needle with the working substance (see Figure 2). The experiment was conducted using a Leybold-Heraeus L-700-Q vacuum setup.
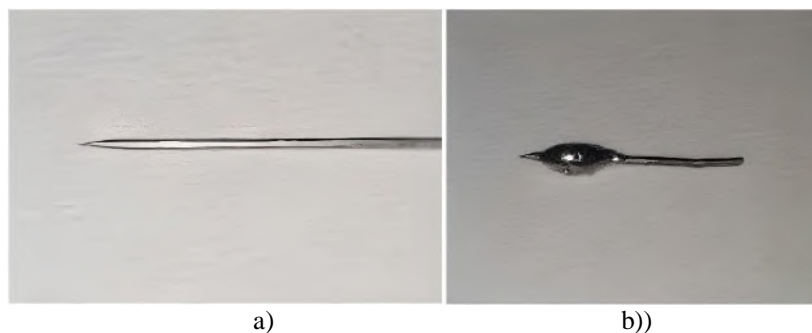

| a) | b)) |

Fig. 2. a) A NiCr needle sharpened in a chemical solution. b) A NiCr needle saturated with the working substance InSb.

For conducting research with a liquid metal ion source, a special container-type structure was designed and manufactured. This structure allowed the generation of ion beams of In, Sn, Au, Ge, B, as well as alloys such as InSb and $InSb_{0.98}Bi_{0.02}$. Inside the container, a needle made of a refractory material was placed, wetted with the working substance. The tip of the needle protruded from a small opening in the container, leading to the emission zone (see Figure 3). The material of the needle was selected for each working substance to ensure reliable wetting of its surface.

To achieve ion emission, the wetted needle is placed in a specially designed container. Containers are made from various materials with high melting points, such as titanium, molybdenum, graphite, etc. In our case, we used graphite as the container material. The choice of graphite as the container material is based on several properties of graphite. Graphite exhibits negative thermal expansion along its basal plane up to a temperature of 700 K. As a result, when the volume of the container housing the needle decreases, the electrical contact between the needle and the positive pole of the applied accelerating field is further enhanced.

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Fig. 3. A needle made of nickel-chromium wire, impregnated with InSb, is placed inside a graphite container.

This leads to the formation of a high-intensity electric field between the needle and the extractor. At high temperatures, graphite does not melt due to its physical properties and naturally does not mix with the working substance. Additionally, the technical processing of graphite does not pose significant difficulties compared to other refractory materials. As a result, the obtained ion beam is homogeneous, consisting solely of particles of the working substance.

In such a design, the working substance is contained in a closed volume, minimizing the evaporation of the melt, which occurs only from the surface of the needle tip and the opening in the container. The consumption of the working substance in the form of ions is very small, so its mass supply can be less than one gram, and accordingly, the container size needs to be small. This is also necessary in terms of power consumption required to heat the container to a high temperature for melting the working substance. To achieve a high temperature of the container (around 1000°C), it is necessary to limit the heat dissipation of the supplied energy. Therefore, a structure was created to secure the container using three thin tantalum spokes. Each spoke has a cross-sectional area of only 1

mm², but the system proved to be sufficiently rigid and resistant to thermal loads. Calculation based on the heat conduction equation was performed to ensure efficient heat transfer and maintain the desired temperature.

$$Q = \lambda \frac{\Delta T}{l} S$$

(where λ is the average thermal conductivity of tantalum, S is the cross-sectional area, l is the length of the heat sink) shows that in order to heat the container to the desired temperature in this design, an electron bombardment power of about 30 W is required. The container has a diameter of 6 mm and a height of 10 mm. It is made of graphite, which is non-wetting to most liquids and has a low vapor pressure.

Local heating of the graphite container with the needle and the working substance is achieved by electron bombardment from the rear side using a tungsten filament heated to the melting temperature of the working substance. With a diameter of 0.5 mm for the tungsten filament, a filament current of approximately 20 amperes was necessary to heat it to the thermo-electron emission temperature. The container holder is attached to a copper anode cylinder, and both the anode cylinder and the cathode holders are cooled by flowing water.

A schematic representation of the liquid metal ion source is provided in Figure 4. A needle made of NiCr material with a tip radius of a few micrometers is placed inside a graphite container and heated to the melting temperature of the working material. The extractor is located at a distance of 1 mm from the needle. With a potential difference between the needle and the extractor of 5-6 kV, ion emission occurs, and a bright spot is observed near the sharp end of the needle. The ion beam is dispersed at an angle of approximately 90 degrees, and the beam current is registered in the collector circuit.
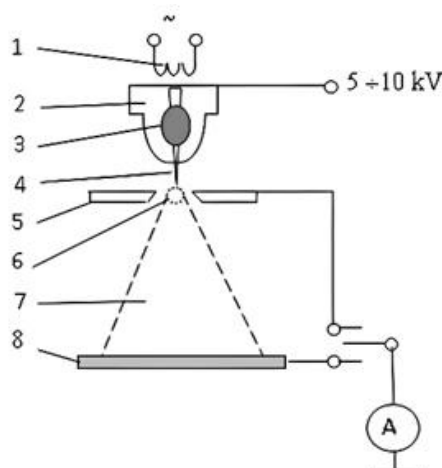

Fig. 4. Ion Source Diagram
1 - Cathode, 2 - Container, 3 - Working substance, 4 - Needle, 5 - Extractor,
6 - Bright area, 7 - Ion beam, 8 – Collector

The use of graphite as the material for the container is justified by its properties. Graphite has a negative thermal expansion in the direction of its basal plane up to a temperature of 700 K. As a result, when

the volume of the container housing the needle decreases, the electrical contact between the needle and the positive pole of the applied accelerating field is improved. This leads to the formation of a high-

intensity electric field between the needle and the extractor. At high temperatures, graphite does not melt and naturally does not mix with the working substance due to its physical properties. Furthermore, the technical processing of graphite does not pose significant challenges compared to other refractory materials. Therefore, the resulting ion beam is homogeneous and consists only of particles of the working substance. One of the key indicators of the performance of any liquid metal ion source (LMIS) is its volt-ampere characteristic (VAC), which provides the functional relationship between the emission current of ions (I) and the extracting voltage (U) applied to the extractor. Proper interpretation of the VAC allows us to accurately assess the suitability and reliability of the LMIS in all operating modes, both after the source's manufacturing and after its long-term use. This issue has always been given special attention in experimental and theoretical studies [4,11]. According to the widely accepted theoretical model by Mayer, the ion emission current for a threshold potential difference is determined by the formula

$$I = 3\pi \sqrt{\frac{2e}{m}} \frac{r\gamma \cos\alpha}{\sqrt{U_{min}}} \left(\frac{U}{U_{min}} - 1\right)$$

From the formula, it can be seen that the ion emission current is influenced by the potential difference U at the extractor, the atomic mass of the generated ions m, the surface tension of the working fluid γ, the radius of curvature of the emitter r, and the Taylor cone angle α. Ion emission begins when the threshold voltage is exceeded. The nature and slope of the VA characteristics of the needle-shaped LMIS depend on the distance between the source and the extractor, the aperture diameter in the extractor, and the emitter material. The influence of electrode geometry on the volt-ampere characteristics of LMIS has been extensively studied in [12]. On Figure 5, the volt-ampere characteristics are shown for the alloys InSb and $InSb_{0,98}Bi_{0,02}$.
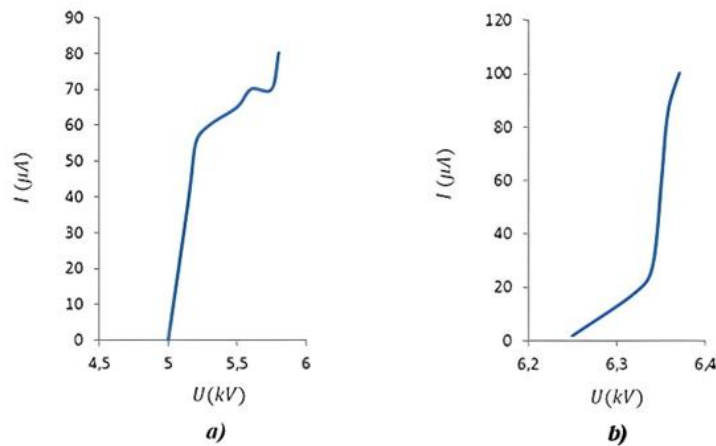

Fig. 5. a) Volt-ampere characteristic (VAC) of the ion source with the working substance InSb; b) VAC of the ion source with the working substance $InSb_{0,98}Bi_{0,02}$.

We used smooth needles, which results in a steeper slope of the VAC. Additional factors include the angle and radius of the needle tip, as well as the diameter of the needle base. As seen in the figure, for both samples, ion emission begins at a threshold voltage and increases with increasing extraction voltage. For the InSb0.98Bi0.02 alloy, the threshold voltage is higher than that of InSb, which is likely due to the higher melting temperature of Bi.

To form the Taylor cone, a higher voltage is required compared to maintaining it. Therefore, the optimal operating emission current is considered to be the one at which the extraction voltage is higher. At this voltage, the emission current is higher with larger tip radius, needle base diameter, and taper angle. The shape of the VAC is significantly influenced by the physicochemical properties of the alloys, such as density, surface tension, surface free energy, sublimation heat, and operating temperature.

CONCLUSION

For the first time, a liquid metal ion source based on these materials was developed to obtain ion beams of InSb and $InSb_{0.98}Bi_{0.02}$ with different masses, charge states, and chemical properties. In accordance with the physicochemical properties of these working substances, a nichrome wire (NiCr) with a diameter of 0.5 mm was used as the material for the needle. The needles were sharpened using electrochemical and mechanical methods to achieve a tip size of (50-70) μm, and they were wetted with the working substance under vacuum conditions at a pressure of $P = 2 \times 10^{-5} mbar$.

The prepared needles, along with additional working material, were placed in a sealed graphite container, which also serves as another primary element of the ion source. The entire experimental system was assembled on the basis of the A-700-Q Leybold-Heraeus vacuum setup. For each working substance, InSb and $InSb_{0.98}Bi_{0.02}$, functional dependencies of the current emission (I) on the extraction voltage (U) were obtained for the first time.

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