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# Evaporators with Induction Heating and Their Applications

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## 1. Introduction

Induction heating is widely used in different fields due to high process productivity and universality, cleanness and possibility of full automation. This process is friendly for environment. Since the induction allows reaching temperatures of 2000 °C and more, this method is very suitable for evaporation. This process is known to be a process of emitting atoms or molecules by hot matter surface. The atoms or molecules form "the vapor". Since vapor pressure depends on temperature, the latter is conditionally accepted as "the evaporation temperature" when pressure of saturated vapor reaches 1 Pa. Data on evaporation temperatures are collected in numerous handbooks, *e.g.* (Banshah, 1994).

The evaporation is a very important process for industrial production and scientific researches. Its main application is deposition of thin film layers and coatings on semiconductors, glass and polymers in electronics and optics as well as on other industrial products including food packaging films, metal strips, *etc.* The evaporation is mostly related with technology of Physical Vapor Deposition (shortly, PVD) that means matter deposition from vapor or atomic/molecular medium. In PVD, the vapor medium, as a source of depositing matter, is generated physically (not chemically!) due to thermal evaporation of some primary source materials; deposition, in turn, must happen as atom-by-atom process, at least as multi-atom cluster process but without any drops or even smallest droplets. There are various evaporation methods for PVD except induction: evaporation from boats and crucibles with resistive (Joule) or radiation heating, evaporation with electron and laser beam heating, cathode arc evaporation, *etc.* The induction technology competes with the mentioned methods and occupies own niche in industrial technology and for preparation of scientific objects.

The evaporation also is a very useful process for refining materials to obtain high purity, for producing micro- and nanoparticles and, thus, it is a base of so-called evaporation metallurgy.

The main features and fundamental possibilities of the evaporating induction technology are the followings:

- electromagnetic induction may provide not only heating evaporated materials but also vapor ionization and high density vapor plasma generation for Ion-Plasma Enhanced (Activated, Assisted) PVD processes, often called as the Ion Plating, accompanied by self-ion bombardment for obtaining high quality coatings;

- the inductor may be disposed aside of a heated object and inside or outside of a technological chamber since electromagnetic induction acts through non-conducting medium (vacuum, gas, liquid, solid) and dielectric envelopes; this allows, in particular, to evaporate chemically active, radioactive and toxic substances in closed envelopes, for example, for electron device or special material manufacturing;
- the inductor may operate at voltages and in conditions avoiding electrical discharges (arcing) around the inductor placed into gas medium under pressure from vacuum to atmospheric one; therefore, it is possible to evaporate not only in vacuum but in gas medium for synthesis of compound coatings and production of micro- and nanoparticle materials by "gas evaporation/condensation" method;
- on the other hand, generation of plasma by electromagnetic field can provide production of micro- and nanoparticle materials by "plasma assisted evaporation/condensation" method;
- usually induction evaporation occurs from crucible, therefore the maximal evaporation temperature is limited by the maximal operation temperature of crucible material;
- the direct contact between the inductor and evaporated material is absent, the operation temperature of the water-cooled inductor is less than 80 °C; hence, the method ensures introducing minimal impurities into coatings; however, the problem of reaction of crucible material with evaporated substance remains;
- electromagnetic field of the inductor-heater may provide mechanical confining of molten metal in a crucible or levitation effect on solid and molten metal and evaporation in the levitation mode, not from crucible, for obtaining very pure thin films and coatings.

Due to advantages and performances of evaporators with induction heating they are interesting for thin film and coating technology and special metallurgy.

Historically, we are not aware of the first researchers and engineers employing induction heating for evaporation but one may find patents, published as far back as at the 1930's, that dealt with induction evaporation. This method has been periodically used for commercial purposes (to produce coatings, thin films and various materials) since. Mainly as simple thermal evaporation for the 1940's-1960's, and then also as with vapor ionization.

The problem of induction evaporation and its applications has been discussed in literature from time to time but very shortly, see, for example, handbooks (Banshah, 1994; Bishop, 2007) and survey (Spalvins, 1980). The more special and full review papers (Anderson, 1975; Tada I. et al., 1989), devoted to this theme, were published many years ago. So, there is a lack of fresh information on contemporary solutions in this field and potential profit of the induction evaporation for industry and science. The authors of the present paper try to fill up this gap but they do not describe all known evaporators with induction heating. Only principle solutions and new those developed in Kiev Polytechnical Institute are presented in the paper.

In this chapter we consider base principles of induction evaporation design, then

- evaporation without vapor ionization, in particular, for microelectronic industry;
- evaporation with vapor ionization for Ion-Plasma Enhanced PVD (Ion Plating); herein, different approaches and electromagnetic structures, including additional electron thermoemitters and ionizing inductor with pulsed power supplying for effective vapor ionization will be presented;
- design of special induction evaporators, in particular, for deposition of coatings onto the internal surfaces of hollow substrates and for levitation evaporation.

## 2. Principles of induction evaporator design and operation

The base design of the typical induction evaporator (vapor source) is shown in Fig. 1. A crucible 1 charged by evaporated material 2 is concentrically surrounded by an inductor 3 that is a circular coil made from water-cooled copper pipe and powered by an alternative current (AC) generator 4. The arrows 5 depict the water flow through the coil tube. The crucible is needed as a container for the melted and evaporated material 2. A medium frequency (MF:  $f = 1-66$  kHz) or radio frequency (RF:  $f = 0.44-1.76$  MHz) current is ordinarily used for power supply of inductors in the evaporators. The inductor generates an alternative magnetic field that, in turn, induces an eddy current (also called as Foucault current) in the crucible body, if it is made from conducting material, or directly in the conducting evaporated material, if the crucible is from non-conducting material. The eddy current provides Joule heating of the conducting crucible or, accordingly, the conducting evaporated material up to the desired temperature. In the case of conductive crucible, the evaporated material is heated from the hot crucible due to heat transfer. The physical laws and main features of induction heating of the crucible and the charge are the same as at the other induction heating processes.

The crucible is disposed in a technological chamber for isolation from the atmosphere air. Vacuum is the ordinary operation environment in the chamber and the lower the pressure of the residual gas in the chamber after pumping out the better the conditions for stable work of the evaporator and for obtaining the high quality products without gas impurities are. The residual gas pressure is order of  $10^{-2}-10^{-3}$  Pa in big web coaters but the pressure of  $10^{-5}$  Pa and less is desired for very high vacuum processes. But in some cases, a working gas is introduced into the chamber.

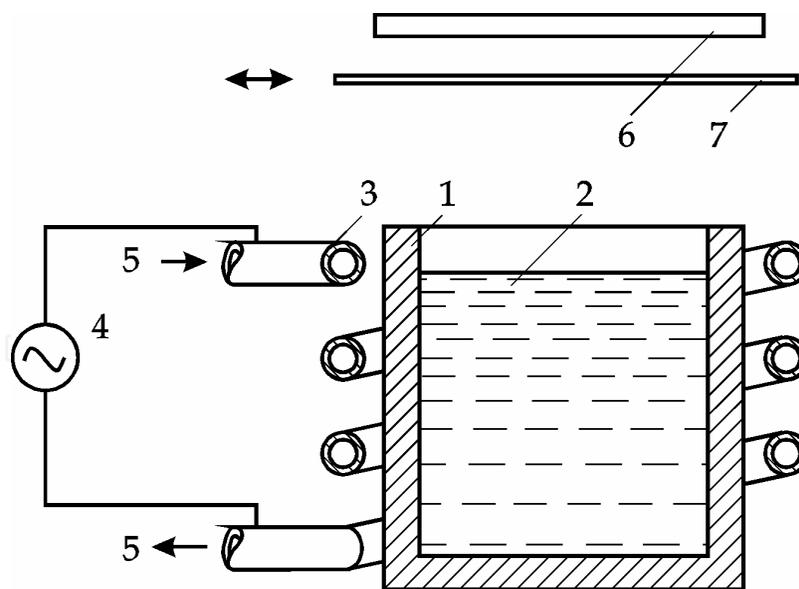


Fig. 1. Simplified diagram of typical induction evaporator. See position names in the text

In PVD system, a substrate 6 is disposed above the crucible to collect and condensate vapor species, and to provide formation of thin film or coating from the evaporated source material on the substrate. A movable shutter 7 is disposed at the front of the substrate 6 to open or close its surface from the vapor flow. There may be additional parts in induction evaporators, for example, heat shields between the crucible and the inductor, and electrical

or protective shield for the inductor. The crucibles for induction evaporation may be of different shape (cylindrical, conical, rectangular) and dimensions (from 1-2 cm in diameter to tens centimetres in length) and with different mass of charge (from grams to tens and hundreds kilograms). Fig. 2 presents photographs of practical design of some water-cooled evaporating inductors.



Fig. 2. Photographs of water-cooled evaporating inductors for use in electronics, two of them are shown with crucibles. The left inductor has the top turn with opposite winding as compared with the rest turns

The diagram in Fig. 3 depicts the structure of PVD process with induction evaporation and the aggregation (phase) state transformation of the primary source materials, charged into crucible, during the process: solid state  $\rightarrow$  liquid  $\rightarrow$  vapor  $\rightarrow$  solid state. Note that some metals (*e.g.* Cr) evaporate by sublimation without melting and passing into the liquid phase at the evaporation temperatures.

If evaporation is carried out in high vacuum, the evaporated species do not collide with residual gas molecules and go by straight-lines without scattering from the crucible to the substrate surface. As a rule, the angular distribution of vapor flow density is non-uniform that leads to a non-uniform coating thickness distribution across the immovable substrates. Very often, they are moved with help of various mechanisms relatively the crucible either to reach high uniformity of coating thickness, or to provide deposition on large substrate surfaces. Substrates are often heated (up to 200-600 °C) in order to improve coating properties.

In systems for vacuum distillation of the primary materials, cooled crystallizers are used instead the substrates. Work of the distillation systems is analogous to the PVD system operation (see Fig. 3).

Powder collectors are used instead of the substrates in systems for manufacturing of micro- and nano-particles (powders) by means of condensation of vapor species directly in the dense working gas introduced into the chambers. In this case the aggregation state

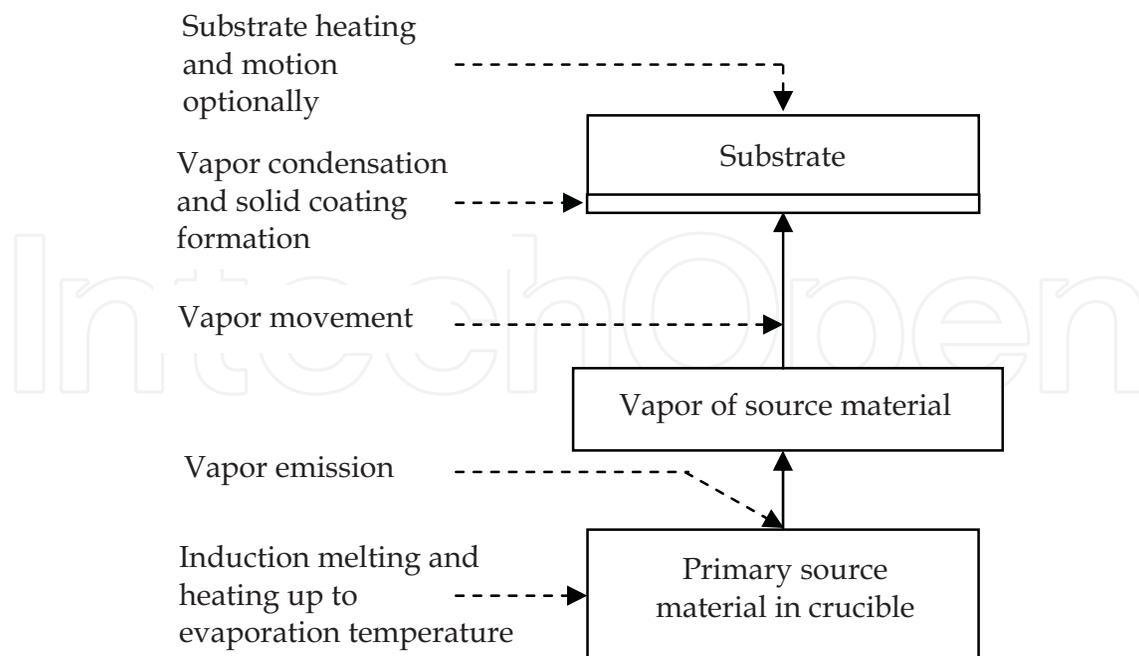


Fig. 3. Diagram of the aggregation state transformation of the primary source material during PVD process

transformation diagram, shown in Fig. 3, changes: vapor condensation occurs on the stage of vapor movement in the gas medium.

The important moment is choice of crucible material. Firstly, the latter must be refractory material. Secondly, it must be chemically inert relatively evaporated materials. Thirdly, the reason for the crucible failure is that some molten metals have a tendency to creep and migrate into pores of the crucible material. When the crucibles are cooled after finishing deposition process, there would be a differential contraction between the bulk crucible wall material and the metal-containing crucible wall surface. The wall would then be under stress and would tend to crack or to spall a part of its surface. One method used to extend the life of the crucibles for evaporating Al is the deposition of alumina into the pores in the crucibles, thus preventing the access of Al to the pores. Zr carbide is also used for coating the internal crucible surface for this purpose. The crucible material, often used in induction evaporators, are practically the same as for resistive-heated boat and crucible (Banshah, 1994) and those are, for example, graphite, pyrolytic carbon, pyrolytic BN, sintered composites  $TiB_2+BN$  and  $TiB_2+TiC+AN$  as well as different other ceramics. The best crucibles material has a dense structure and ensures hundreds hours of operation life or tens of deposition cycles.

The next question, what crucible is to be: conducting or non-conducting? When thickness of conducting crucible walls is greater than the skin-layer thickness, the crucible fully absorbs electromagnetic energy, electromagnetically shields the molten metal and prevents interaction of the inductor field with the metal. As the result, quantity and volume of the melted charge do not affect work of AC generator, and monitoring of the crucible temperature is sufficient for monitoring the molten metal temperature as they are correspond each other. Besides, in this case mechanical effects of the inductor field on the molten metal are absent that ensures the quiet splashless evaporation process without turbulence of the melt. On the contrary, when the crucible is non-conducting, the magnetic

field of the inductor directly interacts with the molten metal. The work of AC generators and transfer of electromagnetic energy into the evaporated material will depend on quantity and volume of the melted charge within the crucible. Also, the inductor field will interact with the eddy current in the molten metal and generate mechanical force acting on the melt. The force can compress the melt, and considerable turbulence with ejection of metal droplets towards a substrate may occur during induction evaporation. See the more detailed explanation of this effect in Section 6. The mechanical effect of the inductor field on the melt has its own advantages and disadvantages but it is often desired to avoid it during deposition of precise thin films for electronics and optics.

Many induction evaporators comprise both a non-conducting crucible and a specially introduced conducting element that serves as an absorber of electromagnetic power generated by the inductor. This element, called as "susceptor", heats the non-conducting crucible due to heat radiation and heat conductivity. When the susceptor surrounds the crucible, it also shields the crucible from the inductor field.

The crucible volume must allow charging a sufficient quantity of evaporating material to complete, at least, a single technological run without the need for replenishing the crucible. When the evaporator is to employ continuously or for long runs, wire feeders are used to add the new portions of evaporated material in the wire form. Sometimes it is possible to introduce an ingot into the crucible through a bottom port.

Consider the problem addressed to wettability of the hot crucible wall surface by the evaporating metal. Such active molten metals as Al, keeping at the high evaporation temperature, are able to wet the wall surface, flow in the form of liquid film upwards to the top due to the effect of surface tension and migrate through the edge to the outer crucible surface, and then evaporate onto the inductor coil. This leads to shorting the coil turns or to electrical breakdown and firing of some kind of parasitic electrical discharge. To avoid appearance of the evaporating metal films on the outer crucible surface, several approaches for crucibles from conducting materials may be proposed. The one is to provide overheating the upper part of the conducting crucible walls or the top edge due to stronger coupling with the inductor electromagnetic field. The overheating must cause enhancing metal atom evaporation from the mentioned crucible surface parts before the atoms can reach the outer crucible surface. The first way to increase the electromagnetic coupling is to provide the crucible with an outwardly directed lip or projection that will be closer to the inductor coil than the cylindrical crucible wall, and the induced current in the lip and, hence, its temperature will be higher. The second way to increase the electromagnetic coupling and to overheat the liquid metal film on the top edge is to make the thickness of upper part of the conducting crucible walls less than the skin-layer thickness at the operation frequency, while the lower part of the crucible, where the melt is, has the wall thickness larger than the skin-layer (Ames, 1966). On the top part, the induced current will heat not only the crucible body but also the metal film on the internal surface of the wall as RF field is able to penetrate through the relatively thin crucible wall. The lower part of the crucible wall is relatively thick and the wall will shield the melt from RF field to avoid turbulence of the liquid metal and undesirable possibility that droplets of the molten metal may be ejected towards the substrate being coated.

We propose to solve the problem of crucible wall wettability by attaching a shield element with low heat-conductivity on the top edge of the crucible (Kuzmichev & Tsybulsky, 2008). Fig. 4 shows the induction evaporator with such element on the crucible. The crucible 1, surrounded by the inductor 2, contains the molten evaporating material 3. Due to

wettability of the crucible wall surface and the high temperature ( $> 1000\text{ }^{\circ}\text{C}$ ), the molten metal wets the wall surface and migrates in the form of liquid film upwards to top edge 4, and then overflows to the outside of the crucible. Vapor back condensation also contributes to deposition of liquid metal on the top of the crucible. The crucible top edge outside is tightly bonded with the ring shield element 5 with low heat conductivity. The outer edge of the shield element 5 is disposed on the water-cooled element 6. The low heat conductivity of the shield element 5 prevents excessive cooling of the upper edge of the crucible 4 and formation of solid metal growths on it. At the same time, due to the lower temperature of the outer edge of the shield element 5 the liquid metal migration is excluded here. The heat-removing element 6 is located outside the lines of sight from the crucible 1, and its dimensions (radial length and cross-sectional area in azimuth) are chosen from the following condition for its thermal resistance  $R_t$  (1):

$$\frac{0,1T_m - T_{col}}{Q} \leq R_t < \frac{T_m - T_{col}}{Q}, \quad (1)$$

where  $T_m$  is the melting temperature of the evaporated metal 3,  $T_{col}$  is temperature of the cooling system 7,  $Q$  is the heat flow to the heat-removing element 6. When selecting the dimensions and material of the heat-removing element 6 are in accordance with condition (1), the temperature at the points of its contact with the shield element 5 does not exceed the melting temperature. This completely stops the flow of liquid metal towards the outside of the crucible below the shield 5, if it is tightly bonded with the crucible wall or forms with the crucible a one body.

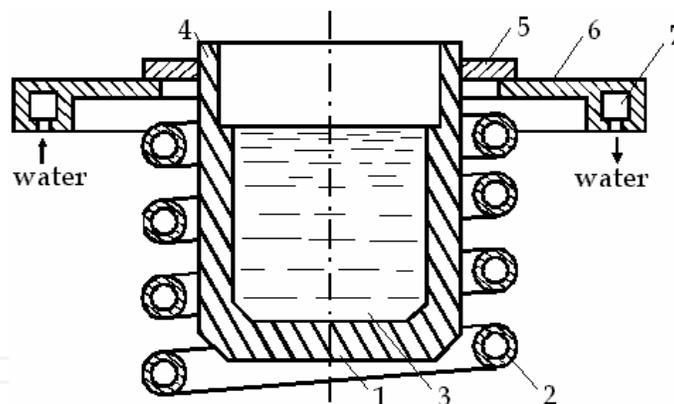


Fig. 4. Evaporator with the elimination of overflow of the melt to the outer crucible side (see position names in the text)

The exemplary crucible with the top-edge shield for evaporation active metals such as Al may be made from sintered conducting ceramics (*e.g.* of  $\text{TiB}_2 + \text{BN}$  composition that was proposed (Ames, 1966) earlier) or from pyrolytic BN. The former case with conducting crucible is illustrated by Fig. 4. The latter case is presented by photograph in Fig. 5, where the top-edge shield of the BN crucibles is, in fact, an outwardly directed funnel-shaped lip. The crucible is produced by plasma chemical deposition from the gas phase; herein, the crucible and its top-edge shield are made as one body. Since boron nitride is dielectric, an external hollow cylindrical susceptor is needed for coupling with RF field of the inductor. Then the susceptor will absorb RF power and heat Al charge within the crucible by heat-

transfer. The susceptor must be from conducting material, for example, from high-pure graphite. The susceptor plays the important role of RF shielding the molten Al and preventing the melt from mechanical interaction with the magnetic field of the inductor. By this reason the wall thickness of the conducting crucible and the susceptor must be larger than the skin-layer thickness at operation frequency.



Fig. 5. Pyrolytic BN crucibles with outwardly directed funnel-shaped lips to avoid migration of liquid metal towards the outer crucible surface

### 3. Induction evaporation without ionization for electronic component manufacturing

Metal deposition on semiconductor wafers and other substrates is the essential part of technological processes for manufacturing electronic components including very large integrated circuits, processors and memory for computers. Metal thin films and coatings fulfil different roles, in particular, they serve as electrodes and electrical wiring in integrated circuits. The commonly used vacuum evaporation methods for this purpose are evaporation from resistance-heated boats and crucibles, induction and electron-beam evaporation. Like the resistance-heated evaporation, the induction-heated one may be free from the ionizing radiation that is very important for many microelectronic structures, for example, metal-dielectric/oxide-semiconductor devices, which are very sensitive to the ionizing radiation. In this relation, the electron-beam evaporation is very dangerous because of accompanying by emission of X-rays from the crucible. By the way note, many plasma deposition processes (e.g. ion sputtering or chemical plasma deposition) also may be dangerous due to ultraviolet and high-energy particle generation. On the other hand, the induction evaporation is able to provide deposition rate that is much higher than at the resistance-heated evaporation and comparable with rate at the electron-beam evaporation. The vapor-emitting surface of induction-heated crucibles is larger than the resistance-heated surface; therefore the former generates more uniform and wide vapor flow than the latter. This allows simplifying the substrate fixture/holder design for obtaining uniform deposited layers. The cost of induction evaporators is higher than the cost of resistance-heated evaporators but comparable with the cost of the electron-beam evaporators. The common feature of the all evaporation methods is generation and employing of low-energy species for formation of

metal thin films. The energy of the species is thermal by nature and is order of 0.1 eV. For comparison, the average energy of species, forming thin films in ion sputtering processes, is about 5-10 eV. The low energy species are required to create delicate micro- and nanoelectronic structures. Thus, the induction-heated evaporation may compete with other evaporation methods in PVD technologies in electronics and close fields.

In order to ensure no ionizing radiation, one must prevent the induction evaporator from electrical breakdown, sparking, ignition and firing of any kind of electrical discharges as well as from electron and ion emission by surfaces of evaporator parts. These electrical processes may generate charge particles (electrons and ions) with energy up to 1 keV and higher, ultraviolet radiation and soft X-rays, so, the factors promoting electrical and radiation damage of the sensitive electronic structures. Obviously, the first condition for preventing from any discharges is providing the high vacuum in the deposition chamber that is the lowest residual gas pressure (less than  $10^{-4}$  Pa) and cleanliness of evaporator parts. However in the vacuum evaporating systems, other factors may cause the discharge processes, too.

Discuss from this point of view the simple induction system shown in Fig. 1. The system is quite able to deposit thin films in some operation regimes but does not guarantee no ionization processes by the following reasons: i) high inductor voltage (hundreds volts and even up to 1 kV), then high difference of potentials between the inductor and the crucible or other chamber parts and presence of the strong electromagnetic field promote ignition and firing of an electrical discharge in vapor flow going towards the substrate; ii) the migration and overflow of liquid metal to the outside of the crucible and then firing an electrical discharge in vapor medium between the lateral crucible surface and the inductor coil are possible; iii) high temperature heating of the crucible and, in particular, overheating of the crucible top edge may cause thermoelectron and thermion emission from the crucible surface; thermoelectrons and ions, in turn, are accelerated by the inductor voltage and may affect the structures on the substrate or ignite the vapor discharge. Proceeding from the said, one may conclude the evaporator with the shielded inductor area and stopped liquid metal migration to the outer crucible side, presented in Fig. 4, as well as the crucibles, presented in Fig. 5, are more suitable for thin film deposition without ionization effects than the shown in Fig. 1.

In order to illustrate another variant of induction evaporator quite acceptable for metal deposition on semiconductors, consider the evaporator proposed by (Phinney & Strippe, 1988) and presented in Fig. 6.

The evaporator 50 with an inductor 22 is disposed in a vacuum chamber on a base 11. The evaporator comprises a crucible 58, a RF susceptor 57 for coupling with the electromagnetic field of the inductor, an inner heat shield 55, surrounding the crucible, and an outer heat shield 53. A thermally isolating hollow column or spacer 52 supports the outer heat shield 53. The contained within the outer heat shield 53 is a second thermally isolating spacer 54, which supports and thermally isolates the inner heat shield 55 from the outer heat shield 53. Within the inner heat shield 55 there is disposed a third thermally isolating spacer 56, upon which there is disposed the RF susceptor 57, which in turn supports the crucible 58. This crucible is provided with a flared lip 59, which extends outwardly from the inner edge of the crucible above the upper edges of the two heat shields 53 and 55. The lip 59 protects the top exposed edges of the shields 53 and 55 and prevents any of the species, being evaporated from within the crucible, from depositing on the inner edges of the heat shield 55. The crucible 58 contains a charge 19 of material (Cu in the given case) to be evaporated.

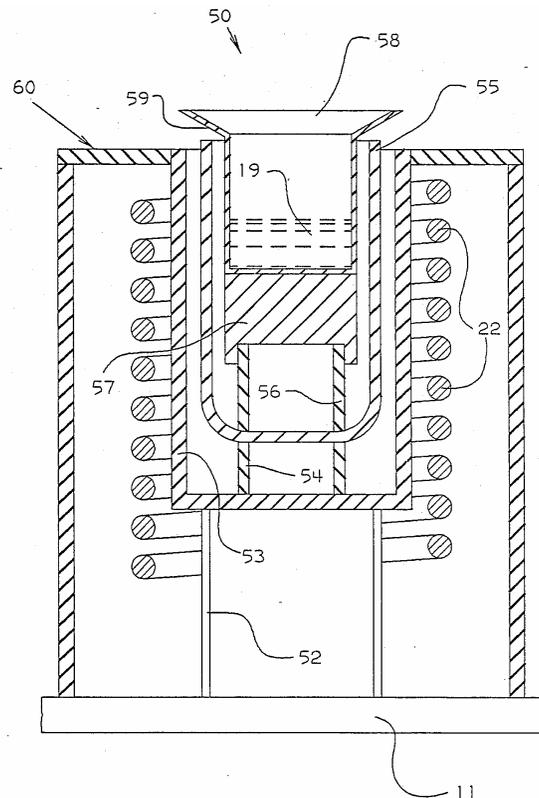


Fig. 6. Induction-heated evaporator by patent (Phinney & Strippe, 1988). Ssee position names in the text

The multi-turn inductor 22 surrounds the above-described structure. There is also an outer casing 60 coupled to the outer heat shield 53 to surround and enclose the inductor 22. The casing 60 prevents any of the evaporated species from depositing on the inductor 22, the outer heat shield 53, and the thermal-isolating column 52. This casing may not be used but because it aids in extending the life of the inductor and eliminates discharge phenomena in the vicinity of the inductor as well as the need of cleaning the column 52 and outer heat shield 53 after every evaporation run, it is very desirable.

The RF susceptor 57 is made of a solid, columnar block of graphite carbon 3.5 cm in diameter and 1.3 cm height. The crucible 58 is 2.1 cm in diameter and 1.7 cm deep, and is made of a material (*e.g.* Mo) that has a lower vapor pressure than the material to be evaporated (*e.g.* Cu). The crucible is filled with approximately 30 g of material, such that it is partially filled (*i.e.* the material within the crucible forms a pool approximately 0.8 cm height). The susceptor 57, being a relatively large mass and being very receptive to inductively heating by RF frequencies, assures that the charge 19 contained within the crucible 58 is quickly heated in controllable manner to the desired levels necessary to melt and evaporate the charge 19 as well as to provide it self-fractionating after its replenishment. Such susceptors are especially required when the charge is a low vapor pressure material (*e.g.* Cu or Au) that is not very absorbent of RF energies. Thus, it is necessary to select both the configurations and compositions of the susceptor 57 and crucible 58 so as to absorb the maximum amount of RF energy and convert it into heat, and to transfer the heat by radiation and conduction to the charge 19 within the crucible 58. The other components of the evaporator 50 are selected to minimize this RF absorption so that the level to which they become heated is minimized.

One can see from the above-described crucible dimensions the depth of the crucible is less than its diameter that is the evaporator generates the sufficiently wide vapor flow to the substrate and does not create the problem with uniformity of deposited films. The problem arises if the depth/diameter ratio is increased to the point where the depth is greater than twice the diameter.

Since it is necessary to heat the contents of the crucible rapidly and effectively (*i.e.* with low heat losses), one uses the thermally isolating system for the crucible and the RF susceptor. The system consists from two concentrically disposed cup-shaped heat shield 53 and 55 as well as three thermal insulator/supports/spacers 52, 54 and 56 in the form of hollow columns. The materials of the thermally isolating system parts must be refractory, transparent to radio frequencies and have low heat conductivity. Practically, these materials are found among refractory dielectrics.

Pyrolytic BN is the exemplary material for the inner heat shield 55 and the thermal insulating spacers 56. Pyrolytic BN is used as the inner heat shield because it prevents infrared radiation from being emitted from either the susceptor or the crucible. In this way the inner shield acts to retain heat within itself, *i.e.* in the crucible area and susceptor area, while shielding the outer shield 53 from infrared radiation. Pyrolytic BN is used for the support spacer 56 not only because it is a good thermal isolator but also because it is formed with the highest purity and does not interact with the carbon of the susceptor 57 at the high temperatures at which evaporator operates. Silica and quartz are the exemplary materials for the other parts of the thermally isolating system. Other materials having the similar properties could be used. Of course, the common demand to the all materials used for manufacturing the evaporator parts is high purity and no interaction each with other and with evaporated substances.

A special mechanism is provided within the evaporation chamber for automatically feeding a wire, of the material in the crucible being evaporated, into the crucible to automatically replenish the charge 19 as it becomes diminished by evaporation.

It is important to note, the choice of above-described dimensions of the evaporator parts, and especially the commensurable volumes of the graphite carbon susceptor and molten material prior to the evaporation provides the process of self-fractionation of the molten material before the evaporation and thus enhances the properties of the film as-deposited, in particular, eliminates conductivity-detracting impurities from the deposited film. Since this moment is critical for technology of precision thin films for microelectronics and optics, discuss it more detail.

It is known that during the initial heating and melting phase any impurities contained within the charge would, during the evaporation thereof, emit species of impurity material (*e.g.* oxide, slag, *etc.*) from the melt. This phenomenon (known as "spitting") results in large quantity of species of the source material and impurities in solid or liquid state being transported to substrates. The spits or impurity species in any form cause irregularities, such as spheres, lumps, or voids, in the deposited films. The irregularities reduce the conductivity of the films, causing electrical and structural defects and the like that result in a lower production yield. Patent (Phinney & Strippe, 1988) proposes the following procedure of self-fractionation during the film deposition cycle with periodical replenishment of the evaporated charge.

The Cu charge 19 of 30 g is disposed within the crucible 58 (see Fig. 6). At the beginning of the cycle, the substrates (semiconductor wafers) are located in the vacuum chamber and the one is evacuated. 450 W of RF power is then applied to the inductor. The susceptor 57

begins to heat and transfer this heat by a radiation and conduction process into the charge in the crucible, and the charge begins to melt with little or no evaporation occurring. This power level is maintained until the temperature of the charge stabilizes at 1200 °C. During this time, to protect the substrates from any undesired evaporation or contamination, a shutter closes the substrates. When they are to be coated, the RF power is ramped to about 2 kW, and is maintained there for approximately 135 seconds. During this time the temperature of the charge 19 is increased and self-fractionation, i.e. fractional distillation, of the charge occurs. As this fractional distillation begins, any contaminants in the charge (such as oxides, or the like) adhere to the surface of the melt to form a slag-like surface layer. As the RF power is increased, the temperature of the charge also increases and the fractional distillation continues until the contaminants collected on the liquid surface of the melted charge become either sublimated or vaporised due to limits set by respective vapor pressures of the material used in the charge and its oxides and other contaminants. The impurities migrate to the wall of the crucible where they are drawn up. In copper, this occurs because a high vapor pressure of copper evaporated species is created, which pushes the surface contaminants and the like to the side walls of the crucible that are at a temperature slightly higher than that of the melted charge. Once this fractionation process is completed the temperature of the crucible has peaked at 1900 °C.

The RF power is then decreased to a power level of 780 W. This power level corresponds to the desired rate of deposition of 4 Å/sec of the charged material onto the substrate surface (for the crucible-substrate distance of 71 cm). 45 seconds after this decrease in RF power, when the temperature of the charge 19 stabilizes at about 1500 °C, the shutter is withdrawn to open the substrates and to permit the evaporated species of the source material to coat the substrate surface. This power level is maintained for an additional 150 seconds at which time the shutter is then positioned in front of the substrates to prevent further deposition. Simultaneously with the shutter closing, the power level is raised to 1000 W and is maintained at this level for 90 seconds. This causes a slight rise in temperature of the charge. 24 seconds after the shutter closes and the power level rises, the wire of the source material is fed by the wire feed mechanism into the crucible 58 to replenish the charge 19 therein. As the wire melts and replenishes the charge the temperature of the charge decreases. At the end of 90 seconds the power is again decreased to the standby power level of 450 W and the temperature of the charge continues to decline to 1200 °C at which temperature it again stabilizes.

By carrying out the technological cycle, as described above, the crucible 58 is heated in a thermally controlled manner to provide self-fractionation. This self-fractionation eliminates any spitting of Cu or Cu oxides. Self-fractionation of the charge is especially important when the material of the charge has impurities that are introduced during its replenishment. The employing of several shields for the outer surface of the crucible 58 and the total shielding of the inductor 22 together with evaporation of copper, not inclined to wet the crucible wall surface and to migrate upwards to the top edge of the crucible, ensure no parasitic electrical discharges and no ionizing radiation emission from the evaporator.

Thus, the right constructive design and employing various heat and electromagnetic shields are very important in order to create the induction evaporator being without electrical discharges and ionizing radiation emission. However the electrical design of the induction evaporator is important too, because electrical discharges are, in the first instance, electrical processes caused by electromagnetic fields. The often-used approaches, well known from the induction heating technique to avoid electrical discharges, are as follows:

- lowering the operation frequency, *e.g.* from 440 kHz down to 66 kHz and less;
- grounding the upper inductor turn with a copper strap well connected to the base plate of the chamber near the inductor, it provides the ground potential for the upper turn which would be most subjected to the vapor;
- eliminating a stray magnetic field above the inductor in the space of vapor movement by several ways: i) to introduce an electromagnetic shield above the inductor, the shield must have an orifice for the vapor flow and separate the inductor area and the space where vapor flies, ii) to wind one or two top turns of the inductor in the opposite direction relatively the rest lower turns in order to compensate the magnetic field of the lower turns in the space where vapor flies (see the photography of such inductor in Fig. 2, that is the left inductor), iii) to introduce a shorten turn (*i.e.* a ring) over the upper inductor turn, the current induced in the ring will generate the opposite magnetic field relatively the inductor field, iv) to decrease as much as possible the inductor voltage that is to use a single-turn inductor instead the multi-turn inductor.

The most of the listed approaches are realized in the induction evaporator shown in simplified form in Fig. 7 (Kuzmichev & Tsybulsky, 2008). The real sample of the evaporator is presented by its photograph in Fig. 2. The induction system of the evaporator belongs to the concentrator-type inductors known from the induction heating technique. The induction system factually is a step-down RF transformer comprising the concentrator body 1 and the multi-turn coil 2 from copper pipe connected to a RF power source. The concentrator 1 is manufactured from copper and has the narrow radial gap (split) 3 coupling the outer surface 4 of the concentrator with its internal surface 5. The latter surrounds the space where the crucible 6 and other parts may be disposed.

The coil 2 serves as a primary winding of the transformer. The outer surface 4 of the concentrator 1 under the coil 2 serves as a second one-turn winding that is as a load winding of the transformer. The internal surface 5 of the concentrator 1 serves as a one-turn inductor for heating the crucible 6 (or RF susceptor if it introduced into the evaporator). The facing each to other surfaces 7 of the radial gap 3 in the concentrator body 1 serve as plate buses (conductors) connecting the second one-turn transformer winding with the one-turn heating inductor. It is clear that all the mentioned surfaces 4, 5, and 7 pass RF current induced by the coil 2 within the skin-layer. Such arrangement of the secondary circuit of the RF transformer allows to minimize the working voltage of the heating inductor and the stray magnetic field above the concentrator in the space where vapor flies as well as to simplify grounding the top turn of the coil 2 together with the concentrator body 1. All these factors ensure no electrical discharge and no ionizing radiation emission from the evaporator. It is worth to note the RF transformer play a role of a matching element providing effective transportation of RF power from the generator to the heated crucible. Our use of such an inductor-concentrator reduces the power of the RF generator working at frequency of 440 kHz by more than 2 times.

The described induction evaporator is combined with the pyrolytic BN crucible preventing from liquid metal migration towards the outer crucible side (see Fig. 5 and Fig. 8). The crucible is produced by plasma chemical deposition from the gas phase onto the graphite base, removed after deposition. The crucible and its top-edge shield in the form of the outwardly directed funnel-shaped lip are made as one body. The conical shape of the shield surface provides the alignment of the crucible and the concentrator and drain back of liquid metal into the crucible from the upper surface of the shield. The pyrolytic BN crucible is disposed in a cup-shaped graphite susceptor and they together are disposed within a cup-

shaped pyrolytic BN heat shield and then within the central hole of the concentrator. The evaporator has been designed for deposition of Al and Al alloy thin films for electronics, the power of RF generator is 5 kW, frequency is 440 kHz, the deposition rate is about  $0.1 \mu\text{m} / \text{min}$ .

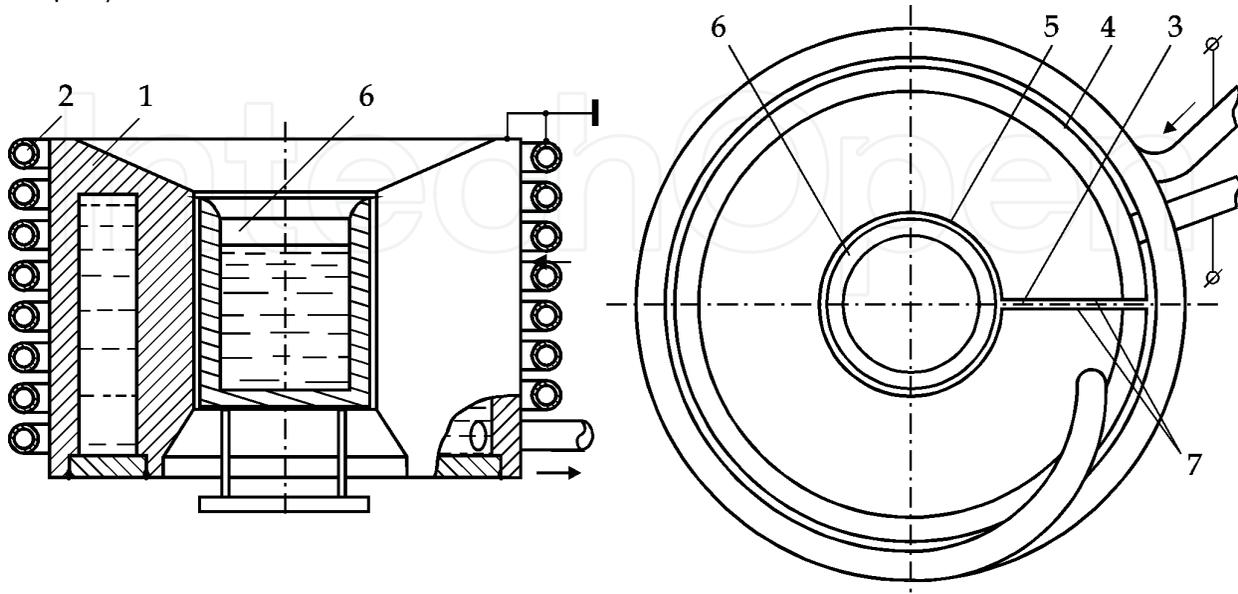


Fig. 7. Evaporator with inductor-concentrator (see position names in the text)



Fig. 8. Evaporator with inductor-concentrator equipped with crucible, which allows avoiding migration of liquid metal towards the outer crucible surface

#### 4. Induction evaporation with vapor ionization for ion plating

##### 4.1 What means the ion plating

Ion bombardment of substrate surfaces during deposition of thin films or coatings is well known to strongly modify the properties of the deposited material. The modification may be very useful as it may enhance adhesion of the films and coatings, make denser their microstructure, increase anticorrosion properties, *etc.* A source of bombarding ions is usually the plasma of electrical discharge maintained in a gas medium or, that is more right

for evaporation process, in the gas/vapor medium. Argon is the often-used gas for ion generation since it is inert and well ionizable gas, hence, the ions  $\text{Ar}^+$  and few of metal ions are considered to bombard the substrate. In reactive processes a proper reactive gas is added, and reactive ions also bombard the substrate. A negative potential is applied to the substrate for acceleration of the bombarding ions. D. Mattox has described such technology in the 1960's and named it as "the ion plating" by analogy with the galvanic electrodeposition process. In his review book (Mattox, 2003), he gives the history and foundations of this technology as well as cites the other names for similar deposition processes, in particular, "ion assisted deposition" - IAD or "ionized physical vapor deposition" - iPVD.

As a prior art for the ion plating technology, it may be pointed out the similar process patented by B. Berghaus in 1938, which includes placing coated articles under a negative bias potential into gas medium, firing gas glow discharge with the articles as a cathode and use of a crucible with molten material as a vapor source (Berghaus, 1939). However the proposal by B. Berghaus is not known, at least, by literature data as practically realized. So, the ion plating technology has been developed since the 1960's due to very attractive results obtained by D. Mattox.

Initially, the ion plating employed a thermal vapor source, mainly resistance-heated boats, and a plasma gas discharge. The resistance-heated vapor sources are employed, as they are simplest ones. The gas discharge is employed since it is a simple way to obtain ions. Besides, the ion plating in gas environment provides good surface coverage over three-dimension substrates due to scattering evaporated species and randomization of their trajectory in the gas medium when gas pressure is about 1 Pa and higher. The application of electron beam evaporators to ion plating with the higher deposition rates than the provided by resistance-heated sources meets with difficulties of electron gun exploitation in the gas medium. So, the induction evaporators that are able to work in the gas environment and ensure the high deposition rate of wide range of materials are better candidates for the ion plating processes. May be by this reason, B. Berghaus proposed to use just the induction evaporator in his ion plating process (Berghaus, 1939).

A simple evaporation system with a bare inductor coil, without shielding, similar to the presented in Fig. 1 has been used for ion plating directly in a gas containing chamber without producing any sparks and arcs in the inductor area (Spalvins, 1976). The system had a ceramic crucible 2.5 cm in diameter and a four-turn inductor connected to a 5 kW generator through a 20:1-ratio step-down transformer. The voltage across the inductor was 70 V with the operation frequency of 75 kHz (the conventional frequency for induction generators of 440 kHz could not be utilized in the gas ion plating process because severe arcing in the inductor occurred in gas glow discharge conditions). The operation conditions were Ar pressure: 2-2.5 Pa; the negative substrate bias potential: 3-5 kV; substrate ion current density: 0.3-0.8 mA/cm<sup>2</sup>. Very good results of metal and alloy (Au, Pt, Ni, Fe, Cr, Inconel) deposition on various metal surfaces with excellent adhesion of coatings with thicknesses from 0.15 to 50  $\mu\text{m}$  have been obtained (Spalvins, 1976).

#### **4.2 Induction evaporation with vapor ionization in gasless environment for ion plating**

Obligatory introducing of the inert gas into the evaporation chamber in the "classic" gas ion plating leads to some shortcomings, in particular, to gas capture by the coatings and limitations of operation parameters. The much better approach is to use the metal vapor plasma as an ion source and to provide the bombardment by own metal ions that is the self-

ion bombardment. Such gasless approach was named as “the pure ion plating” (Hale et al., 1975). And in this case the induction evaporation has advantages over the resistive-heated or electron beam-heated evaporation since the electromagnetic field of the inductor may provide effective vapor ionization unlike the mentioned processes. To confirm this, consider some results of our experiments with an evaporation system similar to the presented in Fig. 1.

A bare three-turn inductor (its inductance was  $0.3 \mu\text{H}$ ) surrounded a molybdenum crucible 3 cm in diameter (see the photograph of the middle inductor in Fig. 2). An ion collector was placed 20 cm above the crucible. A 5 kW 440 kHz generator powered the inductor through a step-down transformer. The maximum working temperature of the crucible was  $1400^\circ\text{C}$ . The residual gas pressure after gas evacuation from a chamber was about  $10^{-3}$ - $10^{-4}$  Pa. No special gas was introduced into the chamber.

With the crucible grounded and the top inductor turn under a high potential (the lower turn was grounded) a plasma discharge appeared at the inductor voltage of about 300 V. Fig. 9 shows the voltage-current characteristics of the ion collector, when it was under negative voltage ( $-U_c$ ), for the discharges in Cu and Cr vapor. As can be seen from the figure, the curves resemble the voltage-current characteristics of the negatively biased plasma probe with the trend toward saturation of the ion current  $I_c$ . The collector ion current was order of hundreds of milliamperes, with the average current density being  $2$ - $13 \text{ mA/cm}^2$  for Cu evaporation and  $2$ - $8 \text{ mA/cm}^2$  for Cr evaporation. The ratio of the metal ion flow to that of neutral metal atoms at the collector was  $0.05$ - $0.1$ . The latter number may be considered as a vapor ionization degree.

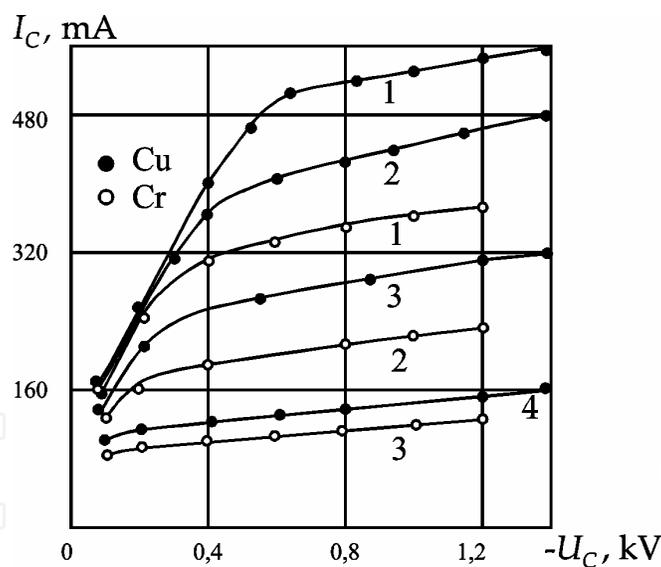


Fig. 9. Voltage-current characteristics of the ion collector for inductor voltage of 500 V (1), 400 V (2) and 350 V (3)

Analysis of the above-mentioned and other related data testifies to the fact that in the described unshielded induction system the appearance and maintenance of the metal plasma discharge essentially depends on shape and disposition of the crucible and the top inductor turns, what parts are grounded as well as the secondary ion-electron emission from the crucible walls and the top turns. The RF magnetic field above the crucible affects electron trajectories, making them longer, and raises, due this, the efficiency of vapor ionization. Therefore this kind of vapor discharge may be classified as a version of magnetic

field supported glow discharge with RF power supply. By increasing the height of crucible walls, one can easily produce a discharge with a hollow-cathode/crucible as an electron-emitting electrode; in this case the upper inductor turn serves as an anode for the hollow-cathode discharge at positive half-waves of the inductor voltage. Thus, the tested induction evaporation system showed that the inductor could simultaneously function as an evaporator and as an ionizer of metal vapor in gasless environment.

It is well known from gas discharge electronics that introduction of a thermionic cathode into a discharge system strongly facilitates discharge ignition due to free electron injection by the cathode. This approach was realized with the above-described induction evaporator. A ring cathode, made from tungsten filament 0.3 mm in diameter, was placed above the crucible and was grounded together with the lower turn of the inductor; so, the filament and the upper turn created a diode with crossed electric and magnetic fields. Passing heating current through the filament from an external power source to cause thermoelectron emission gave effect in some decreasing of the ignition voltage for the vapor plasma discharge, but after discharge ignition the filament heating current had no visible effect on the collector ion current. The same data were obtained with the simpler system with a closed filament cathode at the ground potential. In the latter case the filament was a closed turn heated by the induction current. These results may be explained by the lower electron emission of the filament as compared with the secondary ion-electron emission of the wall surface of the grounded crucible because of small filament surface area and high work of electron exit from tungsten.

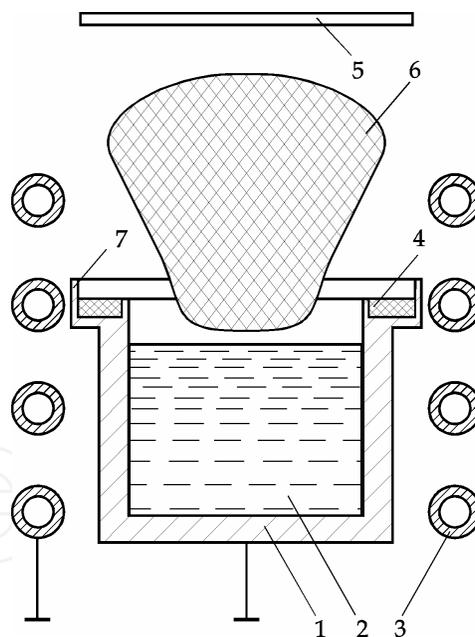


Fig. 10. Schematic diagram of evaporator with crucible containing electron emitting insert (see position names in the text)

In order to successfully realize the approach based on employing thermoelectron emission, an induction system with a special crucible containing an electron emitting insert is proposed (Kuzmichev & Tsybulsky, 2006a). Fig. 10 schematically depicts the system. A cylindrical cup-shaped conducting crucible 1 with a molten metal 2 is surrounded by an inductor 3; its upper turn located above the top edge of the crucible 1. The top crucible edge is a lip flaring towards the inductor coil, and it contains the insert 4. The latter is made from

refractory material with low work of thermoelectron exit, for example, from sintered  $\text{LaB}_6$  or composition  $\text{W}(\text{Mo})+\text{LaB}_6$ . Due to location of the insert 4 close to the inductor coil 3 the insert temperature is sufficiently high to cause thermoelectron emission. The high temperature also provides cleaning of the insert surface from any contaminants by evaporation. An ion collector 5 is disposed above the crucible 1. When the inductor 3 is powered from a 440 kHz generator, the metal 2 evaporates from the crucible and the thermoelectron emission from the insert 4 takes place, a brightly shining plasma cloud 6 appears above the crucible that means ignition of metal vapor discharge. Providing the top edge of the crucible with an additional wall/shield 7 increases vapor ionization due to longer trajectories of the thermoelectrons emitted by the insert. Testing the induction evaporation system with crucible containing the thermoelectron emission insert showed that its use gave increase of ion collector current about 1.5-2 times as compared with the system without the insert.

Lifting the upper inductor turn towards a substrate or the ion collector in our case (see Fig. 10) means, in fact, dividing the inductor coil 3 by two parts: the lower part, surrounding the crucible, is the heating inductor; and the upper part above the crucible is the ionizing inductor; herein, both the parts are connected in series. There is a proposal on induction evaporating systems (Japanese patent application, 1978) provided with two inductors separately for evaporation (the first heating inductor surrounds a crucible) and for vapor ionization (the second inductor is disposed above a crucible and surrounds the vapor flow), see Fig. 11. The first system (Fig. 11a) is provided with the inductors connected in series. This approach needs one RF generator that simplifies RF circuits and allows to automatically stabilize evaporation and ionization processes. Indeed, when evaporation rate increases, the vapor density grows up and the power, consumed by the ionization inductor, increases. Hence, the power for the evaporation inductor decreases and evaporation rate

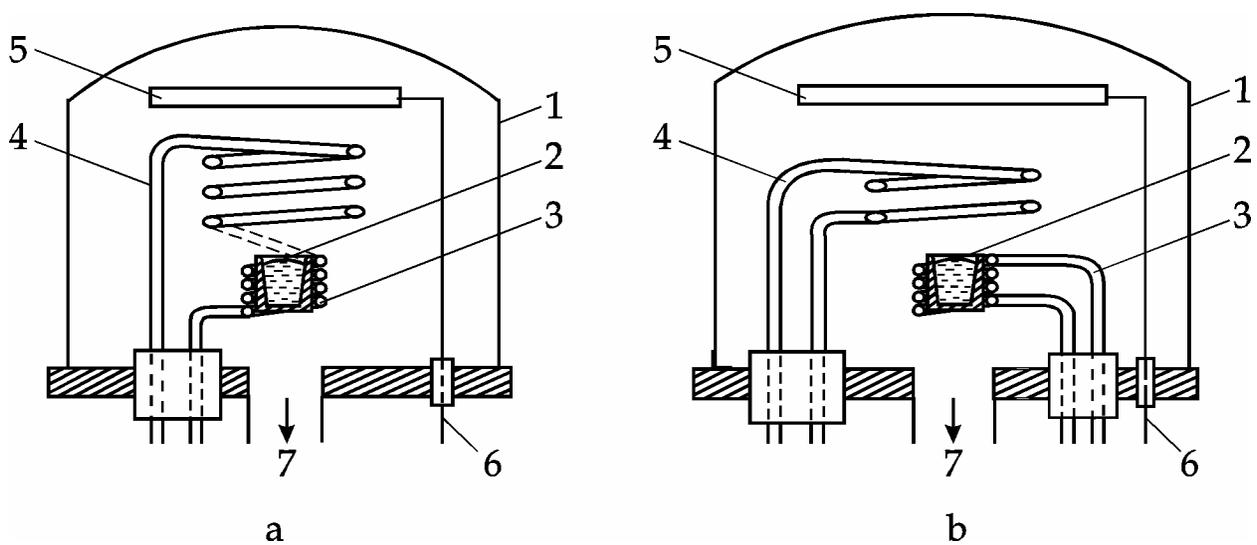


Fig. 11. Schematic diagrams of evaporators with two inductors for evaporation and vapor ionization. (a): the inductors are connected in series; (b): the inductors are connected to different power sources (Japanese patent application, 1978). 1 is a chamber, 2 is a crucible with molten evaporating material, 3 is the lower heating inductor for evaporation, 4 is the upper inductor for vapor ionization, 5 is a substrate, 6 is a conductor for applying a bias voltage to the substrate, 7 is pumping out of the chamber

decreases, too; that is such two-inductor system works in some equilibrium regime. If one desires to rise the evaporation rate, it needs to increase the RF generator power. The second system with separate power supply of the inductors (Fig. 11b) has advantage in separate control of evaporation and ionization processes; also one can use two different frequencies, which are optimal for evaporation and vapor ionization, but such system is more complex. The considered-above induction evaporators with vapor ionization employ bared (unshielded) inductors. This may lead to limitations in use of high power generators at radio frequencies (e.g. 440 kHz and higher) because of arcing in the crucible/inductor units. High RF power is needed for enhancing vapor ionization degree. Besides, ion sputtering of induction system parts, which are under negative potential relatively the vapor plasma and contact with the plasma, leads to introduction of undesired impurities into the deposited coatings. So, the high power stable pure ion plating needs the specially designed shielded induction evaporator/ionizer systems. Consider two examples of realization of shielded ion plating sources.

The first source, called "the i-Gun Ion Source", is described in (Hale et al., 1975). It is similar to the induction system presented in Fig 1, but in this case the bare inductor is surrounded by a grounded cylindrical cup-shaped metal shield with a cut on its wall along the axis; a heat shield is placed between the crucible and the inductor. The grounded metal shield serves to inhibit coupling of the RF into the surrounding plasma, which would otherwise result in side discharge appearance and significant power losses. The plasma discharge may appear only above the crucible. The heat shield, made from alumina and being transparent to RF, allows the RF energy to couple to the conducting carbon crucible while to inhibit crucible heat losses. The tube conductors for connecting the inductor with a RF generator are also covered by grounded metal shields. In result, the electrical design of the i-Gun Ion Source allows the bare inductor to continuously operate without arcing at 440 kHz power up to 5 kW. The deposition rates of about 25  $\mu\text{m}/\text{min}$  at distance of 25 cm are possible for Al or Cu. The efficient use of power offers approximately three-fold operation energy saving over electron beam evaporation of such metals as Al.

The second high rate ion plating source is described in (White, 1977) and schematically depicted in Fig. 12. A conducting carbon crucible 1, containing evaporated material, is supported by a nonmagnetic dielectric support 2 and surrounded by a heat shield 3, made from alumina. All these parts are surrounded by a multi-turn working inductor 4, which is externally covered by a cup-shaped metal shield 5. The conductors 6 for connecting the inductor 4 with a RF generator are also covered by metal shields 7. A conductor 8 is to apply the ground potential to the middle turn of the inductor 4. The inductor 4 and the conductors 6 are made from copper pipe to provide water-cooling. The shields 5 and 7 have cuts along their walls in order to inhibit RF power losses. The copper pipe forming the turns of the inductor 4 is generally covered completely by fiber insulation 9 (such as aluminium oxide fiber batting) that is required to maintain proper spacing and to prevent shorting between turns. The fiber insulation batting 9 supports the tubular heat shield 3 and the dielectric cylindrical block 2, supporting, in turn, the crucible 1. The conductors 6 are covered by dielectric material such as Teflon to prevent shorting between conductors 6 and their shields 7. The metal shields 5 and 7 as well as the crucible 1 are at floating potential that serves to inhibit coupling of the RF power into the plasma. The shield 5 has no metal part on its top to provide coupling RF field of the inductor with a plasma discharge that can appear only above the crucible.

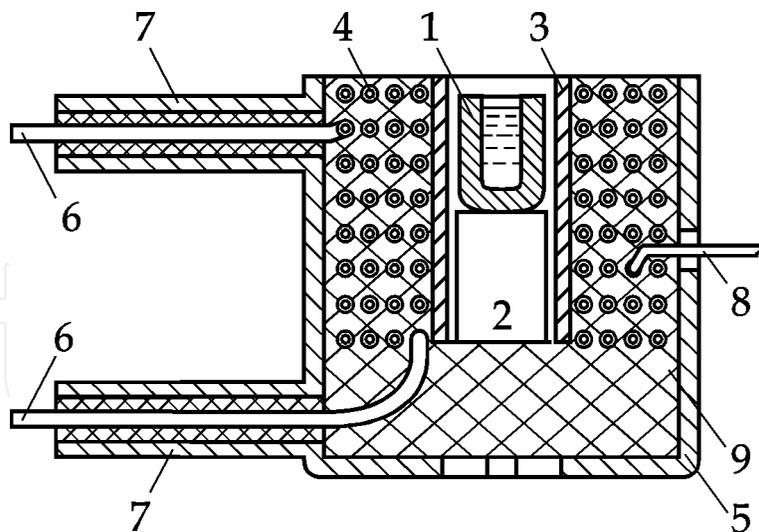


Fig. 12. Induction evaporator for ion plating source (White, 1977) . See position names in the text

Vapor species of the material, being heated and evaporated in the crucible 1, pass along the source axis through the RF electromagnetic field, generated by the inductor 4, in the area of the strongest field just above the crucible. When strength of the RF field is higher than the threshold value, the plasma discharge arises due to vapor species ionization. This plasma discharge relates to so called "Inductively Coupled Plasma". The main feature of the discharge is its firing in metal vapor gasless environment. Electrical design of the ion plating source prevents from arcing and parasitic discharges and allows the source to operate throughout a pressure range from ultra high vacuum to greater than atmospheric pressures. So, the considered shielded ion plating sources can operate in the reactive mode of deposition with introducing some quantity of proper reactive gas in the chamber. The sources may provide ion plating of metals and semiconductors such as Al, Cr, Cu, Si, Ti, *etc.* and of oxides, nitrides, sulphides  $\text{Al}_2\text{O}_3$ ,  $\text{HfO}_2$ ,  $\text{HfN}$ ,  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$ ,  $\text{TiO}_2$ ,  $\text{ZnS}$ ,  $\text{CdS}$ , *etc.* on different substrates (metal articles and foil, semiconductor wafers, glass, polymer films).

#### 4.3 Evaporator with pulse modulation of inductor power supply

The evaporator, in which the same inductor generates the electromagnetic field for heating of the evaporated material and for ionization of the vapor, has the disadvantage associated with the inability to separately adjust the evaporation rate (*i.e.* vapor density) and the degree of vapor ionization because of simultaneous influence of the inductor electromagnetic field on the processes of evaporation and ionization. This disadvantage can be overcome by introduction of the amplitude pulse modulation of the RF power, at which the average value of the inductor power will determine the intensities of the crucible (or susceptor) heating and the metal evaporation, while the amplitude of the inductor power will determine the degree of vapor ionization. The evaporation will be practically continuous because of the great thermal inertia of the crucible (susceptor) and the molten metal, but the vapor ionization will be pulse modulated. Indeed, when the inductor power amplitude is above a threshold value, the plasma discharge occurs in the vapor medium, and when the inductor power amplitude is below the threshold value, the discharge breaks off at once. In such approach the strong non-linear dependence of the degree of vapor ionization upon the instantaneous values of the RF power that is the inductor electromagnetic field strength is used.

There are many ways to provide amplitude pulse modulation of RF power well known for radio engineers but, in induction heating technique, one uses mainly the self-exciting triode tube generators, power control of which is commonly made by regulation of either the filament current (to control the cathode emission current), or the anode voltage, or the grid resistor. The first method has great inertia and the constraints on the allowable minimum and maximum filament currents, as well as a decrease of triode tube life; so this option does not fit for pulse modulation. The control of the anode rectifier voltage with thyristors or thyratrons is advisable only to adjust the average power of RF heating. The fast amplitude pulse modulation of the anode voltage with vacuum tubes is quite complex and cumbersome. Also, the modulation of RF power by the regulation of the grid resistor is of little interest.

One may suppose, the quite effective is the modulation by periodical applying of negative voltage pulses to the grid of the generating triode in order to periodically close and open the triode. Earlier, the applying of high negative voltage to the grid was used for emergency shutdown of the generator. The regulation of the ratio "vapor ionization degree/vapor density" at the fixed amplitude pulse modulation may be carried out on the principles of pulse-width or pulse-frequency modulation.

The method of the pulsed grid modulation was proved with the self-exciting triode generator working at 440 kHz, the triode was of GU-89A type (made in Russia). The rectangular modulation pulses of negative polarity with amplitude up to 800 V and duration of 0.03-5.0 ms (the pulse front duration was about 10  $\mu$ sec) were generated by a transistor submodulator with independent control of the pulse duration and the frequency of pulse repetition. Three-turn inductor, made from water-cooled copper pipe, surrounded the crucible 32 mm in diameter, made from a conductive material (Mo or TiB<sub>2</sub>+TiC+AlN ceramics). See design of such induction system in Fig. 1. The charge of Cu or Cr was loaded into the crucible. The upper turn of the inductor was connected to the high terminal of the secondary winding of the RF step-down matching transformer and was disposed above the crucible, that provided establishing the RF electromagnetic field over the crucible and the metal vapor ionization in a glow-like plasma discharge, when the voltage applied to the inductor was above 300 V.

Fig. 13 shows the simplified time diagrams of the voltages on the triode grid  $u_g$  and the inductor  $u_i$ . Here  $T$  is the repetition period of the modulating grid pulses. When the negative modulation pulse opened the generator triode and stopped the RF generation, the damping of oscillations in the inductor did not happen instantly, but over time  $\tau_d$  about 40  $\mu$ sec, due to the high Q-factor of the triode anode circuit. When the pulse duration  $\tau < \tau_d$ , some inductor voltage was maintained throughout the negative modulation pulse (it did not down to zero) as shown in Fig. 13a. Then after the end of the negative pulse, the oscillations in the anode circuit self-excited and the inductor voltage  $u_i$  recovered nominal value during the time interval shown as  $\Theta$ . The recovery process duration  $\Theta$  depended on the degree of damping of oscillations in the anode circuit and the inductor and the parameters of the heated matter.

If the pulse duration  $\tau > \tau_d$  and the RF oscillations were completely damped during the action of the negative modulation pulse, then the process of their recovery in the anode circuit and the inductor strongly delayed (see Fig. 13b). The recovery time  $\Theta$  reached 500  $\mu$ sec. There was even a "dead" period  $\Theta_1$  in the generation of the inductor current when the voltage on the inductor was practically absent, although the negative modulation pulse already passed. The reported features of transients were caused by that the triode generator operated in the self-excitation mode.

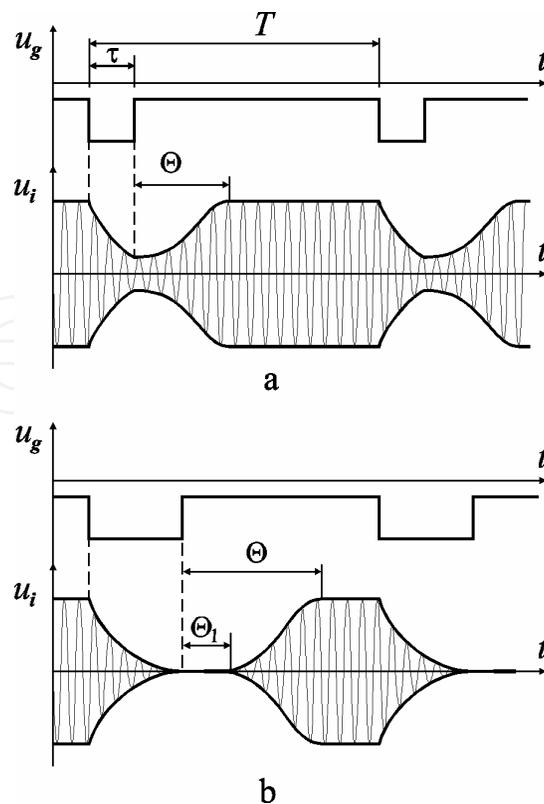


Fig. 13. Diagrams of voltages on the generator triode grid and the inductor

Thus, there are the restrictions on the minimum duration of the modulation pulses (in our case, it must choose at least 50-60  $\mu\text{sec}$ ) if one wants to get 100 % of the amplitude modulation. With a less pulse duration, the modulation depth decreases, *i.e.* RF heating continues during the action of the negative modulation pulses but the ionization stops. It is also worth-while to choose the duration of the pause between pulses ( $T-\tau$ ) more  $\Theta$ , and, of course, much more  $\Theta_1$ . In order to exclude the regime with the "dead" period  $\Theta_1$  and provide more fast regulation, one should use the partial amplitude modulation or external excitation of the triode generator during the "dead" period.

By varying the duration of the modulation pulses ( $\tau$ ) and the frequency of their repetition ( $1/T$ ), one could choose different modes of the evaporation and the vapor ionization. This allowed to independently regulate the temperature of the crucible within 1000-1600  $^{\circ}\text{C}$  and the ion current to the substrate, located above the crucible, within 100-500 mA. Since the pulse modulation enables continuous control of the average power of heating, the employing of the stabilized anode voltage source for power supply of the RF generator may be excluded. The same approach may be applied to semiconductor RF generators for induction heating.

Pulse modulation of the inductor power supply for the separate regulation of the evaporation and the vapor ionization may be also accomplished with the thyatron (or thyristor) pulser, which provides impact exciting of a ringing oscillating circuit. The inductor serves as an inductance in this oscillating circuit. Similar pulsers were used for induction heating in the electronic industry (Mastyayev, 1978). The pulser generates megawatt pulses, the average power of which is order of some kilowatts and supports the evaporation, but the instantaneous megawatt power provides the effective plasma discharge firing and the vapor ionization. Herein, the frequency and the amplitude of the inductor

current oscillation as well as the frequency of repetition of the impact inductor excitations determine the ration “vapor ionization degree/vapor density”. The work of such pulser is also considered in Section 4.4

#### 4.4 Two-inductor evaporator with impact excitation of vapor ionizing inductor

The induction evaporation with vapor ionization may be realized in two variants, namely: with use of one inductor, which simultaneously evaporates and ionizes, and with two inductors, one of which evaporates and the other ionizes vapor. The former case was considered in Section 4.2 and Section 4.3. Now consider the case with two inductors. The schematic design of such system is shown in Fig. 11b. This variant gives possibility to use two different frequencies for separate power supply of the evaporating inductor and the ionizing inductor. As a rule, the higher the frequency, the stronger the vapor ionization is, therefore the ionization needs frequencies of about 1 MHz and more (the often used frequency is 13.56 MHz). The optimal evaporation frequency is less than 1 MHz, mainly 66-440 kHz for relatively small crucibles. The ordinary way to power supply the ionizing inductor is use of the continuous non-modulated RF current generated with electronic tube or transistor generators. But there is another way to power supply the ionizing inductor that is employing the thyatron (or thyristor) pulser with impact exciting of a ringing oscillating circuit (see the end of Section 4.3). The pulser is able to generate megawatt pulses, providing the effective vapor ionization at frequencies less than 1 MHz. Employing such frequencies lower than 13.56 MHz gives a technical profit in simplification of RF schemes and impedance matching systems. This approach has been successfully proved with the thyatron pulser, the simplified electrical scheme of which is depicted in Fig. 14.

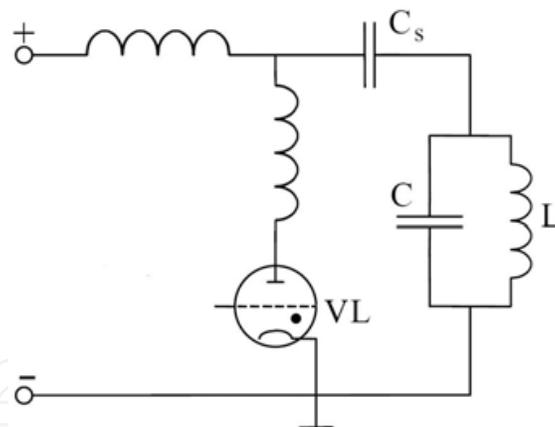


Fig. 14. Schematic diagram of the thyatron pulser for power supply of the inductor for vapor ionization

The five-turn ionization inductor  $L$  together with the capacitor  $C$  forms a self-oscillating circuit or, in other words, the oscillating contour with resonance frequency  $f$  of 0.88 MHz. Also in Fig. 14, VL is the hydrogen thyatron of TGI-1000/25 type (made in Russia) and  $C_s$  is an energy storage capacitor charged from a high voltage anode power source. The repetition frequency  $f_r$  of thyatron ignition is set of 1-20 kHz. As the thyatron being ignited, the storage capacitor  $C_s$  quickly discharges through the thyatron and the LC-circuit, exciting in the latter the self-maintaining free sine-like oscillations but these oscillations are dying ones. When the oscillations cease, the storage capacitor  $C_s$  again charges from a high voltage power source and the process repeats itself after the next act of thyatron ignition. So, the

pulser provides periodical current oscillations in the ionizing inductor and generation of the electromagnetic field by him. This field is able to ionize the vapor and maintain a plasma discharge in the vapor medium due to the very high field strength. It is clear the plasma discharge is a pulse-like one and repeats itself with the frequency  $f_r$ .

The ionization inductor was disposed above the crucible with Al charge as an evaporated material. The power used for evaporation was up to 3 kW, so the metal vapor pressure in the inductor area was order of 0.1 Pa. Electrostatic probes were placed in several points in the vicinity of the inductor, and an ion collector under negative bias voltage of 100-1000 V was disposed above the inductor. The collector was used as a substrate holder, too. The vapor plasma discharge occurred after energizing the inductor L above some critical level (the level of energizing was determined by the voltage of charging  $C_s$  from the anode power supply source). Yet, the ionizing process had a delay that was several half-waves of the inductor current oscillation. Then the plasma discharge continued until oscillations in the inductor died down.

The pulse mode of the discharge led to the pulse modulation of the ion current in the collector (or substrate) circuit. There was a delay (some tens microseconds) in appearance of the ion collector current relatively the inductor excitation pulse as seen in Fig.15, where  $t$  is time,  $j$  - collector ion current density and  $u$  - envelope of free dying sine-like oscillations of the inductor voltage. It is a result of the finite time of plasma expansion outside the inductor. This delay as well as the ion current pulse duration are greater for larger distances between the inductor and collector. Within the 15 cm distance from the crucible with the inductor current oscillation duration  $< 10 \mu\text{sec}$ , the ion current density on the negative probe was 0.1-12 mA/cm<sup>2</sup> and the ion current pulse duration  $\sim 50 \mu\text{sec}$ . The measurements made with help of the probe above the inductor have shown that the charge particles density of Al-vapor pulsed plasma was the order of  $10^{10} \text{cm}^{-3}$ , the electron temperature  $\sim 15 \text{eV}$ , plasma potential was 50-90 V with the 2-3 kV inductor voltage,  $f = 0.88 \text{MHz}$  and 2.5 kW evaporation power. The detail study of this vapor plasma discharge allows concluding that was a glow-like discharge in crossed fields and the inductor served as a magnetic field generator, on the one hand, and as a spreaded electrical electrode, on the other hand. The efficiency of energy transfer from the inductor to the vapor plasma discharge depended on the charge particle concentration or the plasma density inside the inductor. That is why it increased when the following inductor current oscillations were laid on the tail of the previous ones. In this case there was some residual concentration of charge particles within the inductor area already at the moment of applying the following pulse to the inductor, and the discharge began to develop again but without the delay in the first half-wave of the inductor current.

Therefore, the higher repetition frequencies of excitation pulses or the pulse packet regime is more suitable for vapor ionization and vapor plasma generation. However in the former case the average power of the discharge rises substantially and, hence, the pulse packet regime is more preferable. The latter regime presents itself as the succession of groups of excitation pulses and each group or packet in turn contains a few pulses. The pauses between pulses inside the packet are to be smaller than the deionization time of pulsed discharge plasma. The packet repetition frequency is chosen to obtain the required average power of vapor plasma discharge.

Fig. 15 shows the time diagrams of collector ion current density and envelope of the free dying sine oscillations of the inductor voltage for packets containing one (a), two (b) and three (c) excitation pulses. Inside packets, the following pulses were laid on the tail of

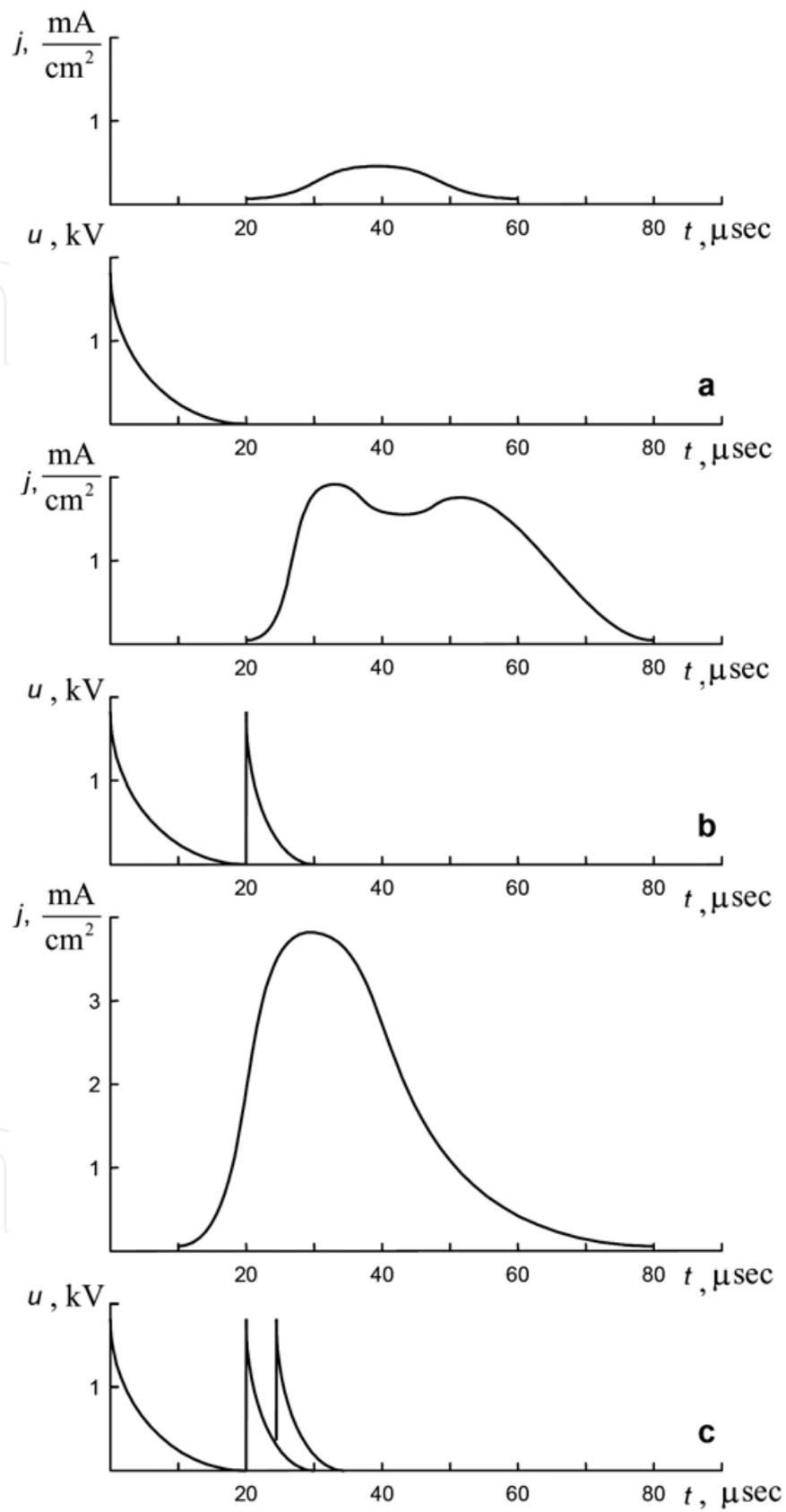


Fig. 15. Time diagrams of ion collector current density  $j$  and envelope of sine-like inductor voltage  $u$  at impact excitation of vapor ionizing inductor. Al-vapor,  $f = 0.88$  MHz

previous ones. As it can be seen, the ion charge given to the collector during one pulse packet and divided by quantity of pulses inside packets is greater for two- and three-pulse packets. Increasing the quantity of pulses inside packets and the delays between them led to varying the shape and amplitude of the ion current pulses. It is seen also, inductor current oscillations after the second and the following excitation pulses died more quickly that indicates more effective energy absorption by the plasma discharge during these pulses.

This system is used for deposition of metal thin films (Al, Ni, Cr, Cu, *etc.*) on a substrate surface with self-ion bombardment. It is suitable especially for coating the temperature sensitive substrates. Other advantages consist in the low plasma potential and weak interaction of the discharge plasma with chamber walls because the duration of the active discharge stage is smaller than the time of flying of vapor plasma species through the space between the ionizing inductor and the substrate. Due to high pulse power of inductor current oscillations, their frequency may be lower than for usual continuous low pressure RF discharges.

### 5. Evaporators with induction heating through dielectric walls of closed envelopes

The production of tube ozonizers and some electronic devices (often called as electronic tubes) deals with metallization of inner surface of dielectric tubes. Also, manufacturing of some special materials and electron components needs evaporation of chemically active, toxic and radioactive substances within closed envelopes. Since induction heating is carried out by the electromagnetic field, which is able to penetrate without losses through dielectric walls, it may be used for these purposes with employing relatively simple equipment. For instance, consider the typical induction system for metallization of the inner surface of tubes, schematically depicted in Fig. 16. A water-cooled inductor 1, made of copper pipe, surrounds a crucible 2 with a charge of evaporated material. The crucible 2 together with a

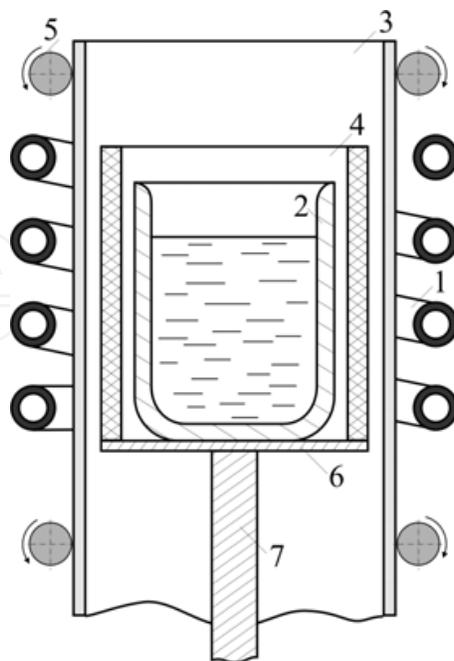


Fig. 16. Schematic diagram of induction evaporator for metallization of the inner surface of dielectric tubes (see position names in the text)



Fig. 17. Photograph of the induction evaporator for metallization of the inner surface of dielectric tubes. 1 is the secondary winding of the RF auto-type transformer, 2 is the working inductor for crucible heating, 3 is the heat shield for protection of substrate from overheating, 4 is the glass tube (substrate)

cylindrical heat shield 4 is disposed within a tube 3 coated inside by metal. The heat shield 4 reduces heat losses of the crucible and protects the tube 3 from overheating. The crucible material is sintered conductive ceramics, for example, of  $\text{TiB}_2+\text{TiC}+\text{AlN}$  composition, graphite or Mo. The shield material is BN or alumina. The tube 3 is centred with help of guides 5. The crucible is mounted on a table 6, attached to a rod rack 7. Fig. 17 presents the photograph of the induction evaporator for metallization of the inner surface of dielectric

tubes developed in Kiev Polytechnical Institute. The feature of this evaporator is combination of the one-turn working inductor with the step-down (11:1) RF auto-type transformer. Both windings of the transformer and the inductor are water-cooled. This evaporator is intended for deposition of metal coating (Al, Cr, Cu, Zn) with thickness of 0.1-2.0  $\mu\text{m}$  on the inner surface of glass tubes 28-34 mm in diameter. After the air evacuation from a chamber, where the evaporator is set up, the glass tube is heated with an incandescent infrared lamp, then a generator of 440 kHz is switched on, and the metal charge in the crucible is heated to a temperature of evaporation. During the coating process the tube slowly rises up to get the thin film coating even on its length.

There may be various modifications of evaporation systems with the induction heating through dielectric walls, and all of them have such an advantage as possibility of the induction system disposition outside the vacuum chamber, in the atmosphere side, that makes service of them easier in comparison with disposition of the systems in vacuum. The evaporator presented in Fig. 17 is able to work in such manner.

## 6. Evaporation with electromagnetic confinement of melt. Levitation evaporation

Common technique of inductive evaporation employs different types of crucibles as containers for melted materials. However, crucibles are often the weak and even undesirable element of evaporating devices because of interaction and chemical reaction of the molten materials, especially, reactive metals such as Al, Ni, Ti, Zr, etc., with the crucible. Great difficulty is usually encountered in selection of crucible materials for evaporating high reactive metals. This problem is more serious than in the case of inductive remelting technology for refining of materials since the evaporation temperatures are much higher than the remelting temperatures. Interaction of molten materials with crucible leads to introducing undesirable impurities into coatings. For example, impurities of carbon in Ti-based coatings may reach some percents and more when Ti is evaporated from graphite crucibles. Moreover, crucibles may be simply destroyed due to solving their materials or reaction products in molten metals and cracking or crumbling crucible walls. For example, when Al is evaporated from ordinary graphite crucible, the latter becomes embrittled because of forming Al carbide.

One of the ways to solve this problem is to realize evaporation process without a crucible for containing evaporated material or, at least, to remove the molten material from crucible walls towards the central axis of the crucible. But, how to do this? The answer may be in applying the mechanical forces, generated due to electromagnetic induction, to molten conducting non-magnetic matter. Such approach is based on the electromagnetic confinement effect well known in magnetohydrodynamics and plasma physics. Herein, the inductor not only generates the electromagnetic force for keeping liquid metal but also provides heating the metal.

As well known from textbooks, magnetic field with induction vector  $\mathbf{B}$  affects unit element of conducting matter, through which current of density  $\mathbf{j}$  passes, with the force  $\mathbf{F}$  directed accordingly to "the left-hand rule". Mathematically, it is expressed as  $\mathbf{F} = [\mathbf{j} \mathbf{B}]$ . The electromagnetic force compresses, squeezes molten non-magnetic metal and removes it into the space with weaker magnetic field. When the strong skin-effect occurs, the value of electromagnetic pressure  $p_{com}$  (in pascals) may be calculated by the following well known formula (2)

$$p_{com} = 3.16 \cdot 10^{-4} P_0 (\rho f)^{-1/2}, \quad (2)$$

where  $P_0$  is density of induction power on the molten metal surface ( $W/m^2$ ),  $\rho$  is specific resistivity of the molten (liquid) metal ( $\Omega \cdot m$ ),  $f$  is frequency (Hz). The pressure  $p_{com}$  in the steady state is balanced by hydrostatic pressure (by liquid metal weight). One can see increase of  $f$  leads to decrease of the electromagnetic pressure.

Fig. 18 schematically illustrates the effect of liquid non-magnetic metal electromagnetic confinement. Dots and crosses show the current direction in the inductor coil 1 and in the molten metal 2. In Fig. 18, 3 is a crucible made from non-conducting material, 4 is a substrate.

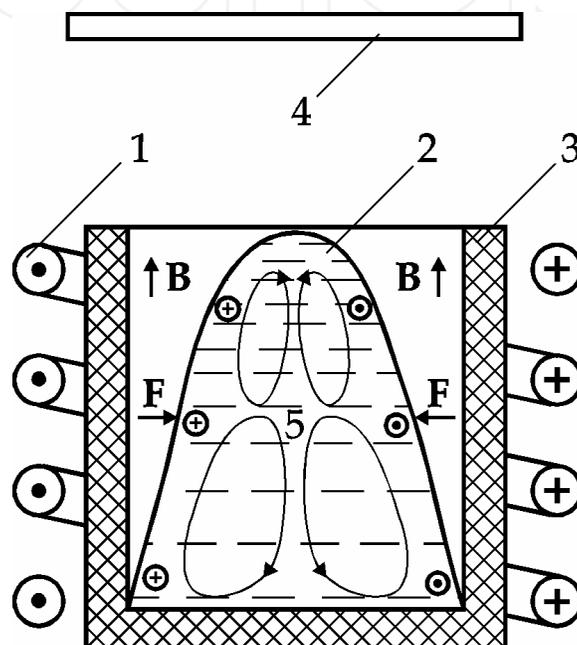


Fig. 18. Schematic illustration of electromagnetic confinement of liquid non-magnetic metal (see position names in the text)

Fig. 18 shows situation when the electromagnetic force  $F$ , generated by the inductor 1, compresses the molten metal 2 and removes it from the vertical crucible walls towards the central axis so, that the liquid metal forms a meniscus at the top. The lower part of the liquid metal lies on the crucible bottom. Accordingly, such system is usually named as providing “the electromagnetic confinement of melt on a support”. If the molten metal has sufficient temperature, its surface emits vapor species going towards the substrate 4 and the crucible walls. The metal, condensing on the walls, flows down to the bottom and then rise up. The balance of electromagnetic and hydrostatic forces as well as surface tension forces defines the shape and position of the meniscus.

The hot metal, condensing on the wall, cannot avoid reaction with the crucible material, therefore such an induction system is suitable for evaporation of non-reactive materials. If induction heating is not sufficient for obtaining the evaporation temperature, one may use an additional heating of the meniscus by electron beam, for instance. The advantage of this system is internal motion of the liquid metal under the external electromagnetic pressure force that provide remixing of chemical elements during metal alloy evaporation and saving coating composition. Fig. 18 depicts simplified trajectories of liquid metal motion (closed lines with arrows): from points with higher  $p_{com}$  (near the mid-plane of the inductor) to

points with lower values of the inductor magnetic field. However motion of conducting liquid leads to its interaction with both own magnetic field and the external field of the inductor, and the real trajectories of liquid metal motion are more complicated than the shown in Fig. 18. The considered case is met in induction coaters with dielectric crucibles and direct heating of evaporated metallic material by eddy current at relatively low frequency ( $\sim 1\text{-}10$  kHz); mass of material charged into crucibles may reach 10 kg and more, RF power is up to 100 kW. It is worth to note the following, forming the meniscus at the centre of the molten metal (*e.g.* Al) allows impurities of metal oxide or carbide to flow down from the top metal surface. Thus, the metal evaporating surface is maintained fresh for a long time to prevent splash generation, and a high quality coatings without pin-holes and other defects are obtained.

The induction system with the melt removed from crucible walls, presented in Fig. 18, is very preferable for remelting reactive materials to refine and obtain high-purity ones since this technology needs temperatures much below the evaporation temperatures, and reactions with the crucible material do not take place. In this technology the crucible bottom is cooled by water, so the lowest part of metal is solid and it serves, in fact, as a crucible bottom and a support for the melt.

Consider what will be if we strongly increase the inductor power. In this case, the electromagnetic force in the inductor mid-plane increases so, that a big metal drop or ball can tear off from the rest liquid metal and soar above the inductor. This is the effect of electromagnetic levitation. To avoid a random non-controlled trajectory of the metal ball, an additional inductor with opposite electrical current is needed above the lower inductor 1 as shown in Fig. 19. The upper inductor 2 will generate electromagnetic force pushing the metal "ball" down and, in result, the "ball" will levitate on a height between the lower and the upper inductors. Fig. 20 depicts the levitation system with the lower inductor of conical shape.

Magnetic fields of both inductors are to create a three-dimensional potential well or electromagnetic trap for the metal ball. For this, the space with weak magnetic field, where the ball will levitate, is to be surrounded by more strong field. The dashed lines in Fig. 20 illustrate the magnetic force line configuration for obtaining such potential well. The liquid metal levitates in a stable space position if the well is symmetric and the balance between gravitation and electromagnetism forces takes place at each point of the levitating liquid metal surface. Accordingly, the primary shape of the metal ball transforms into "top" form

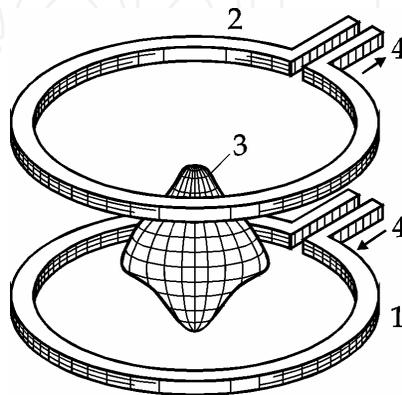


Fig. 19. Two-inductor levitation system. 1 and 2 are inductors, 3 is levitating "ball" of liquid metal, 4 is current direction in the inductors

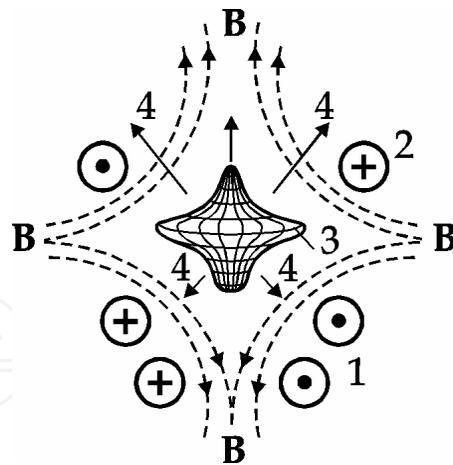


Fig. 20. Two-inductor levitation system. 1 is the lower inductor of conical shape, 2 is the upper inductor with opposite winding, 3 is levitating “top” of liquid metal, 4 is directions of vapor flow; the dashed lines are magnetic force lines

shown in both Fig. 19 and Fig. 20. It is interesting to note, the eddy currents is not induced at “the equator” and “the poles” of the liquid top and the electromagnetic pressure, confining the melt, is absent here, but liquid metal can levitate, not spilling, due to action of surface tension forces. Unfortunately, because of weakness of the surface tension forces, only small quantity of liquid metal (up to tens grams) can levitate.

The feature of evaporating system of such kind is change of vapor flow density during the evaporation process caused by two factors: decrease of the evaporating surface area and continuous movement of the levitating top in the space between the inductors towards a new position with the less magnetic field. Both the factors arise from decreasing the levitating top mass. Nevertheless it is possible to stabilize the vapor flow density if one introduces control of RF power supplying the inductors by the flow density sensor signal.

The evaporating levitation system usually contains the lower conical inductor (as in Fig. 20), the one-turn upper inductor, connected in series with the lower inductor but with opposite current direction, and one RF generator for supplying both the inductors. The evaporation system also contains means to locate a piece of evaporated material (*e.g.*, granule) between the inductors in the space of future levitation before the evaporation process and a crucible (or box) for reception of the residual material after evaporation. Commonly, the evaporated granule is placed in a dielectric holder, which can move along the axis of inductors and lift up the granule to the space of levitation. When the RF generator is switched-on, the electromagnetic force begins to support the granule and the holder is removed down. After heating the granule by the induced eddy current up to the evaporation temperature a substrate shutter (not shown in Fig. 20) is opened and the process of coating the substrate (not shown in Fig. 20) by evaporated material begins. For finishing the process, the RF generator is switched-off and the reception crucible picks up the rest of evaporated granule. The deposition process may be repeated by charging a new portion of evaporated material in the inductor system. The details of evaporating levitation system design and deposition procedure may be find elsewhere (Audenhove, 1965; Vyrelkin et al., 1985).

Since the evaporated material deposits not only on the substrate but also on all surfaces, surrounding the levitating liquid top, and, in the first turn, on the inductor coil, the total evaporated material mass or the time of continuous work of the evaporation system is limited by shorting of the coil. It is possible to enlarge the total evaporated mass at the same time of

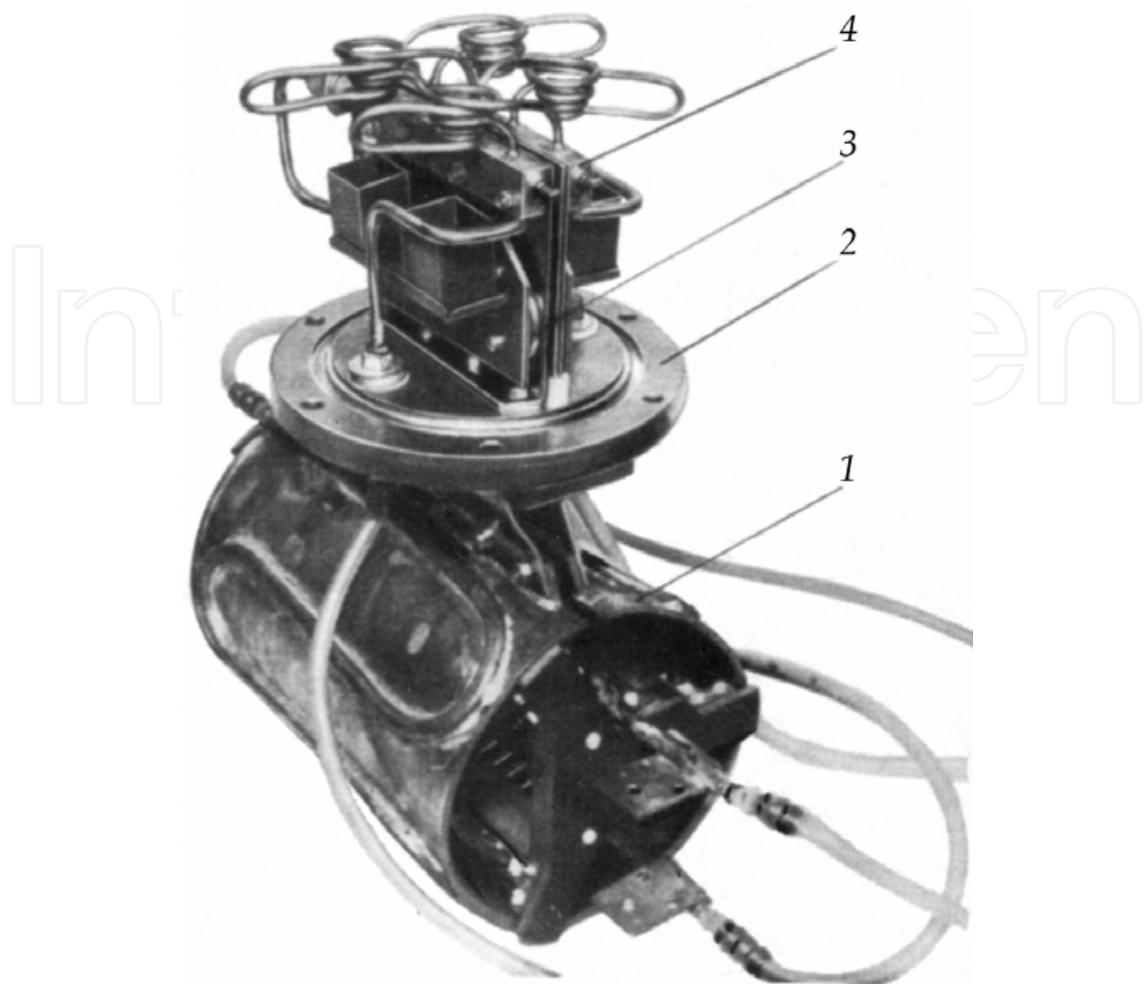


Fig. 21. Photograph of levitation evaporation system with 4-fold inductor unit. 1 is step-down RF transformer, 2 is flange, 3 is crucible unit, 4 is 4-fold inductor unit (Vyrelkin et al., 1985)

continuous work by increase of inductor number. Fig. 21 shows the example of 4-fold inductor unit employed as a part of a compact evaporation system designed in Russia for application in the standard industrial high-vacuum deposition machine instead of resistance-heated evaporators (Vyrelkin et al., 1985). Increase of the inductor number provides also more uniform coating thickness distribution on substrates. The induction system, presented in Fig. 21, has the following parameters given in Tabl. 1.

Average rate of Al deposition during 20 min process with evaporator-substrate distance of 220 mm	0.1 $\mu\text{m}/\text{min}$
Non-uniformity of coating thickness across substrate of 76 mm diameter	5 %
RF power for supply of inductors	10 kW
RF frequency	440 kHz
Evaporated granule mass	up to 2 g
Base gas pressure in technological chamber	less $10^{-4}$ Pa

Table 1. Parameters of evaporating levitation systems with 4 inductors (Vyrelkin et al., 1985)

The main field of application of evaporating levitation system is obtaining high-purity coatings and thin films in laboratory conditions.

## 7. Conclusion

Thus, the principle approaches to design of evaporators with induction heating, determining the main fields of their applications, have been considered in the given paper. A small volume of the chapter does not permit to discuss in details such important branch of application as web coaters with induction evaporation but information on this theme may be found elsewhere (Bishop, 2007). Also, this relates to induction evaporation technology for obtaining and treatment of micro- and nanodispersed materials (Kuzmichev & Tsybulsky, 2006b).

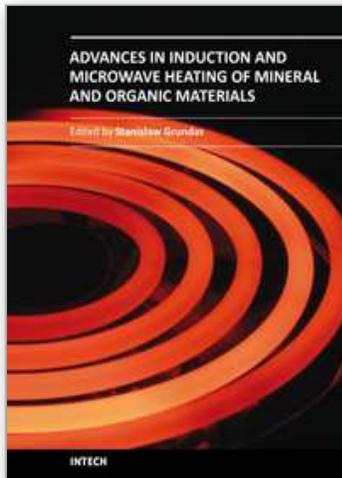
The induction evaporators are able to provide different characteristics of vapor flows including their ionization for very perspective processes of ion plating and, although other evaporation technologies are strong competitors for the induction evaporation, the unusual and interesting possibilities of the induction methods allow them to occupy own niche in industrial technology and for preparation of scientific objects. Moreover, the solutions based on employing pulse technique are to incite engineers towards creation of new types of induction technological and electrophysical equipment. So, we can say the future belongs to the induction evaporation.

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## **Advances in Induction and Microwave Heating of Mineral and Organic Materials**

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