

# High-Frequency Short-Pulsed Plasma-Immersion Ion Implantation or Deposition

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**Abstract** – A new approach in the development of advanced coating deposition and ion implantation method including an application of filtered dc metal plasma and high-frequency short-pulsed negative bias voltage with a duty factor in the range 10–99 % are considered. The ion energy spectrum for different negative bias potential pulse duration (120–1100) ns was measured. The map of different methods of ion beam and plasma material treatment using high-frequency short pulse metal plasma immersion ion implantation or deposition depending on bias pulse duty factor and amplitude for Cu plasma is presented. The ion assisted coating deposition depending on samples conductivity and thickness, plasma concentration, pulse repetition rate and amplitude and duty factor has been examined.

## 1. Introduction

Plasma-immersion ion implantation (PI<sup>3</sup>) using gas-discharge plasma was proposed in the works [1] and investigated in detail in many other works for both pulsed and continuous plasma generation modes. Less work is devoted to metal plasma immersion ion implantation (MPI<sup>3</sup>) [2–13]. The metal plasma immersion ion implantation and deposition named as MePIIID [2–4] was investigated for pulsed metal plasma with rather short bias potential pulse durations (3–10  $\mu$ s) and duty cycle 10–50 %. The regularities of attendant processes for high-concentration ion implantation regime with surface ion sputtering compensation by metal plasma deposition using plasma-immersion approach were investigated in [9, 10].

Use of continuous metal plasma flows, especially filtered of microparticles, for materials properties modification and coatings deposition with ion mixing is of the great interest of high-performance ion implantation and ion-assisted coating deposition methods development [11–13].

This work is devoted to investigation and development of ion streams formation processes, dynamics of near-electrode sheath alternation and ion energy distribution modification, effect of bias potential pulse form, amplitude and duration on characteristics of ion stream formed during high-frequency short pulse plasma immersion ion implantation or deposition (HFSPPIID). Taking into account the opportunity of realization of high-concentration ion

implantation and ion-assisted coating deposition on dielectric substrates, this work includes the consideration of processes accompanying the application of short-pulsed negative bias voltage with high repetition rate.

## 2. Installation and technique

The Scheme of experimental installation is shown in Fig. 1. Vacuum-arc evaporator equipped with a titanium cathode was used for continuous metal plasma flow generation. The arc discharge current was (70–120) A. Shutter-type plasma filter [11–13] was used to remove microparticles from metal plasma flow.

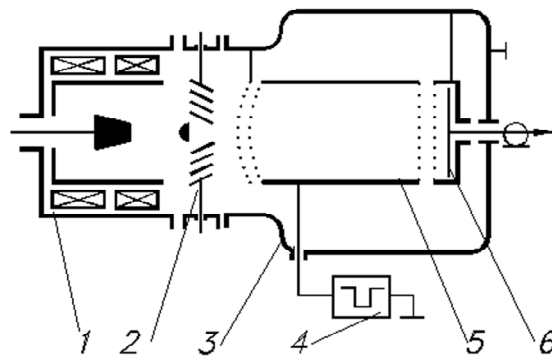


Fig. 1. The scheme of experimental installation: 1 – vacuum arc evaporator; 2 – plasma filter; 3 – vacuum chamber; 4 – high voltage generator; 5 – drift tube; 6 – Faraday cup

The experiments described below were carried out using the vacuum arc metal plasma generator equipped with the plasma filter [11–13]. The vacuum arc plasma generator operated in the dc mode. The arc discharge current was (70–120) A.

To study the behavior of the ions energy spectrum at metal plasma immersion implantation and the time interval of plasma ion-emission boundary stabilization, the time-of-flight method was employed. On the path of the plasma stream, the drift tube of 0.89 m length and 0.1 m diameter was installed; its axis coincided with that of the plasma generator. The drift tube ends were closed by the fine-structure grid. At 2 cm from drift tube end, the Faraday cup was in-

stalled. The pulsed bias potential of negative polarity and variable amplitude and duration was applied to the drift tube. For this a supply source with a pulse duration smoothly adjusted in the range 0.5–2.5  $\mu\text{s}$ , negative bias voltage amplitude from 0 V up to 3 kV and pulse duty factor smoothly adjusted in the range 0.2–0.66 was used. Thus, the grid at the drift tube entrance initially was in contact with plasma and, when the bias potential was applied to the drift tube, the processes similar to those of MPI<sup>3</sup> occurred near this grid. A part of ions, naturally, fell onto the grid, whereas another part having the energy spectrum typical for HFSPPI<sup>3</sup>D came through the grid into the equipotential space of the drift tube. During the ion beam transportation along the tube, its separation by ion energies took place.

Investigation of HFSPPI<sup>3</sup>D method conditions realization and adhesion coating strength were performed at the drift tube replacement by metal and ceramic samples located at a metallic holder.

### 3. Experimental Results and Discussion

In order to clarify the application area of HFSPPI<sup>3</sup>D in the technologies of ion implantation and coatings deposition with ion mixing, it is necessary, first of all, to investigate the influence of pulse duration, plasma concentration, and bias potential amplitude on regularities of the ion beam formation.

The ions energy spectrum for different bias voltage pulse durations are presented in Fig. 2 for Zr plasma. In all cases, the bias voltage pulse amplitude was the same,  $U_{bias} = -2000$  V. Analysis of the energy spectrum shows the following. Ions separation by energies is clearly seen for all bias pulse durations that is conditioned by different charge state of ions. Four distinctive peaks correspond to zirconium ions of charge state  $Z=1-4$ . At the same time, for the energy spectrum of ions with certain, fixed charge state, its significant transformation depending on the bias pulse duration is observed. At short pulse durations ( $\sim 100$  ns), ions with the energy corresponding to the accelerating voltage, i.e.,  $ZeU$ , are practically absent in the energy spectrum. It means that ions were accelerated in an accelerating gap dynamically changed in space and did not pass over the full potential difference. As the bias potential duration increases, the ions of higher energies appear in the energy spectrum. In addition, the average energy of ions in each peak increases, while the energy dispersion decreases. It is typical that each subsequent duration of accelerating voltage forms the ions energy spectrum representing the superposition of the spectrum obtained at a shorter pulse duration and addition corresponding to more energetic ions.

Stabilization of the ion-emission boundary, as follows from the data of Fig. 2 is observed at the bias potential voltage pulse durations exceeding 300 ns for plasma density  $\sim 2 \cdot 10^{15}$   $\text{m}^{-3}$ . Further increasing

accelerating voltage pulse duration leads, first, to the gradual increase of current peak amplitude corresponding to each charge state of ions and then, to widening of this peak at longer durations.

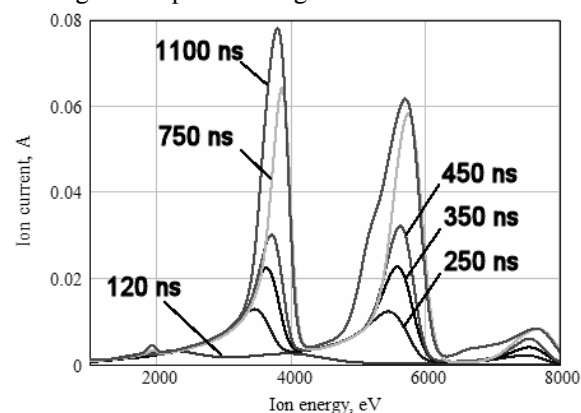


Fig. 2. Ion energy spectra for different pulse durations

So, one can conclude from the data of Fig. 2 that energetically beneficial regimes of short-pulse metal plasma immersion ion implantation can be realized at pulse durations no less than several hundreds ns for plasma density  $10^{15}-10^{17}$   $\text{m}^{-3}$ . In case of short (tens and hundreds nanoseconds) pulse durations, average ions energy will be less than  $ZeU$  because of the dynamics of the accelerating gap formation.

In Fig. 3, the data of bias potential measurements at the metal and dielectric samples surfaces are presented. As evident from the data the potential of the metal sample is fully determined by the bias voltage potential of the pulse generator during the whole pulse.

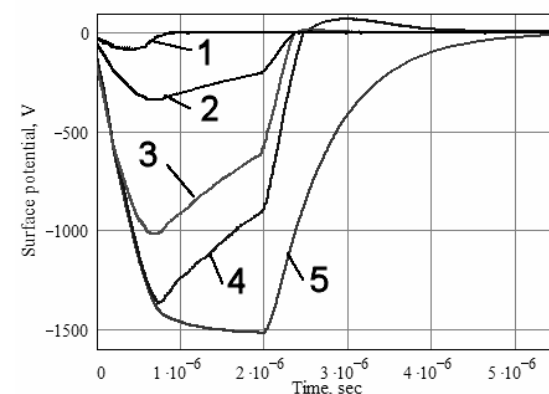


Fig. 3. The potential measured on the surface of the: 1 – glass sample,  $n=2 \cdot 10^{16}$   $\text{m}^{-3}$ ; 2 – glass sample,  $n=2 \cdot 10^{15}$   $\text{m}^{-3}$ ; 3 – glassceramic sample,  $n=2 \cdot 10^{16}$   $\text{m}^{-3}$ ; 4 – glassceramic sample,  $n=2 \cdot 10^{15}$   $\text{m}^{-3}$ ; 5 – metal sample (applied bias voltage)

In contrast to metallic targets when dc bias potential is applied to dielectric samples the acceleration lasts only for short period of time until full charge of the dielectric capacitance. After that the positive charge stored on target surface shields negative bi-

as potential from plasma and ion extraction stops. So, the problem of the investigations was to examine the transient processes near the dielectric surface in short period of time after bias potential application on target.

The analysis of dielectric surface potential waveforms indicates that the potential on the ceramic sample surface is observed during the whole pulse for all used plasma concentration values, while potential value on the glass sample surface greatly depends on plasma concentration. The reason is much greater capacity of the ceramic sample, so it's important to choose the proper relation between plasma and pulse characteristics according to given dielectric sample properties to provide the plasma-immersion ion implantation.

Since the width of the ion sheath depends not only on plasma concentration and kind of ions, but also on bias voltage value, the conditions of "pure" ion implantation into dielectric materials may be provided for different duty factor. The increase of duty factor results in coatings growth rate decrease. Ion implantation without any coating deposition is realized when pulse repetition rate is so high that pause between pulses is enough for expanded sheath filling by unperturbed plasma only.

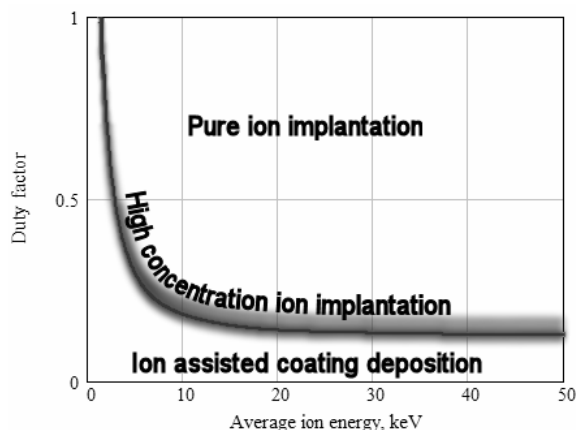


Fig. 4. The map of different methods of ion beam and plasma material treatment for Cu plasma

When the potential of the target surface is zero the plasma deposition on sample surface occurs for dielectric samples just as for metallic samples. If the amount of ions and atoms deposited between pulses is congruent to the amount of ion-sputtered atoms during the pulse the high-concentration ion implantation regime is realized [9–10].

The map of different methods of ion beam and plasma material treatment using HFSPPI<sup>3</sup>D depen-

ding on bias pulse duty factor and amplitude for Cu plasma is presented in Fig. 4.

The area under the curve is intended for ion assisted plasma deposition. The lower duty factor value, the higher coating growth rate. Middle area is intended for high concentration ion implantation, when plasma deposition rate is approximate to ion sputtering one. The area above the curve represents "pure" ion implantation regime, the farther from middle area, the higher surface sputtering rate.

#### 4. Conclusions

Experimentally has been shown that metal plasma based ion implantation as well as high-concentration metal plasma ion implantation with compensation of ion surface sputtering by metal plasma deposition as well as ion-assisted coating deposition can be realized by variation of bias potential ranging from  $0 - 4 \cdot 10^3$  V, pulse repetition rate smoothly adjusted in the range  $(2-4.4) \cdot 10^5$  pps and pulse duration ranging from 0.5 to 2  $\mu$ s.

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