INVESTIGATION OF COPPER SAMPLES WITH ION-PLASMA TREATMENT ON THE HIGH VOLTAGE BREAKDOWNS

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The paper presents the results of studies of the effect of ion-plasma treatment of copper surfaces on their resistance to high-vacuum breakdowns. It has been experimentally shown that the plasma and ion-beam modification of the copper surface leads to an increase of the breakdown voltage from 5 to 35%, depending on the modification method, and reduces the dark current of the anode-cathode.

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INTRODUCTION

The current state of high-energy physics requires the creation of accelerators of a new generation with a high rate of acceleration. Important factors limiting the rate of acceleration are auto-emission loads on accelerating structures and vacuum breakdowns. In particular, one of the main tasks of the CLIC project (Compact LInear Collider), being designed at CERN (European signing center), is the creation of accelerating structures with a record acceleration rate (more than 100 MV/m). In order to ensure the operation of the CLIC and the acceleration rate of 100 MV/m, it is intended that the probability of breakdowns should not exceed 10^{-7} 1/m. Because each breakdown leads to loss of the beam and damage to the accelerating structures, various methods are being investigated ensuring the stability of accelerating structures to high-voltage breakdowns.

Therefore, it is actual the creating and studying the properties of construction materials, which could have the characteristics needed for accelerating structures at the higher work function. One of the way to decrease the breakdown rate in vacuum devices can be work function increasing and, consequently, the breakdown field increasing. The surface modification can be one of the way for this aim.

According to this, it is actual to study the pre-breakdown and breakdown processes to improve the understanding of phenomena and to select the optimal material for electrodes used in vacuum devices and particle accelerators. During recent years, the Institute of Applied Physics National Academy of Sciences of Ukraine (IAP NASU) is a member of CLIC (Compact Linear Collider) collaboration at CERN (European Organization for Nuclear Research). The main direction of join research is to determinate the influence of various factors to breakdown probability in the high gradient electrical fields.

For these goals, there were special special experimental stands at the IAP NASU and at CERN [1, 2]. In the CERN was focused on research of high-voltage conditioning of materials for accelerating structures. In the IPF, the study of the influence of ion-beam and plasma modification of the surface of copper samples on the conditions for the onset and development of high-voltage breakdown was the main research direction.

EXPERIMENTAL STAND IAP

The results presented here obtained on an experimental stand IAP [1].

The experimental setup includes the following elements: vacuum chamber with samples and monopole mass-spectrometer for as RGA (Residual Gas Analyzer) in the vacuum chamber; the system for pre-breakdown current and breakdown registration; the system for vacuum chamber and sample heating; the power supplies; the vacuum pumping system and the equipment for system operation.

The vacuum system design provides the necessary pressure in the different parts of the setup at the preparation step and during experiment process. To minimize the influence of vacuum conditions to breakdown process, the pressure is reached not less than 10^{-7} Pa in the vacuum chamber.

The test samples are placed in the high-vacuum chamber. It has the reciprocating mechanical feedthrough. It is located at the upper flange and allows changing the distance between the electrodes. The bottom flange includes a high-vacuum inlet for a high voltage apply to the cathode. The side flanges are used as windows for visual observation of internal volume of the vacuum chamber and measuring current leads. The sample is fixed at the electrode and can be heating up to 400^{o}C for outgassing. The additional chamber with the monopole mass spectrometer connected to the working chamber.

The electrodes of the discharge gap have tip-plane configuration, and are located in the vacuum chamber. The sample is mounted on the special holder used as cathode. The size of the sample is 11 mm diameter and 2 mm thickness. The sample holder located on the stem isolated from the walls of vacuum chamber. Though high-voltage input (a metal-ceramic insulator), the high-voltage with negative polarity is applied to the cathode. The power supply allows for reached of electrical field strength up to 500 MV/m. The anode has 2.5 mm diameter and rounded end. Before measurement, the vacuum chamber was heated during the day. The mass spectrometer made it possible to measure the mass spectrum of gases in the vacuum chamber. The Ion current anode cathode is measured with accuracy of 0.1 nA.

The discharge gap shown in Fig. 1.

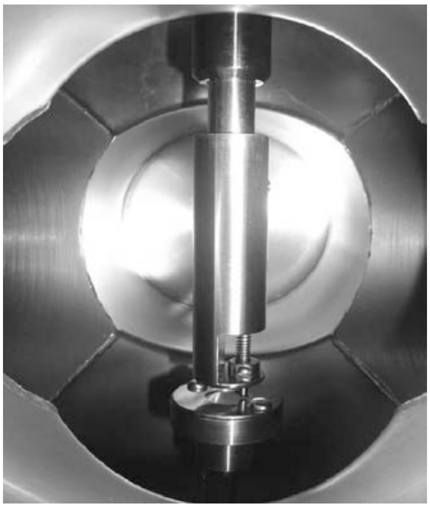

Fig. 1. The cathode-holder with the sample and anode in the vacuum chamber

PREPARATION OF THE SAMPLES SURFACE

Samples of copper with a low content of impurities were taken as materials for the experiments. The anode was a tungsten rod. After the production of the samples, the surface under investigation was subjected to mechanical grinding and polishing. Further, the samples were subjected to a purification procedure, high-temperature annealing in vacuum and treatment in a glow discharge.

Three main types of copper samples were prepared:

1. Surface cleaning – polishing, annealing, cleaning in glow discharge.
2. Ion-plasma coating – coating Ti, TiN, Mo films.
3. Ion implantation treatment.

Half of the sample was subjected to modification. The second half remained unchanged. This made it possible to determine the effect of surface modification on identical copper samples.

Ion-plasma treatment of copper samples was carried out at the Institute of Applied Physics of the NAS of Ukraine – high-frequency magnetron sputtering BC-350, and electron-beam device ELA-60. Ion-beam processing of samples was carried out on the high-dose implanter of the IAP NASU "Vesuvius-5", which allows irradiating samples with both singly charged and multiply charged ions with energies up to 300 keV.

Series of copper samples with coatings were irradiated with Ar^{2+}, Zr^{2+} and N^{+} ions with an energy of 300 keV. The radiation doses varied in the range $(1.2…4)\cdot10^{17}$ cm^{-2}.

Some of the copper samples were subjected to plasma and ion-beam modification of the surface layer. Since the frequency of 12 GHz will be used in the accelerating structures of the CLIC (the thickness of the skin layer for copper is 0.67 μm), the thickness of the modified layer should not exceed 0.67 μm.

Tables 1 and 2 show modes plasma and ion modification copper samples.

Table 1

Modes sputtering thin film coatings on copper samples

Number	Spattering samples	Gas	Coating	Thinks coating, μm
1	Ti	Ar,N	TiN	~0.15
5	Ti	Ar,N	TiN	~ 0.15
6	Ti	Ar,N	TiN	~ 0.15
7	Ti	Ar	Ti	~ 0.2
8	Mo	Ar	Mo	0.08
9	Mo	Ar	Mo	0.08
10	Mo	Ar	Mo	0.4

Table 2

Modes of copper samples irradiation

Number	Surface composition	Ions	Ion energy, кэВ	Dose, 10^{17}cm^{-2}
2	Cu	Ar^{2+}	300	1.6
3	Cu	Zr^{2+}	300	1.2
4	Cu	N^{+}	150	4
5	TiN film	Ar^{2+}	300	1.6
6	TiN film	N^{+}	150	4
7	Ti film	N^{+}	150	4
8	Mo film	N^{+}	150	4
9	Mo film	Ar^{2+}	300	1.6
10	Mo film	Ar^{2+}	300	1.6

EXPERIMENT

In all experiments, the discharge gap between the cathode and the anode remained fixed and was 100 µm with an accuracy of 5 µm. The value of the high voltage gradually increased, creating a constant field in the discharge gap with strength of 50 MV/m and higher up to the occurrence of an irreversible breakdown process.

The value breakdown voltage for each sample of was determined at several locations, to eliminate the influence of the sample surface at which breakdown occurs in previous experiments. For each sample, the breakdown voltage was calculated from many measurements.

DISCUSSION OF RESULTS

INFLUENCE OF ANNEALING AND CLEANING

Part of the copper samples passed the following procedure: polishing; heating in a high vacuum (10^{-5} Pa); cleaning in a glow discharge of argon plasma.

Fig. 2 shows the histogram of the breakdown voltage and the dependence of the dark current on the voltage between the anode and cathode (Fig. 3) for copper samples with different surface treatments (vacuum 10^{-7} Pa).

Figs. 2 and 3 confirm the fact that the state of the surface significantly affects the breakdown voltage. In particular, it is seen that the annealing at 350°C increases the breakdown voltage by 10%, and glow discharge cleaning 20%. This is also confirmed by the dependence of the dark current on the surface cleaning state.

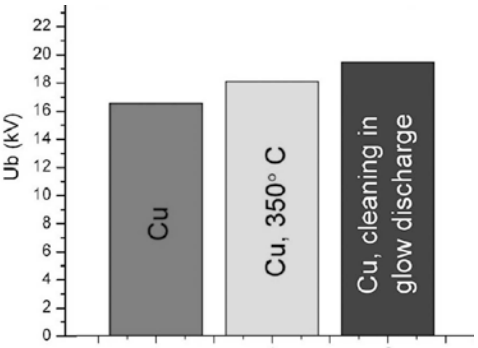

Fig. 2. Breakdown voltage for samples with different surface cleaning

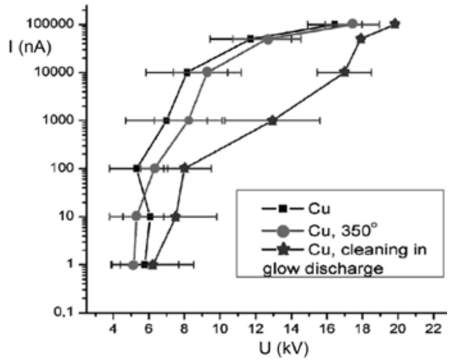

Fig. 3. Dark current for samples with different surface cleaning

COPPER SAMPLES WITH ION PLASMA COATING

Copper samples with thin coatings of metal films with higher breakdown voltages than copper give a stable result of increasing the breakdown voltage (Fig. 4).

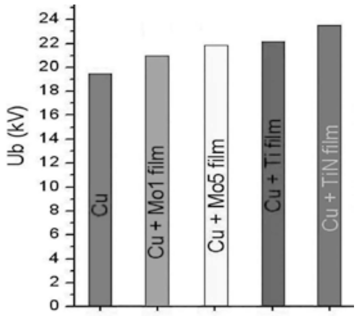

Fig. 4. Breakdown voltage for copper samples with Mo, Ti, TiN coatings

With increasing thickness of the deposited thin film, the breakdown voltage increases and probably approaching the breakdown voltage bulk sample of the same composition. The data on breakdown voltages for samples with thin films deposited correlate well with the results of investigations for bulk samples (Fig. 5).

Coatings with Mo or Ti films increase the breakdown voltage by ~ 16%, and TiN by 20%.

On the surface of copper samples, various defects can be centers of electron emission and stimulate discharge [4]. It can be different inclusions on the surface of the electrodes, grain boundaries, surface defects obtained during its manufacture. In this case, the titanium nitride film smooths and fills surface defects.

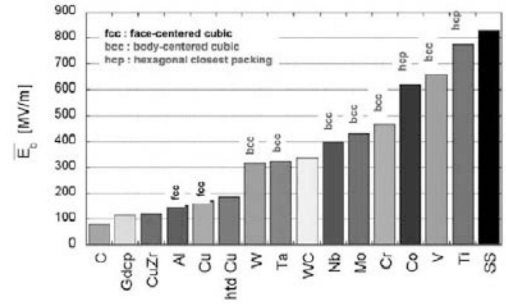

Fig. 5. Histogram of breakdown voltage for massive samples of different materials [3]

Thin titanium nitride films on the copper surface can behave as a barrier to the diffusion of gas from the cathode volume.

In these studies, the anode was made of tungsten.

These conclusions are confirmed by a decrease in the pre-breakdown current on samples coated with titanium nitride, compared with copper samples (Fig. 6).

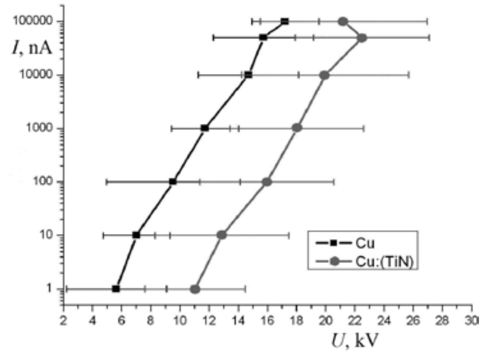

Fig. 6. Pre-breakdown currents for a sample with a TiN films

In [5], the authors showed that the elements of the accelerating structures covered with titanium nitride have a smaller secondary emission of electrons compared to uncoated samples (Fig. 7).

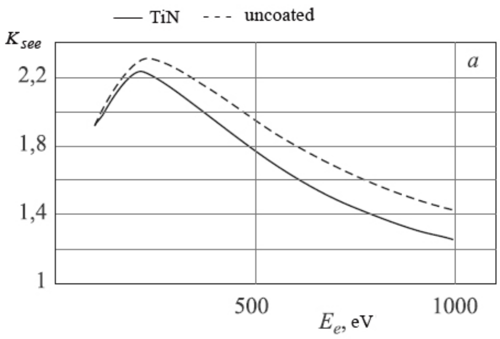

Fig. 7. Secondary electron emission coefficient for stainless steel samples without are coated and with coated of TiN [5]

The authors of this work have received not unambiguous results that require additional research in their opinion, but in our case it is impossible not to exclude such an effect.

ION IMPLANTATION

The results on the irradiation of copper samples by Ar^{2+}, N^{+}, Zr^{2+} ions are shown in Figs. 8-10.

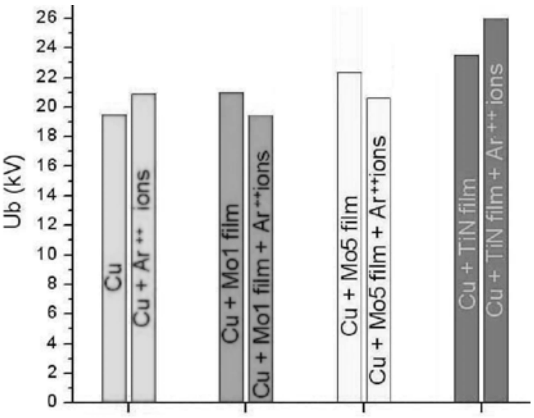

Fig. 8. The results of irradiation of samples with Ar^{2+} ions

Irradiation of copper samples and samples with TiN films of Ar^{2+} ions led to an increase in the breakdown voltage. This is obviously related to the effects of displacement of dissolved gas impurities from near-surface layers by argon ions and smoothing of surface defects.

In contrast, for molybdenum films with different thicknesses, the opposite effect is observed and the breakdown voltage decreases. In this case, for a thinner molybdenum film, the breakdown voltage becomes lower than for a thicker one.

Additional studies of the surface of these samples showed that the irradiation increases the defectiveness of molybdenum films. This probably led to a decrease in the breakdown voltage.

The best result of increase breakdown voltages (35%) was obtained on copper with TiN coating and additional irradiation with argon ions.

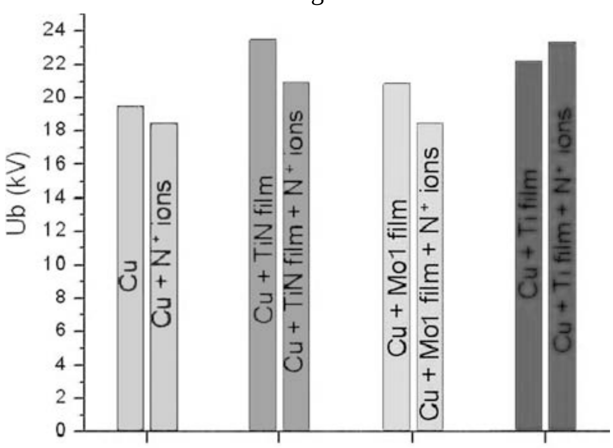

Fig. 9. The results of irradiation of samples with N^{+} ions

The study of samples that were irradiated with N^{+} ions (see Fig. 9) showed a decrease of the breakdown voltage. An exception was a sample with a Ti film on copper. Irradiation of a titanium film with nitrogen ions led to the formation of a thin near-surface layer of titanium nitride, which apparently served as a diffusion barrier, while on all other samples the incorporation of nitrogen increased the probability of breakdown. It is possible that the nitrogen dissolved in the near-surface layers of the material can serve as one of the factors that stimulate the breakdowns.

Implantation of Zr^{2+} ions increased the breakdown voltage of the samples. This is evident from Fig. 10.

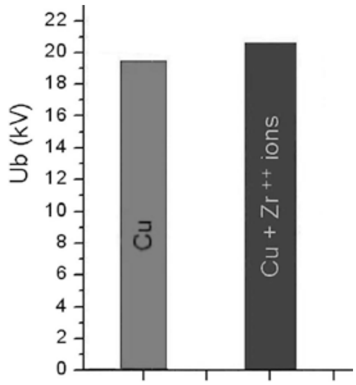

Fig. 10. The results of irradiation of samples with Zr^{2+} ions

Implantation of copper samples with Zr^{2+} ions also leads to an increase of the breakdown voltage. In this case, one can assume an effect for two reasons. On the one hand, as in the case with argon, the displacement of dissolved gases from the surface layers and the smoothing of the surface occurs. On the other hand, the presence on the surface of particles of matter with a higher breakdown voltage increases the resistance of copper samples to breakdowns.

CONCLUSIONS

Plasma and ion-beam modifications of copper samples were performed to study their resistance to high vacuum high-voltage breakdowns.

It has been experimentally shown that the plasma and ion-beam modification of the copper surface leads to an increase of the breakdown voltage from 5 to 35%, depending on the modification method, and reduces the dark current of the anode-cathode. The best result was obtained on copper with TiN coating and additional irradiation with argon ions (E_{Ar2+} = 300 keV).

PLANS

In the next project, it is planned to continue these studies – in particular, to determine in more detail the optimal values of the thickness of the coating, and the dose of irradiation. It is also important to focus on the measurement of dark current, and its dependence on the type of coating and dose of ion irradiation.

It is also important to investigate the dependence of the breakdown voltage and dark current from the distance between the electrodes.

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REFERENCES

1. V.A. Baturin, O.Yu. Karpenko, Ia.V. Profatilova, S.O. Pustovoitov, V.I. Miroshnichenko. The experimental setup for high voltage breakdown studies in the high vacuum // *Problems of Atomic Science and Technology. Series "Plasma Electronics and New Methods of Acceleration"*. 2015, № 4, p. 294-297.

2. Morten Kildemo. New spark-test device for material characterization // *Nuclear Instruments and Methods in Physics Research.* 2004, A 530, p. 596-606.
3. Nicholas C. Shipman // *A thesis submitted to The University of Manchester for the degree of Doctor of Philosophy in the Faculty of Engineering and Physical Sciencec.* CERN-THESIS-2014-27.
4. N.V. Tatarinova // *ZhTF*. 2012, v. 82, iss.11 (in Russian) .
5. O.S. Orlov, I.N. Mmeshkov, A.Yu. Rudakov, A.V. Filipov // *Pisma v EChAYa.* 2014, v. 11, № 5(189), p. 981-985 (in Russian).

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ИССЛЕДОВАНИЕ ОБРАЗЦОВ МЕДИ С ИОННО-ПЛАЗМЕННОЙ ОБРАБОТКОЙ НА ВЫСОКОВОЛЬТНЫЕ ПРОБОИ

В.А. Батурин, А.Ю. Карпенко, В.Е. Сторижко, В.А. Шутько

Представлены результаты исследований влияния ионно-плазменной обработки поверхности меди на её сопротивление сильным вакуумным пробоям. Экспериментально показано, что плазменная и ионно-лучевая модификации поверхности меди приводят к увеличению пробивного напряжения от 5 до 35%, в зависимости от метода модификации, и уменьшению темного тока анодного катода.

ДОСЛІДЖЕННЯ ЗРАЗКІВ МІДІ З ІОННО-ПЛАЗМОВОЮ ОБРОБКОЮ НА ВИСОКОВОЛЬТНІ ПРОБОЇ

В.А. Батурін, О.Ю. Карпенко, В.Ю. Сторіжко, В.О. Шутько

Представлено результати досліджень впливу іонно-плазмової обробки поверхні міді на її стійкість до високо вакуумних пробоїв. Експериментально показано, що плазмова та іонно-променева модифікації поверхні міді призводять до збільшення пробивної напруги від 5 до 35%, в залежності від методу модифікації, та зменшення темного струму анодного катода.