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S.A.Korenev, A.Perry¹

~~PULSED HIGH~~ CURRENT ION BEAM
PROCESSING EQUIPMENT

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¹Ion Surface Modification Technologies Inc., 9965 Carroll Canyon,
San Diego, CA 92131, USA

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INTRODUCTION

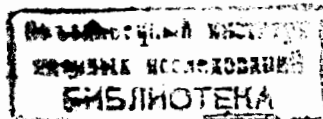
The development of advanced technologies based on the interaction of high current pulsed ion beams with the surfaces of solid materials is very significant at the present time. By way of examples, major development work has been done in the US by the Lawrence Berkeley National Laboratory and at ISM Technologies on MEVVA sources and their applications [1,2], and in the Los Alamos and Sandia National Laboratories on high intensity beams [3,4]. In Russia, extensive work has been done in Tomsk [5-7] and Dubna [8] on intense beam technologies.

A survey of contemporary intense beam technology indicates the need for an ion source of simple and rugged design, and which is also inexpensive for commercial application. Considering, for example, the diode explosion ion source developed in Tomsk [5], this operates with a double pulse power supply the flexibility of is limited by the high voltage generator; the first pulse is negative to create a cathode plasma, and the second is positive to extract the ion beam. For real-world applications, it is necessary to be able to change the operating parameters of the ion beams over a wide range. This means that the voltage generator has to be able to operate under different loads, and the ion diode offers this flexibility. The best power supply available for this purpose is the Arkad'ev-Marx generator which can operate under very different loads. In the present work which is oriented towards technological applications this generator is coupled to an ion source based on explosive ion emission [8].

The production of materials with novel properties is very attractive for research studies, and offers great potential for technological development and industrial application. Although the physics of the interaction processes of these beams with solid surfaces is currently understood only at the level of physical models, the technology of the ion sources themselves is relatively simple and continues to find new applications. One specific area is the deposition of films with concomitant ion beam mixing. A second is the modification of material surfaces by high current, pulsed ion beams. In addition to these two areas, the explosive ion source can be used to generate an intense electron beam. In the present work, the equipment for generating such beams is discussed together with some examples of its applications.

PRINCIPLES OF PULSED BEAM TECHNOLOGY

A pulsed electron beam and a pulsed ion beam are both sources of pulsed energy. This energy is transmitted to solid materials during an irradiation treatment. For example, when operating with a beam current $I = 200$ A, a pulse length $t = 250$ ns, and kinetic energy $E = 500$ keV (i.e. the accelerating voltage U is set at 500 kV), the energy transmitted in one pulse $W = I.U.t (= 200 \times 5 \times 10^5 \times 250 \times 10^{-9}) = 25$ J. This is a very large amount of energy per pulse which is to be dissipated at the point of entry into the substrate and over the limited depth of penetration determined by the kinetic energy. This energy is sufficient to cause processes to occur in the solid state which are very far from equilibrium.



We can summarise the specific effects of either type of beam, electron or ion, as follows. In the case of a *pulsed electron beam*, electrons are small particles with low ionisation losses so that their depth of penetration in to many materials is in the range 10 - 500 μm for kinetic energies in the range 100 - 1000 keV. Adiabatic heating occurs in the substrate resulting in effective electron beam mixing. In the case of a pulsed ion beam, the ions are very large compared with electrons and there are large ionisation losses. The result is a comparatively low depth of penetration, e.g. for kinetic energies in the range 100 - 1000 keV, the depth of penetration is only about 0.05 - 1.0 μm . Adiabatic heating processes occur at these shallow depths and result in the synthesis of non-equilibrium components, ion beam mixing, ion irradiation damage, and other long distance effects. A comparison of the properties shows clearly that each type of beam can be used for different applications depending on the settings of the beam parameters.

The pulse length t of the electron beam needed for rapid thermal processing, especially for compound materials where stable stoichiometry has to be maintained, must satisfy the condition:

$$t \ll t_{(p)} = 2h^2\rho c / \lambda \quad (1)$$

where: $t_{(p)}$ is the heating constant of the sample, h is the depth of penetration, ρ , c , and λ are the density, heat capacity and heat conductivity of the irradiated materials. In this case, the temperature profile is a step function in the irradiated sample which means that the energy released by the electron beam occurs only over the depth of penetration defined by the electron kinetic energy. The temperature T in an irradiated sample over the electron penetration depth can be calculated from the equation:

$$T = 2 W t / h \rho S \quad (2)$$

where: W is the electron beam power density, and S is the irradiated area. For pulsed ion beams, the situation is different and the sample heating has to be calculated from the equation:

$$T = 2 W ((t / (\lambda \rho c))^{0.5} \quad (3)$$

Theoretical calculations and experimental results have confirmed that the parameters of pulsed electron and ion beams can be set so as to use the pulsed energy for different thermally-activated processes in the surface modification of materials.

EXPLOSIVE ION EMISSION AND FILM DEPOSITION WITH ION BEAM MIXING

Detailed discussions of the reduction of pulsed explosive emission to practice have been given elsewhere [7-10], so only the main principles are considered here. Referring to the schematic shown in Fig. 1a, a positive voltage pulse is applied to the anode (termed the

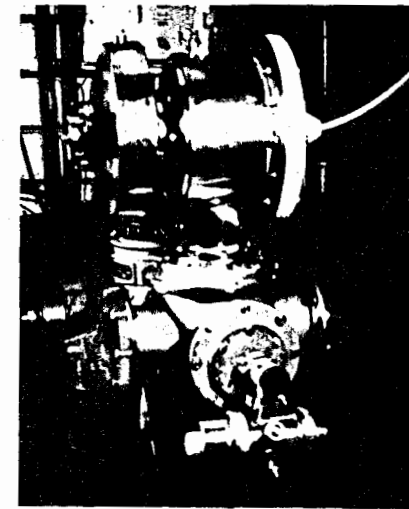
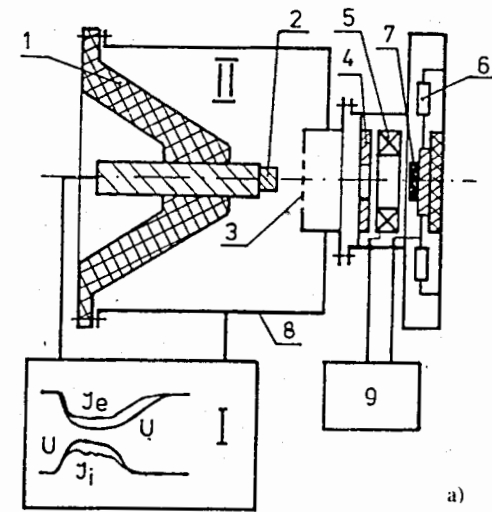


Fig. 1.
a) block-diagram of an electron-ion accelerator mounted onto a small sample chamber showing: 1, the high voltage insulator, 2, the anode (plasma initiator), 3, the cathode (extractor grid), 4 a collimator, 5, a current transformer (to measure the beam current), 6, the film deposited onto a substrate in a small holder, 7, the ion source vacuum chamber, 8, connector for, 9, an oscilloscope
.b) a photograph of an electron-ion source fitted with a small sample holder.

plasma initiator) and a plasma is formed on its surface. Initially there is a condition of autoemission from the heating by the ion current of points, local micro-heterogeneities, on the surface of the anode, and electron emission from the cathode. The atoms in the vapour are ionised and form an anode plasma so that an ion beam can be extracted from it by applying a bias from the cathode, and thus forms a stable vacuum discharge. This type of ion emission is termed explosive ion emission. For the operation of a planar ion source using explosive ion emission, the intensity of the electric field E in the diode must exceed the threshold voltage E_{thr} needed for forming the anode plasma:

$$E = KU / d > E_{thr} \approx 10^7 \text{ V cm}^{-1} \quad (4)$$

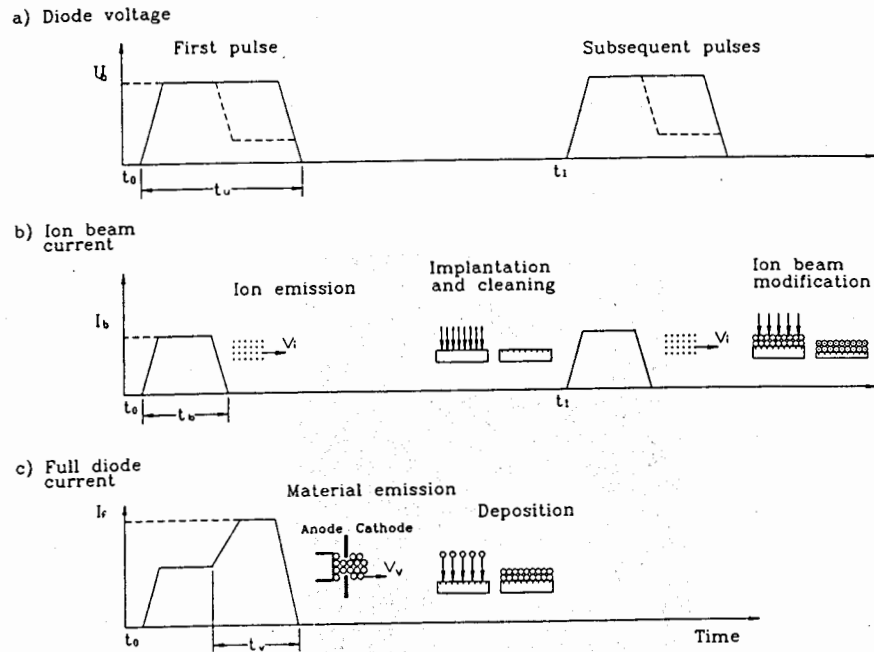


Fig. 2. Time diagram of the processes occurring in the ion diode.

where: U is the voltage applied to the diode; K is the amplification factor determined by the initiator geometry, and d is the distance between anode and cathode. The extraction of the ion beam from the anode plasma is accompanied by an intense emission of anode material, this material is emitted in the form of clusters and macro-particles. These can be condensed out to form a film on substrates located behind the cathode extractor grid [11]. As detailed below, the deposition of films is carried out using the explosive emission of material from the plasma initiator, and then using the extracted beam for ion beam mixing in the next pulse.

The diode can be operated in two regimes:

1. maintaining the voltage pulse length less than the time required for the diode to be short-circuited by the anode plasma, and,
2. lengthening the duration of the voltage pulse so that it exceeds the time for short-circuiting by the anode plasma.

In the first regime, the beams produced are primarily ions with only a limited amount of material emitted by the anode plasma initiator. In the second, there is a far greater amount of material emitted, and films of plasma initiator material can be deposited accompanied by ion beam mixing, as noted above. In this second regime, the significance of the ion beams is clearly far less than in the first, but still brings with it the inherent advantages of ion beam mixing for the deposition of dense, hard films onto the substrate. The operating sequence of the source can be understood from the time diagram shown schematically in Figs. 2a-c which illustrate the various emission processes. In the very first pulse beginning at t_0 , the anode plasma forms (on the plasma initiator), and the diode voltage U_b (Fig. 2a) remains constant over the time t_b taken for the diode to short circuit. During this time (i.e. in the regime 1 noted above) ions are formed from the anode with some erosion of anode material, and an ion current I_b can be extracted from the plasma by the cathode (Fig. 2b) at a velocity V_i . These first ions pass through the cathode extractor grid, and can implant and ion-clean the substrates located behind the grid. This cleaning by the first burst of ion enhances the adhesion of the coating. After the diode short-circuits (Fig. 2c) the voltage drops and the current increases to the full diode level I_f , accompanied by the explosive emission of material from the anode with a velocity V_v (regime 2 noted above). This component of the pulse length t_v can be set, giving a total pulse length of $t_u = t_b + t_v$. The emitted material

passes through the cathode screen and coats the substrates located behind it. The second, and subsequent pulses, starting at t_1 continue the process with the single difference that the ion beam now irradiates the deposited film, modifying its properties. The adjustment of the pulse lengths t_b and t_v gives control of the ion/atom ratio during the film deposition process.

EQUIPMENT

A schematic of the electron-ion source is given in Fig. 1a and it is illustrated in Fig. 1b. As shown, the ion diode is very simple in design, and comprises a stainless steel vacuum chamber, a high voltage insulator made from Capralon on which is mounted the anode (plasma initiator), a cathode (extractor grid), and various diagnostic devices for measuring the ion beam and total currents (integrating Rogovski transformer and Faraday cup), and the pulsed voltage on the anode (a high resistance divider). The complete ion source facility consists of the ion diode coupled to an Arkad'ev-Marx type high voltage generator enabling it to operate as an explosive ion or electron emission source. It can be mounted onto any vacuum chamber containing the substrates to be surface treated. The size of the chamber depends on the particular application, the example shown in Fig. 2 is for the intense treatment of single samples. The base pressure in the ion source and sample deposition chamber is in the 10^{-5} Torr range.

Experimental studies of the ion source in the different regimes have shown the advantages in process flexibility of using a generator based on energy storage in a capacitor, such as the Arkad'ev-Marx generator. This generator can work under a wide range of the loads, where the load in this case is the pulsed explosive emission electron-ion diode. The main operating parameters of this generator are: voltage 50 - 700 kV, pulse duration 300 ns., ion current 1 - 300 A, stored energy 100 - 1000 J, and repetition rate 0.1 - 10 Hz.

The ion source can be built in different geometrical variants. A planar ion source is used for the production and modification of simple planar style films. A coaxial type of ion source is designed for the preparation of films, with ion mixing, on the inside and outside of tubes. Other geometries such as a conic style can be designed for use in specific tasks. The generator can be used in a very simple mode, e.g., as a single step device for intense electron beam irradiation.

The emission characteristics in the ion source mode are presented in Fig. 3a for a variety of materials showing the very high current densities which can be produced and used for the surface modification of materials. Measurements of the emission characteristics of the ion diode have shown that the ion beam current corresponds to the Child-Langmuir law.

The mode of operation can be changed from ion beam to electron beam. This is done by changing the polarity of the voltage on the output side of the pulsed high voltage generator. In this case, the operating beam envelope of explosive electron emission is 1 - 3 kA, and pulse lengths of 0.3 μ s can be achieved. This is a powerful option because it allows the building of a single piece of equipment for universal

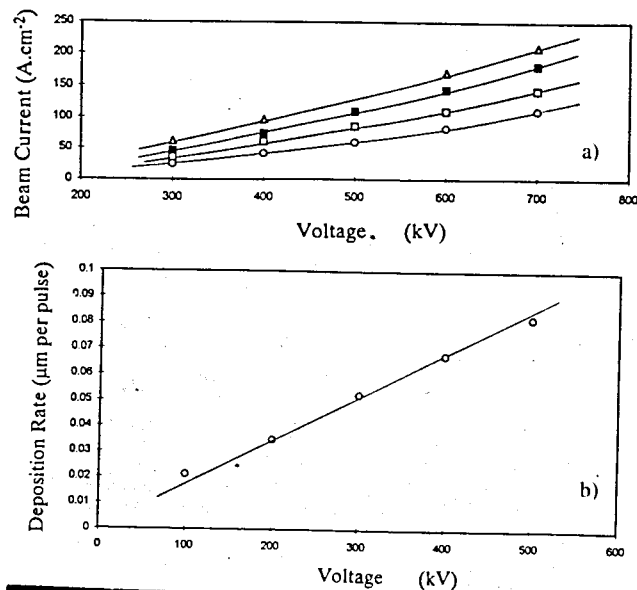


Fig. 3. a) Emission characteristics of the ion diode for typical materials showing the high current densities which can be reached. In these studies, the distances between anode and cathode, and anode and substrate were 7 mm and 30 mm, respectively; the pulse length was 300 ns. The data are for ions of copper, aluminum, carbon and beryllium, represented by round, square, filled square, and triangular symbols, respectively. b) Dependence of the rate of deposition on the voltage applied to the ion diode, geometry as in c), of rhenium. c) The condition of a rhenium anode after approximately 1000 pulses.



i)



ii)



iii)

Fig. 4. Titanium deposited onto: silicon, accelerating voltage 150 keV, ion beam current density about 10 A.cm^{-2} , pulse length 300 ns, for (i) 5, (ii) 20, and (iii) 50 pulses.

application in the deposition of films and for surface modification of films and bulk materials using electron or ion beam irradiation. Compared with other intense ion-electron sources, this equipment is very economic, costing in the range k\$ 10 - 100, depending on the operating parameters, and is compact, weighing only 50 - 300 kg with maximum dimensions of about 2m x 3m x 4m.

This electron-ion source is very flexible. It allows the deposition of many different types of film on different substrates at different temperatures, and the post-treatment with ion or electron beams. The rate of film deposition depends on the parameters of the discharge circuit high voltage generator, the plasma source, and the ion beam. Ion beams can be selected from amongst all metals and conducting materials, and multi-component ion beams can also be produced within single pulses from a multi-component anode plasma initiator.

The rate of film deposition is very high as indicated in Fig. 3b for a rhenium anode. Currently, the main limitation on the stable operation (about 1-2 hours before the anode has to be changed) is imposed by the short lifetime of the anode plasma initiator itself. Intensive loss of anode material by cluster and macro-particle emission leads to a major change in the anode surface geometry. An example of this shown in Fig. 3c where the condition of a (initially conical) rhenium anode plasma initiator is shown after approximately 1000 pulses. One solution is the use of a multi-emitter anode plasma initiator directed towards the cathode.

APPLICATIONS

The current directions of the industrial applications include the deposition of films onto polymer substrates, the production of different targets for nuclear experiments, e.g. rhenium on titanium foil, and the deposition of hard, stable coatings for optical devices. As noted above, the electron-ion source can also be used for surface modification of different materials: semiconductors, high temperature superconductors, metals and the post treatment of films deposited with the device.

Semiconductors

PtSi-Si Schottky barriers have been annealed using pulsed carbon ion beams, which allows nuclear detectors to be made with good resolution. The detector resolution at full width at half maximum (FWHM) was determined using 5.5 MeV pulse-height plutonium alpha particles. The FWHM achieved was better than 22 keV [12].

This equipment offers the possibility of preparing different semiconductor devices via plasma synthesis. Examples are found in opto-electronic integrating devices because the ion diode offers the possibilities of depositing metal films on the semiconductors with concurrent ion beam mixing, followed by electron or ion beam annealing, thus permitting synthesis of different structures.

Metal Films

As indicated above, one of the main advantages of the ion diode is that any metallic or conducting film can be deposited onto any substrate. For example, titanium films can be deposited readily accompanied by ion beam mixing, as shown in Figs. 4a



Fig.5 a)

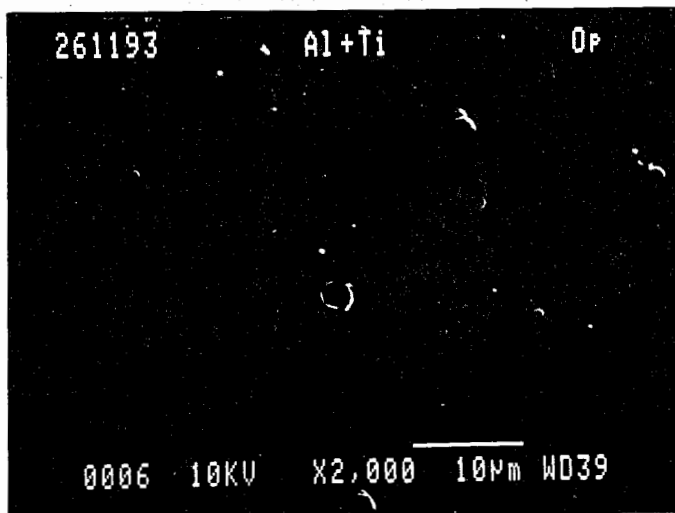


Fig.5 b)

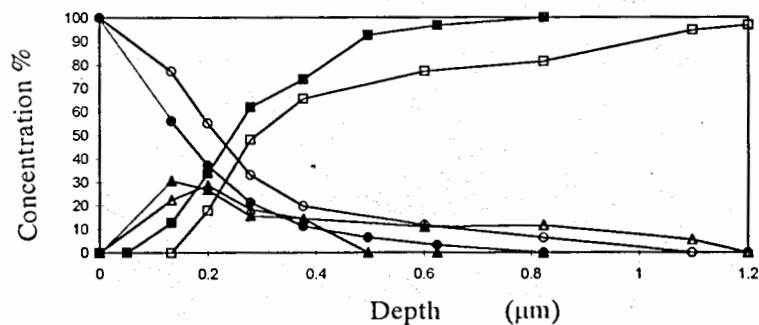


Fig.5 c)

Fig. 5. a) Aluminum deposited onto titanium without short circuiting of the ion diode (i.e., in regime 1). The deposition conditions were: accelerating voltage 250 keV, ion beam current density about 10 A.cm⁻², deposition time 5s, with pulse length and frequency 300 ns and 1 Hz.

b) The film deposited in a) after a single electron beam pulse: accelerating voltage 250 keV, electron current density 700 A.cm⁻², pulse length about 300 ns.

c) Concentration profiles (RBS and SIMS) of the samples shown in a) and b), with the elements titanium, aluminum, and oxygen represented by round, square, and triangular symbols, respectively; the open symbols are the condition as-deposited and the filled symbols are after the electron beam pulse.

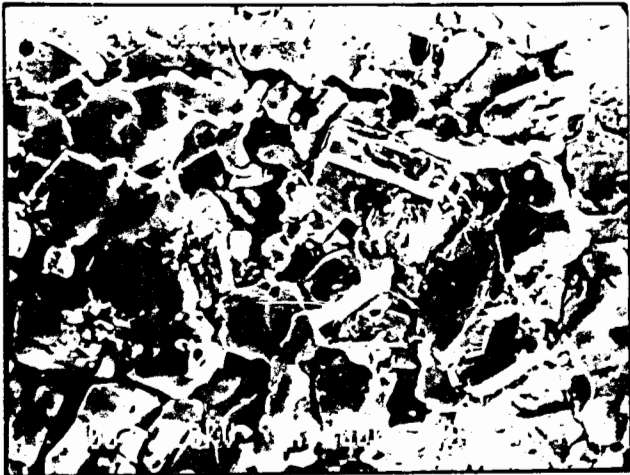


Fig.6 a)

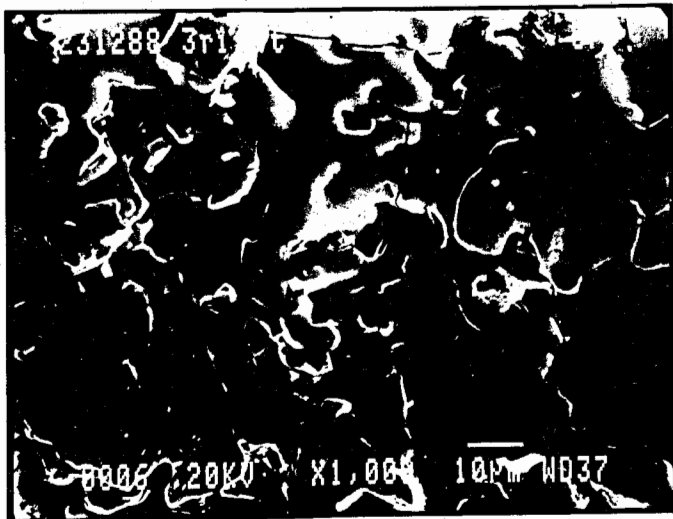


Fig.6 b)

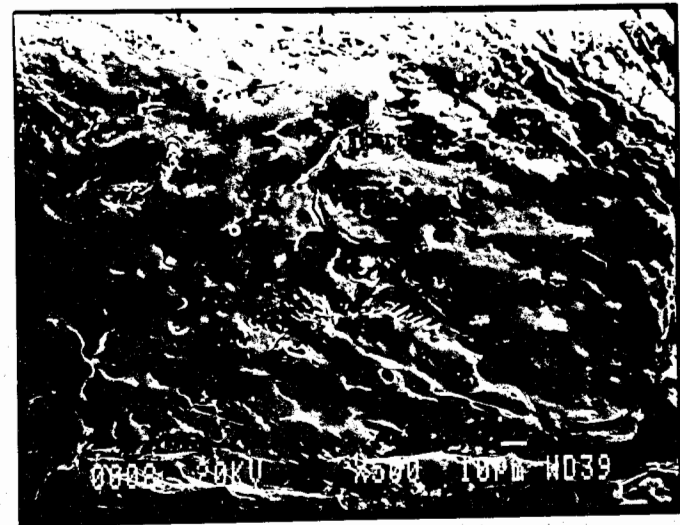


Fig.6 c)

Fig. 6. A Y-Ba-Cu-O superconductors: a) in the as-received condition, b) after ten pulses of electron bombardment with an accelerating voltage 200 keV, current density 1000 A.cm⁻², pulse length about 300 ns, and, c) a Bi-Ca-Sr-Cu-O high temperature superconductor, after pulsed electron beam remelting with ten electron pulses: accelerating voltage 200 keV, electron current density 1000 A.cm⁻², pulse length about 300 ns.

and b for different substrates. Also evident in the micrographs are the undesirable macro-particles the number of which increases with film thickness (i.e. the number of pulses) as shown in Fig. 4b. The source can be used to electron beam post-treat and modify these coatings. This is illustrated here by taking a coating of titanium deposited in 5s onto aluminium (Fig. 5a) where the macro-particles are very evident, and fusing these with a single, intense electron beam pulse (Fig. 5b). A further example is the deposition of aluminium on titanium, materials which are mutually insoluble. A rapid deposition by explosive ion emission brought the surface temperature to 1365 °C, producing the well-known pulsed melting effect [3,4] which, with ion beam mixing, has caused extensive non-equilibrium surface alloying as shown by the concentration profiles given in Fig. 5c (open symbols). The effect of a post deposition electron pulse (Fig. 5c - filled symbols) is to narrow the composition profile and make it sharper, and to remove the oxygen from depths beyond 0.5µm.

This process has been used in the improvement of the thin beryllium or titanium foil vacuum chamber windows used in electron beam accelerators. The deposition of an aluminium film onto the beryllium foil, with ion beam mixing, increased the foil life-time by a factor of 5 - 7 times compared with the standard foil used in these devices.

High Temperature Superconductors

The intense pulsed electron beam option can be used to improve the properties of high temperature superconductors. The surface melting of superconductors such as Y-Ba-Cu-O, and Bi-Ca-Sr-Cu-O causes an increase in the critical current by a factor 2 - 10 times [13,14]. The parallel changes in the surface topography can be seen in Figs. 6a-c. In Fig. 6a the surface of an Y-Ba-Cu-O superconductor is shown in the as-received condition, and an electron beam fused surface is shown in Fig. 6b. A similar surface of a Bi-Ca-Sr-Cu-O superconductor is in Fig. 6c. At the present time there is some cracking in the film after the treatment which indicates the need for process optimisation. The specificity of the treatment should be noted with respect to the discussion of the roles of electron and ion beams in surface treatment, irradiation with intense carbon ion beams has no effect on the superconducting properties nor the surface topography.

FUTURE DEVELOPMENT

The current directions of the development of this equipment are as follows. First, a magnetic filter will be used to reduce the size and occurrence of the macro-particles produced during film deposition. This is particularly significant when working with films for semiconductor device applications. Second, it is necessary to increase the size of the substrates which can be treated and also the size and design of the anode plasma initiators. Finally, it is necessary to increase the time-life of the anode plasma initiator, and control the material emission characteristics.

CONCLUSIONS

The explosive electron and ion source, based on the ion diode powered by an Arkadev-Marx generator, delivers very intense beams with currents in the range 1-200 A. The control variables can be set in the ranges: voltage 100 - 700 kV, pulse length typically 0.3 µs, depending on the ion chosen. In the present work we have demonstrated that it can be used very effectively in the surface treatment of bulk materials, for the deposition of films with concomitant ion beam treatment in the ion emission mode, and for electron beam irradiation and surface fusion in the electron emission mode.

REFERENCES

1. I.G. Brown, J. Vac. Sci. Technol. A, 11 (1993), 1480.
2. J.R. Treglio, A.J. Perry and R.J. Stinner, Surf. Coat. Technol., 65 (1994), 144.
3. R.W. Stinnett, R.G. Buchheit, F.A. Greulich, C.R. Hills, A.C. Kilgo, D.C. McIntyre, J.B. Greenly, M.O. Thompson, G.P. Johnston and R.J. Rej, Mat. Res. Soc. Symp. Proc., 316 (1994), 521.
4. D.J. Rej, R.R. Bartsch, H.A. Davis, R.J. Faehl, J.B. Greenly and W.J. Waganaar, Rev. Sci. Instr., 84 (1993), 2753.
5. G.E. Remnev and V.A. Shulov, Proc. 9th Int. Conf. BEAMS'92, Washington, D.C., 5/25, 1992.
6. A. Didenko, A. Ligachev and I. Kurakin, Effects of particles beams irradiation on metals and alloys surface, Moscow, Energoatomizdat, 1987.
7. Electron Beams in Technology, Editor G. Mesyats, Novosibirsk, Science, 1986.
8. S.A. Korenev, Nucl. Instr. & Meth. Phys. Res., B80/81 (1993), 242.
9. S.A. Korenev, Proc. 3rd Int. Conf. "Energy Pulse and Particle Beam Modification of Materials" (EPM'89), Dresden, Germany, 1989, p. 448.
10. S. Korenev, Rev. Sci. Instr., April (1994), p. 147.
11. S. Korenev, Preprint JINR no. 13-89-615, JINR, Dubna, 1989.
12. L. Hrubchin, A. Guldan, T. Besse, S. Korenev, 1992 Annual Report of Electrical Engineering Institute, Bratislava, Slovakia, p. 147
13. S. Korenev, Nucl. Instr. & Meth. Phys. Res, B65 (1992), p. 550.
14. S. Korenev, V. Sikolenko and S. Shtan'ko, Proc. 2nd All- Union (USSR) Conference on high temperature superconductors, Kiev, 1989, Vol. 2, p. 214.

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