Energetic deposition of niobium thin film by ecr-plasma

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An ECR-plasma reactor has been built to do energetic ion deposition of refractory metals in vacuum. The system uses an E-beam gun to create refractory metal flux. The neutral metal flux feeds into a microwave resonator and forms pure metal plasma created by Electron Cyclotron Resonance (ECR). The metal ions are extracted to a biased substrate for direct deposition. A retarding field energy analyzer is developed and used to measure the kinetic energy of metal ions at the substrate location. A high quality niobium thin film is obtained through this deposition system. The niobium film exhibits an excellent superconducting transition. The niobium ion energy distribution has been measured. The niobium ion at the substrate location has a median kinetic energy of 64-eV with an energy spread of 20-eV under certain plasma conditions.

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INTRODUCTION

For refractory metal thin film deposition, increasing the substrate temperature is not an option to achieve greater surface adatom mobility[1] for many low melting point substrates. Many different methods have been used to achieve film growth with higher adatom mobility. The ion assisted deposition process is proved to be effective to suppress columnar film growth.[2] Biased magnetron sputtering[3]-[4], ionized magnetron sputtering[5], vacuum arc deposition[6] and energetic cluster deposition[7-8] give various degrees of higher adatom mobility.

Metal ion deposition by an ECR plasma has been successful in previous work with copper evaporated by filament heating[9], the present work demonstrates that the same mechanism can be applied to refractory metal vapor created by E-beam gun evaporation provided that a high enough evaporation rate is achieved to obtain an ECR condition. The energetic condensation of refractory metal film achieved through ECR plasma is believed superior to other methods mentioned above in that the energy spread is low, and the deposition is in high vacuum or ultra high vacuum.

In this report, the niobium plasma is realized by applying ECR to the E-beam evaporated niobium flux. A simple retarding field analyzer is developed and calibrated using a commercial ion gun. The niobium ion energy distribution is measured at the substrate location under different bias voltages. The deposition rate at the substrate is around 4 Å/s.

The vacuum pressure during the niobium deposition is 1.2×10^{-6} torr typically. The niobium thin film deposited with higher impact ion energy is expected to have better superconducting quality.

II. THE ENERGETIC DEPOSITION SYSTEM

The energetic deposition system is illustrated in figure 1. The whole ECR plasma reactor is inside a vacuum chamber. The vacuum chamber is evacuated through two stages. First stage pumping reaches 400 milli-torr using a rotary roughing pump. The roughing line is filtered by a reusable Foreline-trap. The second stage pumping reaches UHV level using a cryopump that has 9,000 liters per second pumping speed. A typical mid 10^{-7} torr is achieved after twenty-four hour pumping. In about four weeks, the chamber can reach the base pressure at 8×10^{-9} Torr. Water vapor and hydrogen dominate the residual gas content.

A thermionic type E-beam gun is used to create niobium evaporation. The E-beam gun has a power level up to 10 kW; and the evaporation rate for niobium can be as high as 150 Å/s at 12-inch distance.

A circular waveguide resonator operating in the TE_{112} mode provides RF field. The mode has its electric field roughly vertical, thus perpendicular to magnetic field lines running horizontally. One opening at the waveguide bottom allows the neutral niobium flux to

travel into the waveguide. Two slots are added to the opening hole to reduce the RF radiation out of the waveguide resonator. Another opening hole on top allows the thickness monitor to monitor the neutral flux rate with or without plasma. The circular waveguide length is optimized so that the standing wave maximum is located around the path of the neutral flux. The electric field pattern at the center cross section of the waveguide is shown in figure 2.

A simple ceramic window is installed to transfer up to 1.5kW RF power to ECR chamber while maintaining high vacuum. The window is designed in a way that the ceramic can be easily replaced.

A cheap 2.45 GHz RF generator is available as a commercial product and capable of providing 1.5 kW total microwave power. When the plasma is off, all the reflected RF power is dumped to a water-cooled RF absorber through a circulator. A two channel RF power meter is connected through a dual directional coupler to monitor the input RF power and the reflected RF power.

Heavy gauge copper tubing installed inside the vacuum chamber provides the magnetic field needed to achieve an ECR condition. Current around 1,500 amperes is used to generate a magnetic field around 875 gauss. The field profile is shown in figure 3.

There are eight iron bars distributed outside of coils to reduce the stray magnetic field seen by the E-beam hearth underneath the solenoid.

III. ION ENERGY MEASURED BY RETARDING FIELD ANALYZER

To characterize the energy distribution of the ion beam to the substrate, a retarding field beam analyzer is developed. The size of the analyzer is about 100 times larger than that of the original design.[10] The beam analyzer incorporates three grids for retarding field control, one entrance grid and one collector. All of these are enclosed inside the substrate holder.

The analyzer is tested using an ion gun in the vacuum chamber for ion beams with different energies. The results are shown in figure 4. The red dotted curve is derived from the I-V curve, which is proportional to the number of ions per second with energy in volts denoted on the x-axis. The result is consistent with the ion beam specifications from the ion gun manufacturer. The ion gun uses Argon as its working gas; the pressure of Argon inside the chamber is 1.5×10^{-4} Torr. During the ion gun beam test, the analyzer is at ground potential.

During the niobium plasma measurement, the ion analyzer is floated inside the plasma at certain bias voltages through a separate power supply. It is found that the ion velocities didn't follow the Maxwellian distribution at the substrate position as shown in figure 5-a.

As shown in fig. 5-b, when the bias voltage is added, the ions have on average the added kinetic energy. The niobium ion energy for no bias voltage is 64 eV with energy spread about 20eV(FWHM). When the bias voltage is added, the energy spread increased. The increased spread is likely caused by the ions traveling along different paths with various distances from the grid wires, which has an increasingly large effect as the voltage increases electric field strength.

IV. NIOBIUM THIN FILM DEPOSITION

The vacuum is around 1.0×10^{-7} torr before the deposition and around 1.0×10^{-6} torr during the deposition in the chamber. In the ECR reaction chamber, it is expected to be much better due to the getter pumping by the niobium film on the wall, but is not measured in this location. During niobium thin film deposition, the residual gas content is dominated by hydrogen, which likely comes from the E-beam gun hearth. The RF power is kept roughly at 250 watts input during deposition. The substrate is kept at -33V bias for a sample.

The copper substrate is electropolished by a phosphoric acid and butanol composition in a 50-50 ratio. A 200-micron layer is removed during a 3-4 hour electropolishing.

The surface flatness of the niobium thin film is dominated by the copper substrate surface. The transition temperature can be obtained by measuring the shielding capability by AC induction. The superconducting transition curve is shown in figure 6.

V. CONCLUSION

For a refractory metal such as niobium, metal ionization can be achieved by applying an ECR-plasma process to the E-beam gun evaporated metal flux. Increased deposition energy can be achieved by increasing substrate bias voltage. For future development, an extraction grid can be used to reduce the energy spread seen at the substrate location if desirable. For niobium material or other refractory metals, the increased deposition energy and the high vacuum deposition condition may produce much better thin film quality than has been achieved by other methods.

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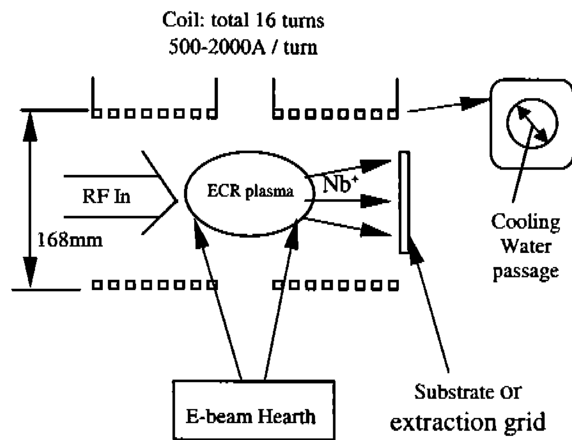

Fig. 1 Illustration of the energetic condensation by ECR in vacuum.

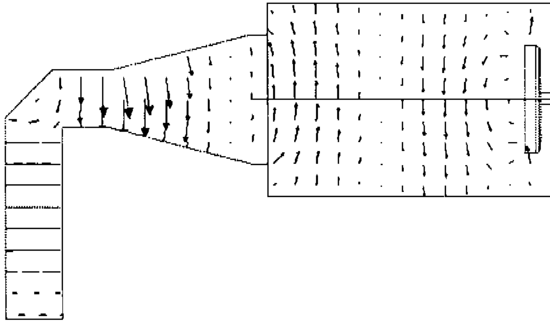

Fig. 2 The electric field distribution inside the circular waveguide. The field is for the standing wave.

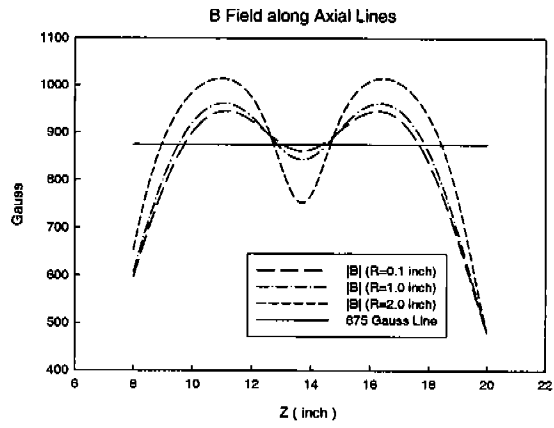

Fig. 3 The magnetic field contour.

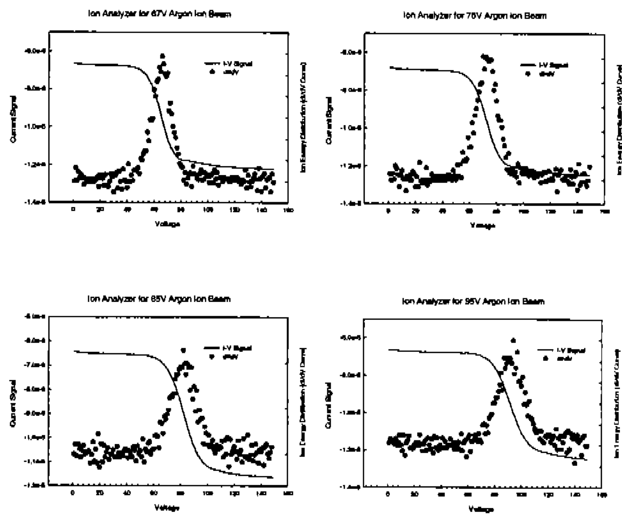

Fig. 4 The ion energy analyzer measurement result for different ion energy beam.

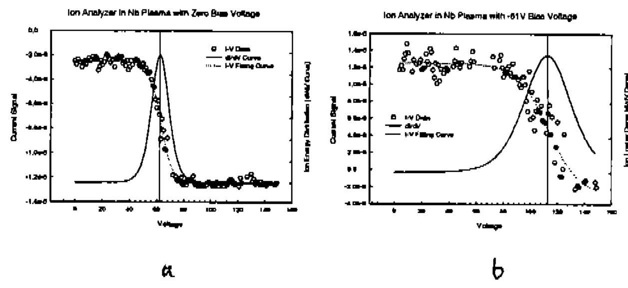

Fig. 5 The ion energy analyzer measurement result for niobium ions at the substrate without biased voltage (a) and with -51V bias (b).

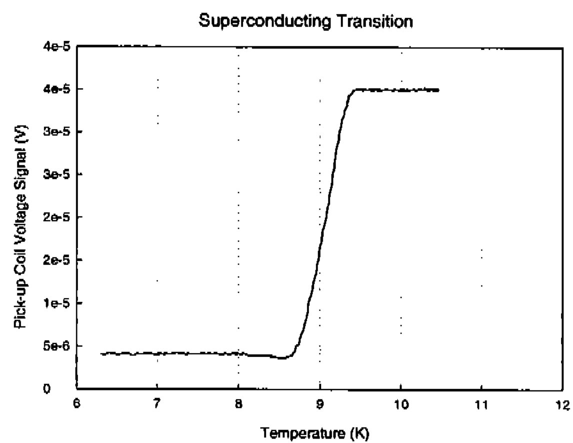

Fig. 6 The niobium thin film superconducting transition curve measured by AC induction.

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