ENERGETIC DEPOSITION IN VACUUM

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Abstract

In hoping to improve Niobium deposition on Copper cavity, a vacuum deposition system has been built to test the idea of Nb energetic condensation on copper substrate. The system directly uses microwave power to create the pure Nb plasma, which can be used to extract energetic Nb ion flux to do direct deposition on copper substrate. In this paper, we briefly describe the system, discuss the potential benefit of this technique and report the initial result of Nb plasma creation and Niobium thin film deposition.

1 INTRODUCTION

Two major accelerator applications have adopted the thin film technology. LEP2 superconducting cavities have applied the thin film technology successfully [1], and Low-Beta superconducting cavity for heavy ion accelerator is also trying to use the Nb/Cu technique, except that the copper's mechanical strength is a major concern for further deployment [2,3]. While the advantages of thin film cavity make it very attractive to the future accelerator applications, the Nb/Cu cavity still has certain problem (Serious Q-drop) needs to be solved.

Currently, magnetron sputtering film deposition is the main technique to make Nb/Cu cavities. There are different processes were being tried to improve the niobium thin film. These are laser annealing [4], DC-post magnetron sputtering deposition [5], and vacuum arc deposition [6]. The other approaches include Wuppertal University's Nb3Sn thin film on niobium cavity [7,24,25], sputter coated NbTiN cavity at Saclay [8] and Nb/Cu clad cavities by Saito etc.[9]

In Jefferson Lab, we have built an energetic vacuum deposition system to explore if a better film can be made and in what extent this technique can improve the Nb/Cu cavity performance. A fast deposition rate of energetic niobium has been achieved in vacuum recently. An extensive work will be carried out to establish the correlation between deposition parameter, film structure and physical property of the niobium thin film.

2 THE MAGNETRON SPUTTERING

The sputtering coating of Niobium on copper is explored extensively at CERN, there is lot of interesting facts on that particular technique.

2.1 Q-degradation

When the copper substrate is prepared by the spinning method, and electro-polished, niobium thin film cavity made by krypton sputter-coating shows accelerating gradient field as high as 22MV/m [10]. It is fair to say, the Niobium thin film cavity is reaching its goal to replace the bulk Niobium cavity. However, the physics of Niobium thin film still needs to be explored further. Specially, whether the Q-degradation is caused solely by non-cleanliness and inferior substrate remains a question.

2.2 Cause of anomalous RF loss

One possible explanation for anomalous RF loss is hypothesized vortex inside the superconductor [11], which could be caused by oxygen, hydrogen, etc.

Malev and Weisser analyzed the data available from CERN's Nb thin film and revealed that the stimulated desorption mainly contributes the oxygen and carbon oxide partial pressure to 10e-7 mbar during the argon discharge sputtering [12]. TEM analysis revealed that the thin film is dominated by the rich density of grain boundary and intrinsic defects [13].

One interesting proposal by Knobloch states that the impurity between grain boundaries and local magnetic field enhancement contributes the Q-slope after BCP process of bulk Nb cavity, while Electropolishing process greatly improved the Q-slope [14]. The proposed mechanism could help explain the Q-degradation for Nb/Cu cavity.

2.3 The coating temperature effect in sputtering

The substrate temperature is one of the most interesting parameters during argon discharge sputtering process. An optimum range of sputtering temperature as 150°C-175°C is reported for better Rs [15]. There is no other information like the film morphology available for further discussion. Also at CERN, Orlandi group has reported the 550°C leads to a quite large grain size as 0.2μm and a RRR as 35. From the report, we also know the deposition rate was higher than the lower temperature case.

2.4 The rare gas effect

From the early morphology studies, the film structure is greatly related with the deposition rate and the adatom surface mobility [17, 18]. By increasing the incident atom energy and incident flux, we can see the increased substrate temperature, also the adatom surface mobility, which leads to the larger grain size. Based on this and the

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*Work performed under DOE Contract #DEAC0584ER40150

findings in [16], Schucan, etc. have tried to use Ar/He mixture to achieve that [19]. Unfortunately the rare gas trapping reduces the grain size and affects the grain growth. The investigation of the rare gas trapping of Ar, Ke and Xe is reported in [20].

Following the discussions from above, we can get conclusion that sputtering film technique has following limitations:

- The working gas is trapped in film, it may cause intrinsic defects inside of the grain.
- The impurities of working gas is no good to thin film.
- Deposition energy is low or hard to control, which does not help to influence the film microstructure.

3 THE ENERGETIC CONDENSATION

Before making this proposed process, we'll take a look at the film structure and the possible deposition method.

Movchan-Demchishin Zone Model is well verified by J.A. Thornton's intensive study [17]. From Thornton's work, we notice that the low argon pressure, high substrate temperature helps to enlarge the column size of thin film. The ion bombardment also assisted the improvement of film structure. All of this leads us to believe if the depositing atom gains more mobility, as it shows in the Fig. 1, it will diffuse quickly in the surface, thus less prone to form the columnar structure. Harberland etc. has studied energetic cluster deposition. A 1,000 atom cluster beam with 5 keV kinetic energy has created near epitaxial thin film growth in room temperature substrate [21].

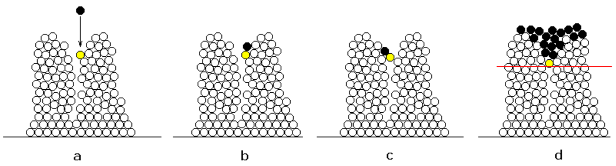

Fig. 1 The illustration of the surface atom nucleation process to show the columnar forming and atom mobility relation.
a), b) the low kinetic energy atom travels and sticks to the surface atoms.
c), d) energetic atom impacts on existing surface, diffuses around.

Beside the heavy energized cluster, three processes can be explored to achieve greater atom's surface mobility.

- Energized metal ion deposition
- Ion assisted.
- High energy Sputtering.

Also without working gas like Argon would be a great plus.

The proposed process is similar to the work carried by Holber [22] and his colleagues from IBM. Basic idea is to use energy controllable metal ions to do direct film deposition, a process usually called energetic condensation. In Holber's device, copper was the only material studied, and worked very well for trench filling in semiconductor industry. The same process using niobium is the best option for a successful thin film compared with the other techniques.

The main advantages are:

1. No working gas like Argon.
2. High Vacuum means reduced impurities.
3. Controllable deposition energy. (Possible to help control the crystal structure.)
4. No macro particles.

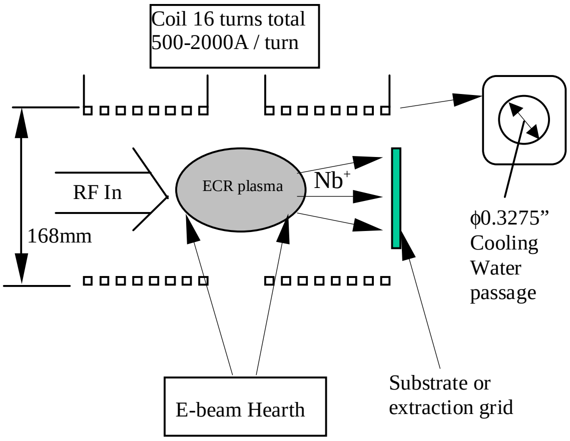

Fig. 2 The illustration of the energetic condensation by ECR in vacuum.

The niobium ions would be produced by Electron-Cyclotron-Resonance(ECR) inside a waveguide resonator. Neutral Niobium flux is provided by E-beam evaporation. The system is illustrated in Fig. 2.

4 THE E-BEAM ECR DEPOSITION SYSTEM

4.1 The RF system design

A cheap 2.45 GHz RF generator is available as commercial product and capable to provide 1.5 kW total microwave power. A diagram is showing the simple circuit of this ECR-RF system.

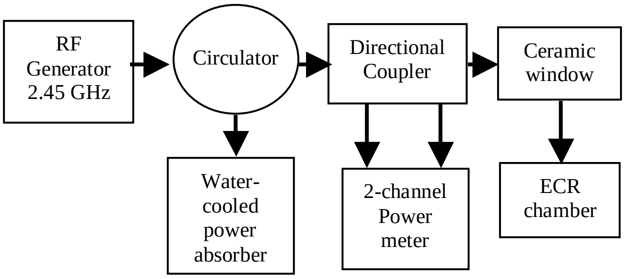

Fig. 3 ECR-RF diagram

A simple window is installed to transfer up to 1.5kw RF power to ECR chamber while maintaining high vacuum. The window is designed the way that the ceramic can be easily replaced. The window position is not the standing wave zero position. Nevertheless, this narrow band RF window is capable to transfer 1.5kW power except one time the ceramic window was destroyed by excessive heating from copper scratches.

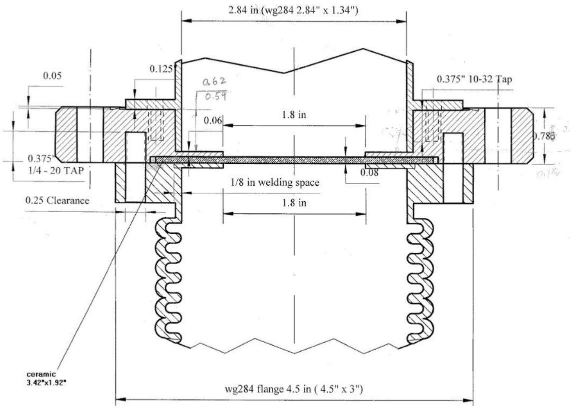

Fig. 4 Ceramic window installation

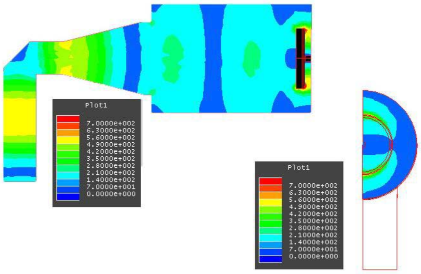

Fig. 5 The electric field distribution inside the circular waveguide.
The field is for the standing wave.

The RF field configuration is designed as shown in Fig. 5. Plasma inside the circular waveguide is not expected to fully absorb the RF power. In order to make the plasma reaction more effective, we locate the standing wave maximum of the RF field at path of the beam flux.

4.2 The magnetic coil system

The magnetic coil showing in Fig. 2 achieves the magnetic field required by ECR condition as 875 gauss. The cooling water is estimated adequate for coil power consumption. The cooling water is around 30 psi partial pressure in 3/8-inch tubing. The magnetic field contour is a typical helmholts type configuration.

There are 8 iron bars distributed outside of coils to reduce the stray magnetic field seen by the underneath E-beam hearth.

4.3 The simulation of electron movement

The ionization cross sections of niobium can be approximately computed by Binary-Encounter-Bethe model [23]. According to Fig. 6, the ionization will likely happen with 30-100 eV electrons.

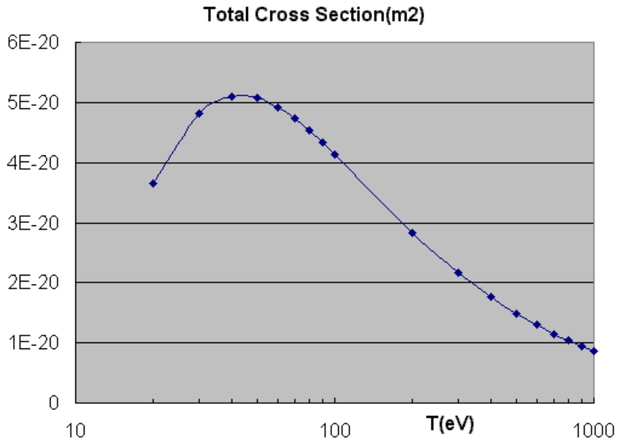

Fig. 6 Niobium total ionization cross section for its 5 outer shell electrons. Estimated from BEB model.

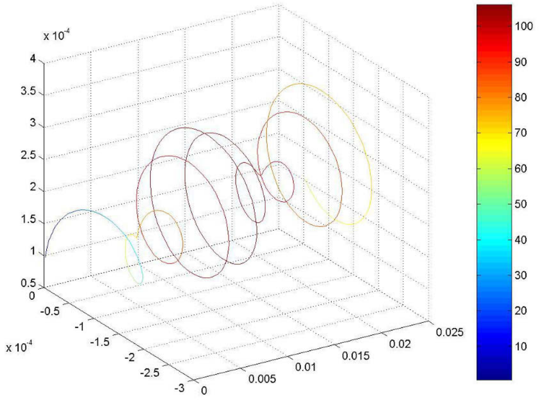

Fig. 7 A typical electron's movement inside the ECR-magnetic field coupled with longitudinal electric field. Color information denotes the electron kinetic energy as eV

A small electron tracking code is written to understand the electron's movement inside the electromagnetic field. The simulation shows that a little off-resonant magnetic field keeps electron kinetic energy oscillates between 30 to 120 eV, which helps to ignite and maintain niobium plasma. Fig. 7 shows one simple example.

4.4 Plasma with Argon, Nb vapor and Copper vapor

The system is tested for Nitrogen, Argon, Copper and Nb vapor. With nitrogen and Argon, the plasma reaction is quite well established, since the gas molecule moves inside the circular waveguide in every direction, and fills everywhere. The copper and niobium vapor moves in a fixed direction, traveling with estimated higher velocity due to the electron beam heating. To achieve the plasma reaction, the equivalent partial pressure of the metal vapor needs to be higher than that of Argon gas. The minimum argon pressure to sustain the plasma reaction is $1.1\cdot10^{-5}$ Torr. While the copper and niobium partial pressure is about $1.0\cdot10^{-4}$ Torr, which comes from metal flux of 65Å/s for copper and 100 Å/s for niobium.

While there is a minimum metal flux requirement for plasma ignition, the RF power plays main role to change the ionization rate. In current system, a moderate 330 watts RF power is used to feed into the plasma reaction. Niobium plasma consumes about 240 watts RF power on a 132 Å/s flux. The deposition rate of 65 Å/s on substrate represents 50% ionization rate when all the niobium ions are assumed extracted. The full ion extraction is observed when the substrate collection current remains unchanged (34mA) above 12V biased.

The system is expected to provide variable deposition rate from 50 Å/s to 100 Å/s for any specific bias voltage range from 15 volts to 300 volts. The higher voltage represents higher energy niobium ions, which may cause the re-sputtering of the film. That raises a question whether a 100 Å/s deposition of 300V ions is achievable. (The question will be answered soon).

5. THIN FILM BY THE ENERGETIC VACUUM DEPOSITION

5.1 The preliminary result of the Niobium thin film

The niobium film has been successfully deposited by this energetic condensation system. The collector current of 35 mA was recorded with 33V collector bias voltage. The vacuum before plasma was $1.0\cdot10^{-7}$ Torr, and $1.5\cdot10^{-6}$ Torr when the plasma is on.

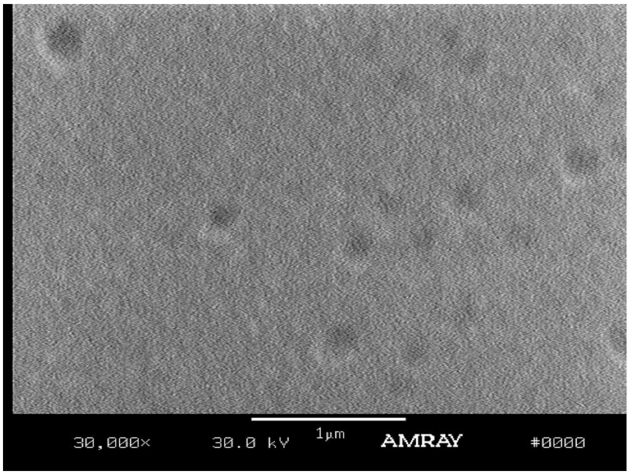

Fig. 8 Niobium thin film on copper. (SEM)

The copper substrate was electro-polished, and about 150 µm thick copper was removed.

A separate thin film on silicon is made at the same time in a bid to look at the film cross section. Fig. 9 shows that the film is at the 1.7 micron thickness.

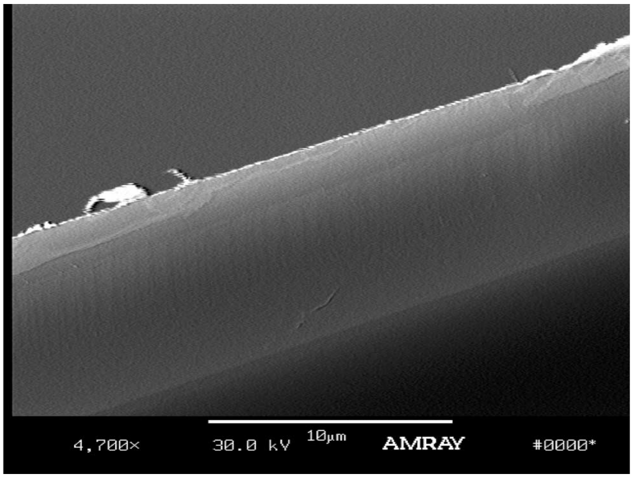

Fig. 9 Niobium thin film cross section on silicon. (SEM)

We relatively measured the transition temperature of niobium film, which is compared with the solid niobium disk.

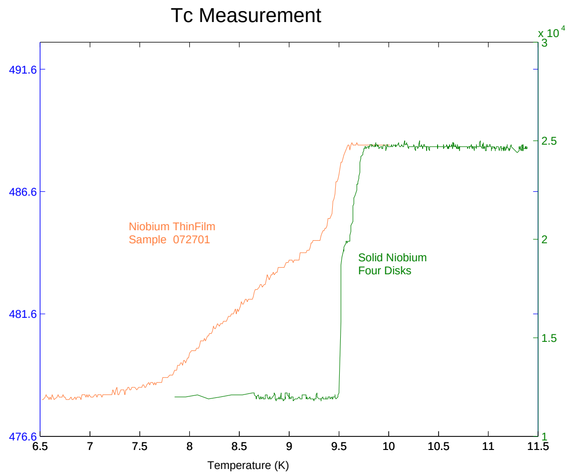

Fig. 10 Niobium thin film Transition temperature in compare with solid niobium. (measured by induction coils, temperature not calibrated)

5.2 The things that need to be studied.

While the deposition system will be improved along the way, following is the list of investigations that are being carried out.

1. Can niobium film with good surface morphology be made? And in what parameters?

The vacuum for the deposition is now at 1.0e^{-7} Torr. That may be good enough for film growth under faster deposition rate.

2. Film quality

Following systems are being used to observe the thin film:

- SEM→ Grain size, number
  Grain structure
  Material composition
  Visible defects
- TEM→ Detailed Grain structure
- SIMS→ material composition local distribution

SEM proved to be a very useful tool for most of the surface analysis required for this project.

3. The transition temperature of niobium thin film.
4. The RRR of the niobium thin film.
5. The critical magnetic field.
6. Field emission test.
7. Measuring surface resistance relatively,
8. The source of the magnetic field limitation.

6 FUTURE PLAN

If the niobium thin film made by energetic condensation proves to be successful, we will explore the plasma transportation for thin film deposition inside a real cavity shaped structure. One should note that the energetic deposition in vacuum is applicable to many other applications, too.

7 ACKNOWLEDGEMENT

The authors are very grateful to Sam Morgan for his mechanical support, Peter Kushnick for his cryogenic support, Tom Elliott for his electropolishing support, John Musson for his RF support, Tong Wang for her help to capture SEM photos and EDS analysis, Julie Oyer for her help on paper editing and many other staffs in Jefferson Lab for their generous help.

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