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STUDY PROGRESS OF PULSE LASER ANNEALING FOR NIOBIUM FILM ON COPPER*

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Abstract

The recent studies of laser annealing on niobium films on copper are reported. Annealing is normally used to deal with the surface, reducing defects and even changing the microstructure of the coating film. Short pulse laser can produce a sharp step temperature field on the film thickness scale (μm), which anneals the surface without substrate heated. The laser annealing experiments of niobium thin film sample have been carried out, and according to SEM and FIB results, Nb films melted and recrystallization occurred. Grains growing up can be observed while the power density of laser pulse increased.

INTRODUCTION

Thin film coated copper cavities are a promising alternative for RF superconducting accelerators with better thermal stability, better mechanical stability and low cost compared with bulk niobium cavities, and have been used in many accelerators. Most of Nb sputter-coated copper cavities suffer from obvious Q-slope with increasing accelerating field. There are many factors contributing to Q-slope, such as surface defect, film granularity, film-substrate binding force and roughness. The microstructure of Nb film on copper behaves like a network of superconducting grains coupled by Josephson junctions (the grain boundaries) [1][2], which gives rise to a RF dependent term in the surface resistance. Reducing the number of defects and grain boundaries per unit area should result in the decrease of surface resistance.

The films qualities seriously affect their superconducting performance, and the ultimate goal is to fabricate the bulk-like thin film, which we try to realize both in better fabrication methods and post-treatment.

The common PVD coating method for superconducting cavities including evaporation coating, magnetron sputtering coating, etc. According to the extended SZD (structure zone diagram) proposed by Andre Anders [3] (Fig. 1), the structure of the film is determined by deposition homologous temperature and the pressure. However, for the thin film deposited on copper, the temperature is limited to copper melting point (1083℃), which means it cannot be annealed with traditional heating. To solve this challenge, we are studying the benefits of short pulse laser annealing for niobium films on copper deposited by magnetron sputtering.

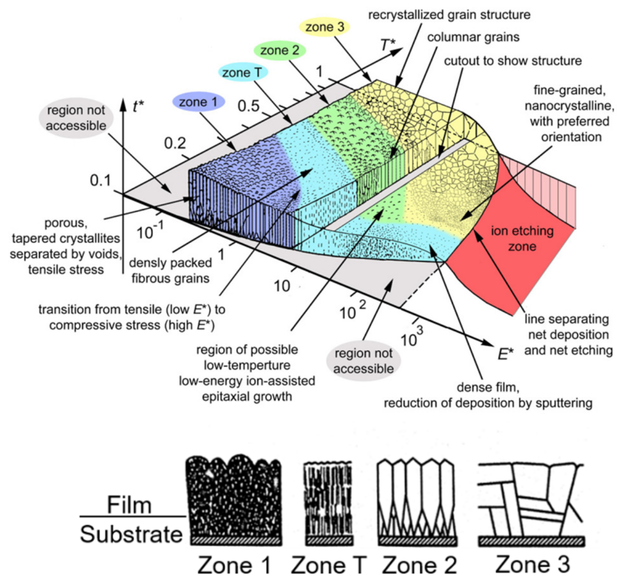

Figure 1: A possible presentation of such extended SZD [3] the figure of different zone. With the increasing temperature and pressure, the film structure changes from zone 1 to zone 3, and the formation of holes and protrusions in grain growth decreases which should make it more similar to bulk material.

EXPERIMENT SET UP AND PROCEDURE

Laser and Sample Preparation

The diffusion of heat generated by laser incident on the film surface satisfies the heat conduction equation, which was simulated by ANSYS first and showed that the pulse width of the incident laser could determine the depth of temperature field distribution. In order to make the heat limited in the film thickness scale, we selected the UV laser pulse of 30ns and changed the voltage and exposure times to explore the proper pulse energy. The simulation results by ANSYS are showed in Fig. 2.

Figure 3 shows the samples we used which were respectively deposited on copper by dcMS (DC magnetron Sputtering) and HIPIMS (High Power Impulse Magnetron Sputtering), both of them have smooth surface and good superconducting performance. The film thickness of the samples is about a few hundred nanometres, while the film of HIPIMS has higher density and compactness, which may benefit the annealing results.

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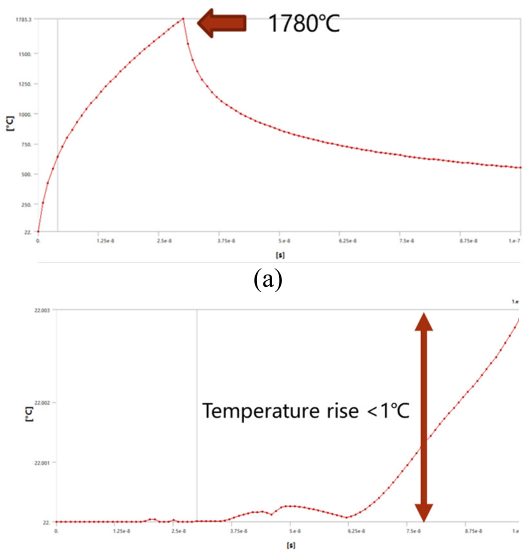
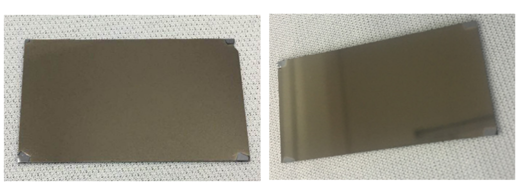


(a)

(b)

Figure 2: The ANSYS simulation of temperature rise over time (surface(a) and Nb/Cu interface (b)) caused by 30ns, $10^7 W/cm^2$ laser pulse. The top temperature of surface can reach 1780ºC while the interface temperature of film/substrate rises less than 1ºC, which can realize the film annealing without affecting the substrate. The power density used in the experiment will be larger, and the top temperature obtained on the surface will be larger too, but the pulse width remains 30ns, thus the depth of temperature field distribution will not be significantly affected.



(a) (b)

Figure 3: Nb/Cu film deposited by dcMS (a) and HIPIMS (b). The film thickness of is about a few hundred nanometres, both of them have smooth surface and good superconducting performance.

Annealing Process

So far, we only experimented with single point irradiation and plan to add laser scanning system in the future.

The laser repetition frequency is typically 1hz. The duration of the effects caused by laser pulse is about 10 pulse widths, far less than the pulse repetition time, thus multiple pulses can be considered as multiple irradiation. We studied the effects of different energies and different exposures times on the surface.

Samples were cut into small pieces and irradiated by 30ns UV laser pulse. Figure 4 shows the simplified laser path into samples. The exposure area of sample is about $0.7 \times 0.35 cm^2$, and the annealing atmosphere was air.

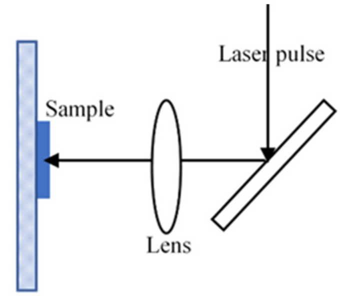


Figure 4: Simplified laser path into samples. The niobium film absorbs laser energy and then produces recrystallization. Such annealing process makes film grain size increase, and reduces impurities effect.

RESULTS AND DISCUSSION

We used the ultraviolet laser pulse (30ns) to anneal the dcMS Nb film first, and there are lots of cracks unfortunately. Figure 5 shows the FIB results and Figure 6 shows the SEM results. As the number of laser pulses increased, niobium films were melted gradually, and recrystallization occurred which made the grain size become larger. FIB images display that the film with smooth surface still have many holes and defects in the grains scale. After laser annealing, the regional compactness was improved and defects were repaired to some degree.

What disappointed us is the cracks also increase with the exposure times. Some samples had vaporization events on their surfaces, even exposing copper substrate. It may relate to the bonding force and the photon energy of UV. Actually, the wavelength of the laser has little effect on the reflectivity of the metal. Infrared to ultraviolet laser could be selected as the annealing heat source[4], while the longer wavelength laser may be selected to avoid the influence of high energy photons onto the surface in the future. Besides, one of the possible contributing factors of cracks is the increase of local area density, and original grains become larger and result in cracks.

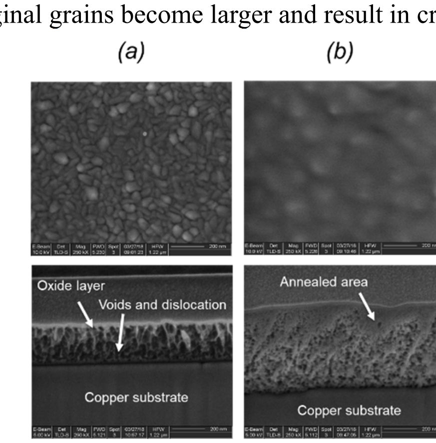


Figure 5: The FIB results of dcMS samples before(a) and after(b) annealed. It can be observed that defects decrease a lot, but the annealing is not very uniform.

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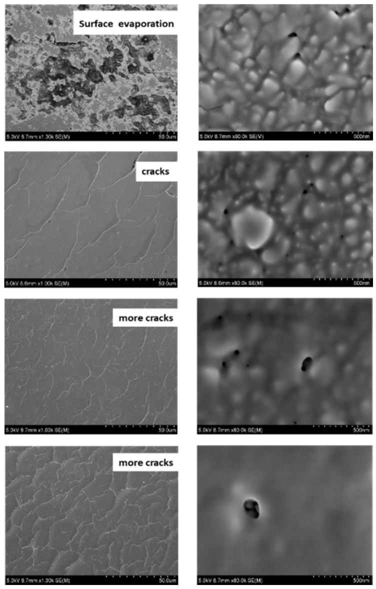

Figure 6: SEM results of annealed samples of dcMS. The voltage of the laser (reflect the power density) is 14kV. Different columns represent different magnification (× 1k and × 80k). With the laser exposure times increased (5s/1Hz, 1~2s/5Hz, 10s/1Hz, 20s/1Hz), the cracks increase too. The film grains melt, and grain boundaries fade away.

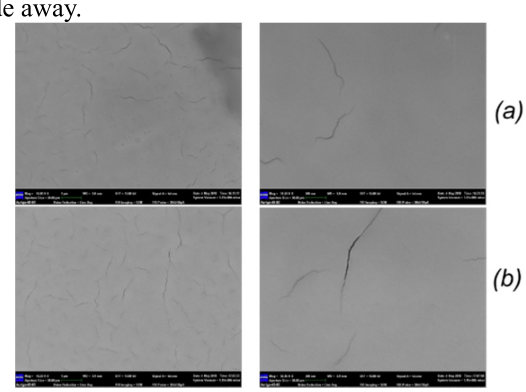

Figure 7: SEM images of HIPIMS samples. (a)~(b) the voltage of the laser (power density) is from 14kV to 16kV (exposure time is 30s). Different columns represent different magnification (× 1k and × 50k).

To reduce the cracks which may be caused by the increase of local area density, we also annealed the Nb/Cu samples deposited by HIPIMS. Figure 7 and Figure 8 showed the surface appearance after annealing. Compared with dcMS samples, the HIPIMS Nb films have better surface performance and what most benefit the annealing process are the compactness and higher film density. With the increase of exposure times or power density, the grain boundaries were gradually blurred and some samples (such as Fig. 8 (b)(c)) obtained very smooth surfaces both on the macro level and microstructure. It is clear that Nb films melted gradually and recrystallized well with HIPIMS samples. When the power density is over high or the exposure too much, cracks still appear, but the vaporization didn't occur which may due to good binding force of HIPIMS films.

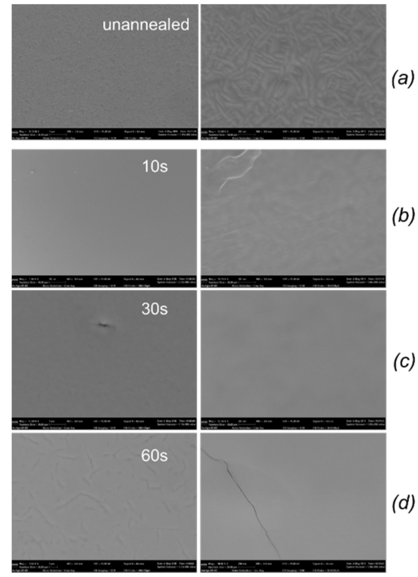

Figure 8: SEM images of HIPIMS samples before and after annealed. There are few cracks and the length of a crack decreases to several hundred nm. (a) Unannealed Nb/Cu film; (b)~(d) The exposure time increased from 10s to 30s, 60s (voltage is 13.5kV). Different columns represent different magnification (× 1k and × 50k).

There are several disadvantages in this experiment, such as the laser power density is not stable enough to repeat the irradiation, and the air annealing atmosphere prevents the further superconducting tests of these samples. The following experiment is preparing and new laser device, laser scanning system and vacuum chamber will be used in annealing system.

CONCLUSION

Niobium thin film coated copper cavities are a promising alternative to bulk niobium cavities for RF superconducting accelerators. As for the thin films, the challenges are improving the surface superconducting performance and reducing defects of the coating film, which can be greatly solved by laser annealing. Our recent studies of laser annealing on niobium films on copper are reported here, including the theorical simulation and primary experiments. Surface appearance measured by SEM and FIB of dcMS and HiPIMS samples are compared after annealing. The laser both wavelength and power density we chose may not very proper to anneal our samples. But it still can be observed that Nb films melted and recrystallization occurred. And because of the higher density and compactness of HiPIMS samples, cracks decreased a lot and better recrystallization happened according to the results. Further experiments will change the annealing environment and exposure method, and superconducting performance tests will be carried out too.

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