ISBN: 978-3-95450-191-5 doi:10.18429/JACoW-SRF2017-THPB057

INVESTIGATION OF NUCLEATION STAGE IN DIFFUSION COATING OF Nb_3Sn ON Nb*

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Abstract

Nb_3Sn has the potential to improve properties of SRF cavities, such as the gradients and the working temperatures. Institute of Modern Physics has launched its Nb_3Sn thin film coated SRF cavity project in 2016. Samples have been successfully coated to study the process of tin vapor diffusion. The main part of the deposition system is a tube furnace, which working temperature can reach 1100℃. Basic material characterization of the Sn-Nb film will be presented in this work.

INTRODUCTION

Niobium is the most widely used material for the present SRF accelerators. While the niobium cavities are approaching the material limit. The next superconducting for SRF cavities should have a higher T_c (the critical temperature) and H_{sh} (the superheating field) for higher working temperature and quality factor. The T_c and H_{sh} of Nb_3Sn are nearly twice that of niobium [1]. So Nb_3Sn has the potential to improve properties of SRF cavities, such as the gradients and the working temperatures.

The Nb_3Sn for SRF applications has a long history. Siemens AG and University of Wuppertal started to developed a diffusion coating recipe to get Nb3Sn cavities in 1970's. They successfully fabricated Nb3Sn cavities with tin vapor diffusion technology. Their susceptibility to ambient magnetic flux has resulted in a $Q_0 \approx 10^{10}$ at 5 MV/m at 4.2 K, but then suffered a precipitous drop [1]. The possible reason is an implicit property of Nb_3Sn. However, all studies of Nb_3Sn cavities fell off by the end of 1990`s until Cornell stated Nb_3Sn programs in 1990's. Now, Nb3Sn programs are now on going at Cornell and Jefferson.

Institute of Modern Physics has launched its Nb_3Sn thin film coated SRF cavity project with Sn vapor diffusion technique in 2016. The coating consists for the process of tin vapor diffusion we used is based on that in Cornell shown in Fig. 1:

I. A degas stage lasting hours
II. Nucleation
III. Ramp-up
IV. Coating
V. Annealing

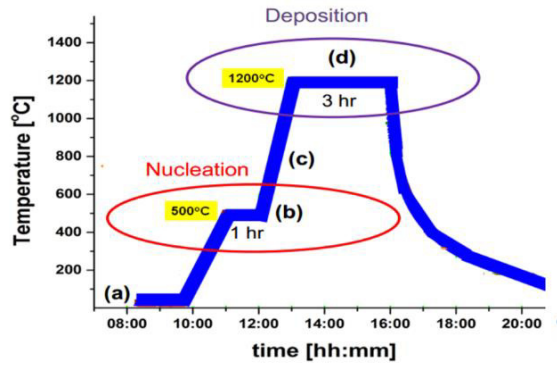

Figure 1: Heating profile used for sample coating. The nucleation step followed by deposition [2].

When researchers at Siemens AG (1970`s) began their Nb_3Sn programs, they found niobium spots not covered with Nb_3Sn film. While the coating process used tin halides or pre-anodized, the problem disappeared. They finally found tin halide (high vapor pressure) initiates Nb-Sn nucleation early by more availability of tin [3]. My study focuses on the parameters of the nucleation step.

THE EXPERIMENTAL

Experimental Set-up

For better monitoring the temperature and vacuum, a tube furnace system was used to perform experiments to imitate the nucleation step. The details of the experimental set-up are shown in the Fig. 2.

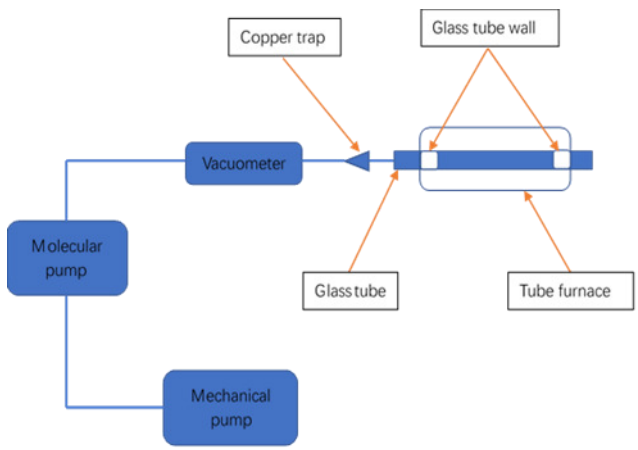

Figure 2: The coating system for samples.

A vacuum port is connected on the quartz glass tube. The system vacuum can reach 1×10^-4 Pa, and the working

*Work supported by the Key Research Program of Frontier Sciences, CAS, Grant NO. QYZDY-SSW-JSC019 and the National Natural Science Foundation of China (91426303).

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vacuum is between 5×10^-4 Pa to ~9×10^-4 Pa when the temperature is at 600 ºC. The samples we used are 10*10 mm^2, and the mass of tin is around 10 gm, much more than the vapor diffusion process needed.

Experimental Details

Before the experiments, we cleaned the system with large quantities of alcohol and then warmed the system without tin or niobium in the glass tube at working vacuum for 16 hours. A mass spectrometer was connected at the end of the tube to monitor the gas change. After the high temperature degassing, the residual gas had little effects on the experiment. The time vs the partial pressure and temperature is show in the Fig. 3.

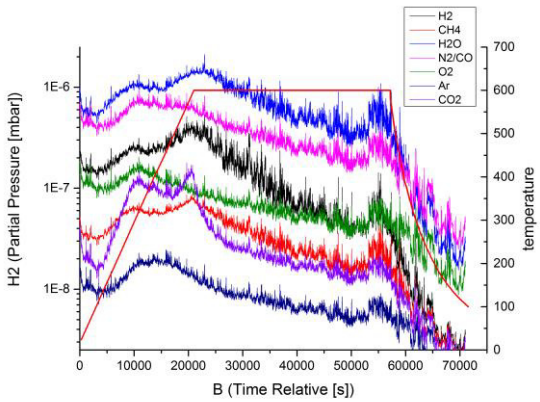

Figure 3: Heating profile used for the system pre-warming. The temperature was divided into three areas. The first part was a degas stage lasting six hours until the temperature reached 600 ºC. The second part was holding stage lasting 10 hours to make sure the gas escaping out. The third part was the source heater turns off and the furnace was nature air cooling. Partial pressure conditions of system were also shown.

Two 10 mm*10 mm niobium samples were coated at each experiment. The samples and the tin source were placed in a glass pot: both samples were placed over the tin source, showed in Fig. 4.

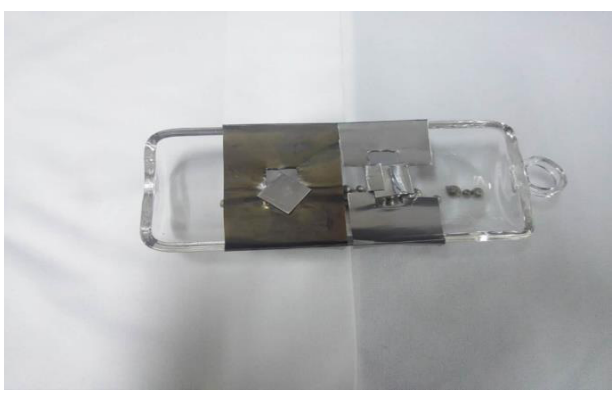

Figure 4: Two niobium samples were placed over the tin source.

The vacuum of the system was pre-pumped to 3.4× 10^-4 Pa. The temperature of the furnace climbed at a rate of 10 ºC per minute until it reached 600 ºC. After maintaining this temperature for one hour, heating ceased, and the system cooled down gradually.

In the next experiments, we remained the climbing rates and the holding time of temperature, only changed the target temperature to repeated experiments. Finally, we got the samples after nucleation step at 500 ºC, 550 ºC, 600 ºC and 650 ºC. During the degas stage and nucleation, the vacuum gauge reading changed with the temperature. Similar structure was observed: a typical one (nucleation at 600 ºC) is shown in Fig. 5.

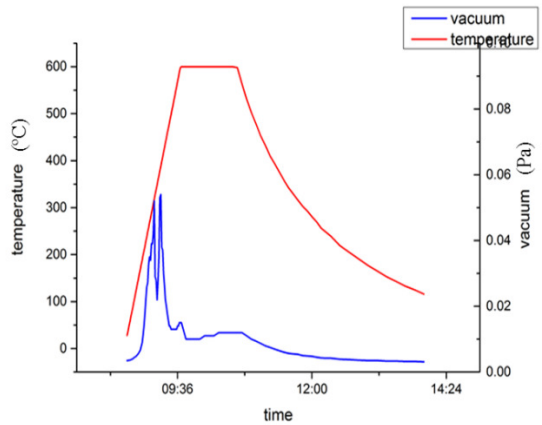

Figure 5: The vacuum gauge reading changed with the temperature. At the degas stage, the curve spectrum of vacuum gauge reading had two peaks. During the nucleation stage and annealing stage, the vacuum was levelled off.

RESULTS

The local composition of the deposited coating was analysed by scanning electron microscope (SEM) and focused Ion beam (FIB). SEM provides images of the coated surface. FIB shows the interface layer between niobium and tin.

SEM images from different coating temperature are shown in Fig. 6. The images show that each sample has coated a layer of thin film. And mounts of tin are in the surface of niobium. Higher nucleation temperature works thicker tin layer on the surface. The compositions of samples at 550 ºC, 600 ºC, 650 ºC are analysed with SEM at the same time. The thickness of all coating layers is less than 1 um. The grain size of tin coated at 500 ºC is less than 50 nm. When the nucleation temperature is 550 ºC, the grain size of granule on the surface is about 100 nm, and atom ratios of niobium and tin are 9:1, much more niobium than tin on the surface. As for 600 ºC, the grain size of granule on the surface is from 80 -500 nm, and atom ratios of niobium and tin are 5:5 or 3:1. There are some Nb3Sn and Nb_xSn synthesized from niobium and tin. The atom ratios of niobium and tin after 650 ºC nucleation are 1:4 or 3:7, and the largest grain size is about 1 μm. But the layer is porous and non-uniformity.

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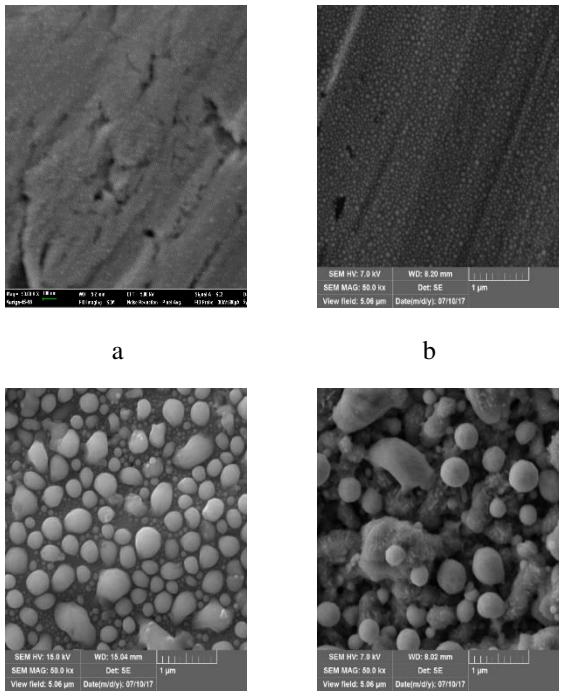

a b c d

Figure 6: SEM images at different nucleation T temperature: (a) T=500 ºC, (b) T=550 ºC, (c) T=600 ºC, (d) T=650 ºC.

Focused ion beam, also known as FIB, is a scientific instrument that resembles a scanning electron microscope (SEM). However, a FIB setup uses a focused beam of ions instead of a focused beam of electrons to image the sample in the chamber. Unlike an electron microscope, FIB is inherently destructive to the specimen. But with FIB tools etching surfaces, we can get the images of the interface.

FIB secondary electron images (see Fig. 7) show that there is almost no interface between tin and niobium in the samples coated at 550 ºC for 1 hour. But complete coverage of the surface is noticed at these samples. The space between tin and niobium is noticed about 26 nm. Dark areas are also observed between the tin layer and the niobium layer.

Improving the coating temperature to 600 ºC without changing the coating time, the thickness of the tin layer is not changed. The biggest difference is that there are some large particles coming to compound, independent of the tin layer and the niobium layer, with no contacting one by one either. The grain sizes of these large particles are noticed to be about 100 nm to 234 nm.

Further raising the coating temperature to 650 ºC, images show the particles become much larger. The biggest Grain size is noticed to be 826.2 nm. And the particles are coming to contact each other. The interface between tin and niobium is fist to be observed. Some particles are contract with the interface. The thickness of this interface is to be 245.6 nm. But the tin layer is noticed to be thinner by eyes.

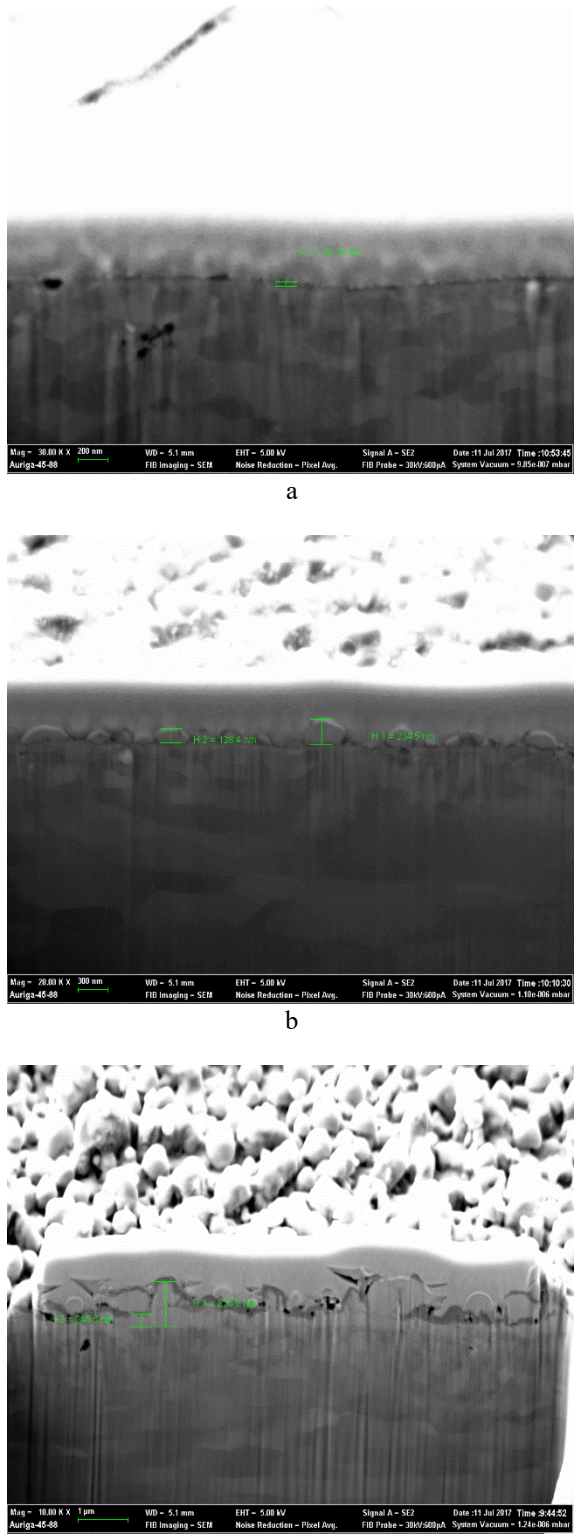

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Figure 7: SEM images after FIB at different nucleation T temperature after FIB: (a) T=550 ºC, (b) T=600 ºC, (c) T=650 ºC.

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CONCLUSION AND FUTURE PLAN

With studying the local composition and topography of Nb_3Sn nucleation stage, temperature is found to influence the compositions. It is also found that particles are noticed earlier than the interface. This is, nucleation stage might have contribution to the coating process.

The parameter of temperature may play an important role to determine the process of the nucleation stage. It is plan to investigate compositions of particles and the evolution of topography during the nucleation process.

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