A15 SUPERCONDUCTORS BY THERMAL DIFFUSION IN 6 GHz CAVITIES

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Abstract

In the framework of the research for a valid alternative to Nb for RF applications, Nb_3Sn has been investigated.

Nb_3Sn is produced using the *liquid tin diffusion* method. A bulk Nb 6 GHz cavity is introduced into molten Sn (dipping step) and heat treated (annealing step). The process temperature must be higher than 930°C, to avoid the formation of spurious low T_c phases.

The experimental procedure has been progressively modified to obtain a homogeneous, stoichometric and compact film with satisfactory superconducting properties. The "*double process*" is particularly promising: the sample annealing is partly performed in Sn vapour, partly in vacuum (T_c = 16-17 K and ΔT_c = 0.3-0.5 K, no residual Sn traces on the sample surface, no Sn rich phases).

Having good results with A15 samples, doesn't mean obtaining performant Nb_3Sn superconducting resonators. For this reason a hundred of small 6 GHz cavities, completely equal in shape to the real scale model [5], was built and several Nb_3Sn 6 GHz resonant strucures have been produced and tested with encouraging results.

Nb_3Sn BY LIQUID PHASE DIFFUSION

Considering the goal is to coat a large number of cavities, this technique is probably the less expensive one: it employs a low technology equipment and it is quite fast [2, 3].

It consists in the bulk Nb introduction into molten 99.99% Sn for a short period of time (2h max) and in its further annealing outside the molten bath. During the dipping process, the diffusion of Sn into the substrate begins.

Experimental Procedure and Results

A high vacuum cylindrical reaction chamber is used, it contains an Alumina crucible for the Tin bath and a linear manipulator to move the samples from the top to the bottom and vice versa. The lower part of the system can be heated by an irradiating furnace, while the upper zone can be cooled trough a water jacket [2, 3, 4].

The annealing procedure has been carried on in three different ways, in order to improve the coating quality [3]. Initially, we performed the heating treatment simply extracting the sample from the bath and keeping it hot, just above the molten Sn (*Sn vapor annealing process*). We obtained samples with T_c up to 17.7 K and $\Delta T_c \sim 0.11$ K, but they are affected by the problem of unreacted Sn on the surface and traces of Nb-Sn spurious phases.

We tried to improve the film properties removing the Sn crucible from the vacuum chamber before performing the annealing step, in order to stop the Sn vapour flow investing the sample (*vacuum annealing process*). We obtained Sn free films, but their superconducting characteristics were not satisfactory.

However, we produced and tested one small cavity (6 GHz) just to have a starting point. The corresponding Q vs E_{acc} curve is depicted in Figure 1.

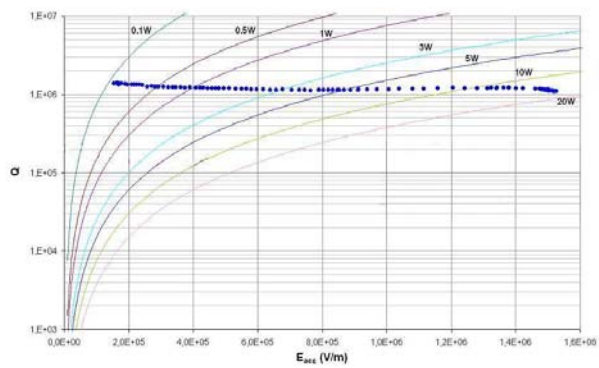

Figure 1: The Q value is lower than the quality factor of a pure Nb cavity (f = 6 GHz, T = 4.2 K, Q_{BCS}(Nb) ~ $3x10^7$), E_{acc} (max) is closed to 1.5 MV/m. The maximum output power of the rf amplifier was reached.

Finally, we performed what we call the "*double process*", consisting in a first annealing in presence of Sn vapour, followed by a second heating treatment in its absence. We gained intermediate results with T_cs 16.6-17.5 K and ΔT_cs of 0.15-0.28 K, (no Sn traces on the surface or different Nb-Sn phases into the grown coatings). This kind of process seems to be extremely promising and it has been chosen to coat the next small cavities.

The New Experimental Apparatus

In the meanwhile the "Double Furnace System" (Figure 2) has been built to avoid air contaminations of the films due to the vacuum chamber opening (the Sn crucible has to be removed to perform the annealing without Sn).

Figure 2: Our "Double Furnace System". It was built to simplify the experimental procedure. Now it is possible to perform the annealing in vacuum without opening the chamber to remove the Sn crucible.

We produced several cavities that have been measured through the 6 GHz rf test apparatus which has been deeply described elsewhere [1].

Two of the curves we obtained are depicted in Figure 3: we chose them because they are a representative sample of the results we gained.

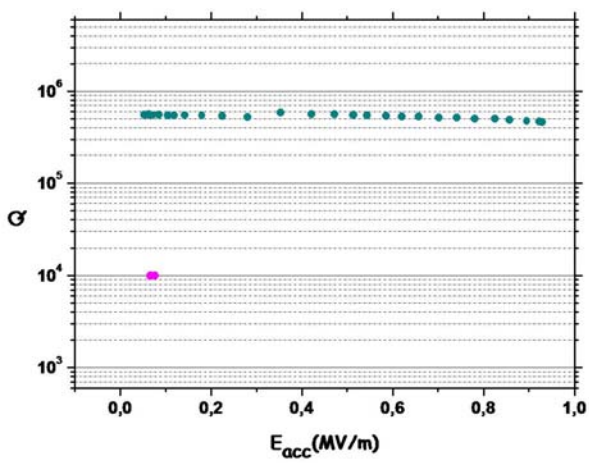

Figure 3: Two examples of the Q vs E_{acc} curves we obtained (f = 6 GHz, T = 4.2 K, $Q_{BCS}(Nb) \sim 3x10^7$). In both case the quality factor is lower than the pure Nb one.

Discussion

We still have two unsolved problems:

- ureacted Sn droplets;
- spurious Sn rich, low T_c phases (Nb_6Sn_5 and $NbSn_2$);

As shown in Figure 4 a big quantity of Tin is evidently present in the bottom part of the cell, while it is not possible to see Sn traces on the surfaces of the cut-offs.

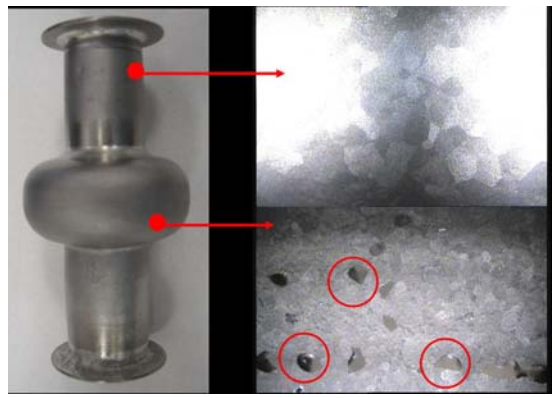

Figure 4: Nb_3Sn 6 GHz cavity obtained using the "double process". The unreacted Sn problem is still present.

Sn droplets are still there because of the cavity shape: it is much more complicate than a small sample one. Looking at the Nb-Sn phase diagram [3] it is easy to understand if a Sn excess is present, the cooling down step has to be as fast as possible to avoid the formation of spurious phases. We move the cavity in the cold zone (where a water jacket is fixed) then we stop and open the external furnace. Probably our cooling down procedure is not quick enough.

PRESENT WORK

These considerations helped us to move on following three different directions:

- Change of the process parameters (temperature, time, cooling speed);
- Chemical polishing;
- Cavity vibration.

Change of the Process Parameters

The first possibility is to increase the working temperature (T). Now the maximum T we can reach is 1050 °C (sensor fixed to the external furnace). The vacuum chamber is made of Inconel and we can't go above the temperature stability range of the material (T < 1100 °C). The second chance is to prolong the annealing time. In the meanwhile the cooling step has to be modified: because of the necessity to increase the cooling speed as much as possible, a new apparatus must be drawn.

Chemical Polishing

We tried to remove unreacted Sn and/or the first Nb-Sn non-stoichiometric layer through the electropolishing of the cavity.

The used solution is the classical 1:1:2: it flows into the cavity towards the zone of the cell that is Sn contaminated.

In the following graph an example of the Q vs E_{acc} curves we obtained is depicted.

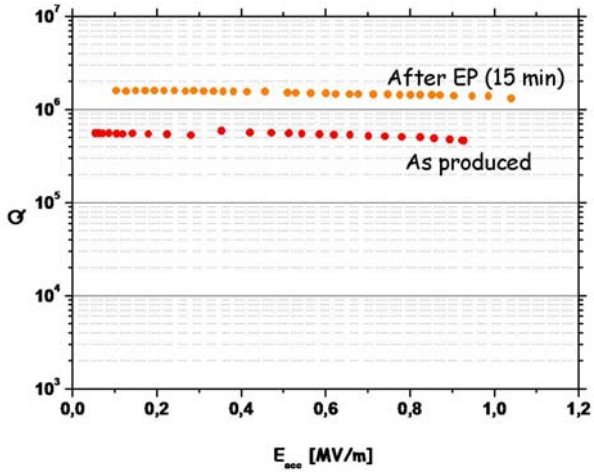

Figure 5: An example of the Q vs E_{acc} curves we obtained (f = 6 GHz, T = 4.2 K, $Q_{BCS}(Nb) \sim 3x10^7$) for one of the cavity before an after the chemical treatment. The quality factor increases but it is one order of magnitude lower than what we gain for a pure Nb 6 GHz.

The quality factor increases but it is one order of magnitude lower than what we gain for a pure Nb 6 GHz resonator.

There are two different explanations. The first one is we are removing what compromises the cavity performance (i.e. unreacted Sn, spurious phases). The second possibility is we completely removed Nb_3Sn and we have just a kind of "dirty" Nb. The idea is to go on with the chemical treatment measuring the cavity progressively and to cut some 6 GHz resonators to analyze their internal surface in detail.

Cavity Vibration

The third direction we are following consists in the cavity vibration during its extraction from the liquid Sn bath. This solution may cause liquid Sn droplets to fall down, promoting the elimination of unreacted Sn.

We drew a kind of "clip" to bring the vibration to the linear feedthrough to which the 6 GHz cavity is fixed. The vibration is produced through an external vibrating device.

The first cavity has been produced and tested and the result we gained is the following (Figure 7). The quality factor is two orders of magnitude lower than the pure Nb one.

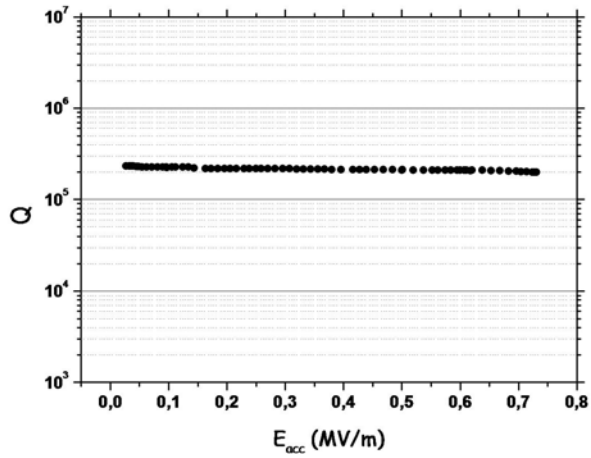

Figure 7: The first Q vs E_{acc} curve we obtained (f = 6 GHz, T = 4.2 K, $Q_{BCS}(Nb) \sim 3x10^7$) after the vibration treatment. The quality factor is two orders of magnitude lower than what we gain for a pure Nb 6 GHz.

We made the cavity to vibrate for two hours, immediately after the resonator extraction from the liquid Sn bath, during the annealing step (with vapor). We have to study the best condition of vibration to optimize the falling down of the droplets.

CONCLUSIONS

We obtained many Nb_3Sn samples changing progressively the process parameters to optimize the procedure. The chosen technique consists in a double annealing, one in presence of Sn vapor and a second one in vacuum. We can produce a lot of 6 GHz cavities in a fast and easy way and they are an efficient tool to test the A15 material properties in RF. The main problem we observed is the presence of unrected Sn on the cavity cell bottom part. We are going on to get rid of it chinging the process parameters, using the chemical treatment and the cavity vibration. We are still far from the optimization of the process but with 6 GHz we can move on fast.

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