THE PROGRESS ON Nb₃Sn AND V₃Si

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Abstract

In the framework of the research for a valid alternative to Nb for RF applications, Nb_3Sn and V_3Si have been investigated at LNL.

The chosen technique to obtain Nb₃Sn is based on the molten Tin diffusion method and it has been progressively modified to improve the samples superconducting properties (T_c and ΔT_c) and to eliminate Sn traces on the Nb₃Sn surface. Our "Hybrid process" seems to be the most promising ($T_c = 16.8$ K and $\Delta T_c = 0.16$ K, no residual Sn traces on the sample surface, no Sn rich phases).

The present point is to test the obtained material RF properties: the best recipe used for samples has been applied to coat a 6 GHz Nb cavity.

At the same time we are going to study different techniques to achieve Nb_3Sn good superconducting cavities.

One of them is the so called mechanical plating. Secondly we are going to study the multilayer technique: the first attempt gave a superconducting deposition with $T_c = 17,9$ K and $\Delta T_c = .0,02$ (four contacts measurement).

The V₃Si intermetallic compound has been obtained using the thermal diffusion of Silicon into Vanadium: bulk V is heat treated in a SiH₄ atmosphere for several hours and then annealed in vacuum. The samples superconducting properties are encouraging $(T_c \approx 15,5 \text{ K and } \Delta T_c \approx 0,2 \text{ K}).$

To check the material RF performances we prepared a V₃Si 6 GHz cavity (sylanized for 4h at 850°C with $p(SiH_4) = 5x10^{-3}$ mbar): it has been measured several times after a heating treatment in vacuum progressively longer (6h, 12h, 36h, 84h at 850°C).

At the moment, we are trying to use plasma during the silanization process to avoid the hydrogen presence into the growing films. The first V₃Si coating grown has $T_c = 15,7$ K and $\Delta T_c = 0,22$ K: this result is already comparable to what we had with the "traditional" technique.

Nb3Sn BY TIN THERMAL DIFFUSION

Nb₃Sn 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 [9, 10].

It consists in the bulk Nb introduction into molten 99,99% Sn for a short period of time (2h max) and in it's further annealing outside the molten bath. During the dipping process, the diffusion of Sn into the substrate begins. As shown in the Nb-Sn phase diagram [1, 2, 3, 4-7, 8], it is extremely important to work at a temperature higher than 930°C to avoid the formation of spurious (low T_c) phases.



Figure 1: The Nb-Sn phase diagram. Only a narrow region around the stoichiometric composition (18-26 at.%Sn) lead to the A15 phase. High compositional homogeneity is reached only above 930°C.

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 [9, 10]. After fixing the sample to the Nb rod with a niobium wire, we start pumping the vacuum system. We usually bake the chamber using the external furnace (~150°C) to reach a base pressure of 10^{-8} mbar.

Then the temperature is increased: as mentioned above it is important to guarantee the bath T is higher than 930° C. The following step consists in the substrate dipping inside liquid Tin.

The annealing procedure has been carried out in three different ways, in order to improve the coatings quality [10]. Initially, we performed the heating treatment simply extracting the sample from the bath and keeping it hot, just above the molten Sn (*1 step process*). We obtained samples with T_c up to 17,7 K and Δ T_c ~ 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 films 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 (2 step process). We obtained Sn free films, but their superconducting characteristics were not satisfactory. Finally, we performed what we call the "hybrid 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 $\Delta T_{c}s$ of 0.15-0.28 K., (no Sn traces on the surface or different Nb-Sn phases into the grown coatings).

The best recipe has been used to coat a 6 GHz Nb cavity.



Figure 2: Nb_3Sn 6GHz cavity obtained using the "hybrid process". The unreacted Sn problem is still present.

As shown in Figure 2, 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 cut-offs surfaces.

Presently we are going to modify a little bit the process to adapt it better to the more complicate cavity shape before performing RF tests.

In the meantime the "Double Furnace" System 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). Now it is ready and the first samples are in production.

Nb₃Sn BY MECHANICAL PLATING

The mechanical plating technique is essentially a batch process for the bulk coating of small parts such as fasteners, washers, springs and spring clips, steel stampings and nails. This technology is an effective means of applying one metal over another as a coated deposit without using electrical current. It is used to apply a number of metallic deposits such as zinc, cadmium, copper, tin, aluminium and other mixed alloys onto a wide range of substrates. Typically deposits are smooth matte to semi-bright in appearance. Mechanical deposits can be characterized to some extent by their relative thickness. Standard plating builds are typically 8-12 microns. Processing operates at room temperature using the tumbling action of a barrel to create mechanical energy. In the presence of glass bead impact media and proprietary promoter chemistry, this energy is used to uniformly "cold-weld" powdered metallic particles onto the substrate.

We think this simple method could be applied to the production of superconducting cavities. We filled a 6 GHz Nb cavity with Sn pieces (D \approx 5 mm) and tumbled it for some hours. We want it to be Sn coated in order to perform a thermal treatment to obtain the Nb₃Sn A15 phase on its internal surface.

It is possible to see a change in the cavity colour: it is the evidence of some Sn deposition. The 6 GHz resonant structure has been annealed for 1h at 950°C and measured.

The first attempt has not been successful: the Q value of the cavity is the one of pure Nb. It is probable that washing the cavity we removed the sticking Tin from the surface. In the next trial we are filling the resonant structure with Sn powder and zirconia balls hoping to increase the Sn deposited and its adherence on the surface.

Nb₃Sn BY MULTILAYER DEPOSITION DOUBLE MAGNETRON

Experimental Setup

The multilayer depositions are performed in a cylindrical stainless steel vacuum chamber equipped with two planar balanced magnetrons sources. On the top source is mounted a 2 inches niobium target and on the bottom one a 2 inches tin target [11].

The sample holder is in the centre of the chamber and it is composed by an aluminium disk connected to a rotary feedthrough. In this way we can rotate the substrates and alternatively expose them to Nb and Sn plasmas.

The distance between targets and samples is 60 mm and during the deposition we use Argon as process gas. The films annealing is performed after the deposition, in a vacuum chamber heated by an external irradiating furnace.

During the sputtering process the angular velocity of the sample is constant; in particular it makes a complete round every 13 seconds. The substrate used are made of sapphire (10x10 mm). At the beginning we performed the deposition of N

At the beginning we performed the deposition of Nb and Sn separately in order to study their deposition rate. To obtain the desired A15 structure of Nb₃Sn, we calculated that the Nb thin film thickness must be 4.5 by the Sn coating one. We found that with our chamber and magnetron sources design, we have to supply about 0.18 A to the Sn magnetron source and 2 A to the Nb source. We progressively increased a little bit the Sn source current value because of Sn high vapour tension and its consequent easy reevaporation during the process.

Before the multilayer deposition, the chamber is evacuated at a base pressure $\leq 10^{-8}$ mbar and, during every sputtering process, we use Ar at a pressure of 5.0×10^{-3} mbar.

In the following table the parameters used during one of the depositions are reported: Nb magnetron is supplied with 2.0 A and Sn target with 0.22 A as summarized in Table 1.

Table 1: Sputtering deposition parameter.

Sputtering	Current	Voltage	Power
target	(A)	(V)	(W)
Sn	0.22	587	126
Nb	2.0	437	880

The first step consists in the deposition of Nb (2 minutes) in order to eliminate Oxygen contaminations in the chamber. Then we start depositing simultaneously Nb and Sn for 30 minutes. The last step is the deposition of Nb in order to have a "thick" Nb film on the top: this is important because Sn could evaporate during the post annealing process.

We proceed with samples thermal treatment in vacuum for a few hours.

Results and discussion

In the following graph there are the samples T_cs vs the ratios between Sn and Nb magnetrons currents of some of the first samples deposited. The increase of the Sn quantity and/or of the annealing temperature imply an higher T_c value.

The best sample we got has $T_c = 17.9$ K, $\Delta T_c = 0.02$ K and RRR = 3.6.(Deposition conditions: I(Sn) = 0.22 A, I(Nb) = 2 A; p(Ar) = 5.0 x 10⁻³ mbar. Annealing: 1.5 h at 930°C).

All the superconducting properties measurements have been performed trough the resistive method.

Looking at the X Ray diffraction patterns, it is possible to state there is no evidence of the presence of low T_c spurious phases (Nb₆Sn₅ and NbSn₂) or unreacted Sn (see for example Figure 4).



Figure 3: T_c vs I(Sn)/I(Nb) of some of the samples deposited on sapphire (four contacts measurement). The increase of the Sn quantity and/or of the annealing temperature [from 600°C (square markers) to 930°C (triangle markers)] imply an higher T_c value.



Figure 4: X Ray diffraction pattern of one sample

POST MAGNETRON

Experimental Setup and first results

In the framework of the multilayer deposition used to obtain the Nb₃Sn A15 phase, we built another experimental apparatus. The Nb-Sn cathode is fixed at the bottom of a cylindrical vacuum chamber and it can be cooled down trough an internal coaxial water tube (Figure 5). The substrates (or the 6 GHz cavity) are fixed to a substrate holder connected to a linear feedtrough to move them up and down. An external coil is mounted in the sample zone: we use a magnetron sputtering technique in a post magnetron configuration.

The first sample has been deposited at $p(Ar) = 2 \times 10^{-2}$ mbar and setting up a magnetic field of about 450 Gauss. We sputtered for 30 minutes then the coating annealing has been performed in another chamber equipped with an external furnace. The film obtained is 700 nm thick and its superconducting properties are $T_c = 17.3$ K and $\Delta T_c = 0.13$ K (four contacts measurement).

The next step will be the deposition inside a 6 GHz cavity.



Figure 5: A schematic of the system built to coat Nb substrates or 6 GHz cavities using the post magnetron sputtering technique. On the left the complete vacuum chamber is shown, on the right the detail of the deposition zone.

Table 2:	Sputtering	deposition	parameters.
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Sputtering	Current	Voltage	Power
target	(A)	(V)	(W)
Nb/Sn	0.1	330	20

V₃Si BY THERMAL DIFFUSION

The V-Si system A15 phase forms congruently from the melt at 1935°C. Its homogeneity interval presents a maximum at 1800°C where it is stable between 19 and 25 at.% silicon. As shown in Figure 6, below 1200°C this range is limited to 24-25 at.% silicon [12]. V-Si T_c increases linearly while reaching the stoichiometric composition so the maximum critical temperature (17.1 K) is achieved with the 25 at.% of silicon [13]. The resistivity variation in normal state, before transition, has been studied in detail: at the stoichiometric composition RRR values of 80 are achievable [13.



Figure 6: V-Si phase diagram

Literature

 V_3Si thin films were obtained by thermal annealing under vacuum of a few vanadium and silicon layers sequentially deposited by electron beam evaporation. Resistive critical temperatures up to 16.2 K with very sharp transition widths were measured [14].

Another attempt to obtain superconducting V₃Si films has been made using the cosputtering method (facing target magnetron). There was no transition above 4.2 K in the case of coatings deposited at room T. This behaviour is related to the V₃Si films high disorder. Some samples, sputtered at room temperature, have been annealed at 800°C for one hour and a half and the best one got a RRR = 4.5 and T_c = 14.4 K.

The reactive sputtering of V-Si films by a DC facing target magnetron configuration in Silane/Argon atmosphere has been investigated too. The process temperature strongly influences the film composition as well as its quality. Stoichiometric superconducting V₃Si coatings have been obtained under different silane partial pressures and film deposition rates. The best samples (T_c max = 16.2 K, RRR max = 16.7) were sputtered at 500°C at a low deposition rate and then annealed at 800°C in a SiH₄ atmosphere [15].

Bearing in mind the just described experiments, V_3Si samples were grown by Si thermal diffusion into vanadium [16].

Bulk V annealed in a silane atmosphere gave good results. After an etching of about 40 μ m in a 1:1:1 acid solution, the V substrates were annealed up to 850°C in SiH₄ for 20 h and then in vacuum for 40 h. Films properties, obtained using the AC inductive measurement system, were encouraging: T_c close to 16 K and Δ T_c less then 0.4 K.

This work has been pushed ahead changing the samples process temperature, silanization time and annealing time in vacuum.

Experimental set up and method

The used vacuum system is equipped with a rotary preliminary pump and a turbomolecular one. The silane cylinder is contained in a constantly ventilated gas cabinet.

To optimize the parameters, we decided to do some experiments just with small bulk vanadium samples [17].

The V substrates are chemically etched before their mounting inside the bell jar. Many different solutions have been tried and finally we chose HNO₃ 65%, H₂O, NH₄F (25:12:1). The metal surface is smooth, it guarantees a controllable reaction with a good erosion rate (15,5 μ m/min) and its heating can be easily maintained under control.(20 \rightarrow 38°C).

Our bulk vanadium substrates are fixed in the chamber (trough Nb wires): they are then heated at high T (\geq 800°C) using a set of lamps (Figures 7-8), left in a silane atmosphere for some hours and annealed in vacuum for a certain period of time (generally tens

of hours). The sample T is monitored with a Chromel-Alumel thermocouple, positioned exactly on the substrate surface. SiH_4 pressure and process temperature and time have been progressively changed to get better superconducting properties.

The stand built for the lamps is represented in the following figure (number 7). In the photograph it is possible to see a 6 GHz cavity: the idea is to silanize such small resonators to test in a very fast and easy way the V_3Si RF properties. The heating system is surrounded by a shielding structure to improve the thermal performances, avoiding the chamber and the used gauges damages. Figure 8 shows the IR lamps working.



Figure 7: Samples heating system. In the photograph it is possible to see a 6 GHz cavity: the idea is to silanize such small resonators to test in a very fast and easy way the V_3Si RF properties



Figure 8: Samples heating system image. In the photograph it is possible to see two substrates with the corresponding termocouples

Results and discussion

The procedure to obtain V_3Si films counts two phases. During the first one the sample undergoes heating treatment at high temperature in a SiH₄ atmosphere. The Si coating formation starts: it progressively diffuses into the vanadium substrate to form the A15 compound. The process variables are: SiH_4 partial pressure, T and duration but also the metal surface finish and the base vacuum level are crucial for the experiment success. In the second part, a heating treatment in vacuum is performed. It has three different purposes: to push ahead Si diffusion, to promote the V₃Si film crystallization and to eliminate the trapped H₂ coming from SiH₄ decomposition.

Figure 9 is a sample SEM image: on the left the V substrate is clearly visible while, on the right side it is possible to see the obtained A15 film.



Figure 9: A V₃Si film SEM image. This sample has been obtained with $p(SiH_4) = 5x10^{-4}$ mbar, silanization time = 10 h and annealing time = 20 h).

The A15 is compact and its thickness is less than $1 \ \mu m$.

XRD analysis have been performed to verify the coatings compositions. Looking at the following figure (number 10), it is easy to distinguish pure V peaks and V₃Si ones. There are not other V-Si phases or material contaminations. V₃Si 3F has been silanized for 10 h at 825° C (p(SiH₄) = $5x10^{-4}$ mbar) and heat treated in vacuum for 20 h at the same T.



Figure 10: A V₃Si film XRD spectrum

Because of the desirable application of this compound to resonant cavities, it is really important to verify the superconducting properties of our samples. We used the PPMS (Physical Properties Measurement System) to achieve this goal. The $V_3Si T_c$ of the best samples is around 15.5 K.

We tried to coat a 6 GHz cavity: it has been silanized for 4 h at 850° C (p(SiH₄) = 5 x 10^{-3} mbar) and annealed for 6 h at the same T. The first RF test was unsuccessful: the possible explanation is the presence of unreacted Si on the cavity surface. We decided to measure it several times after a heating treatment in vacuum progressively longer (6h, 12h, 36h, 84h at 850° C).

The cavity quality factor progressively improves but it is still lower than the pure Nb one.

Plasma

Now we are studying the plasma role during the silanization process. In Figure 11 the SiH₄ plasma is clearly visible (IR lamps are off). Using the glow discharge we can induce the silane decomposition before its adsorbtion on the V substrate surface. The exact sequence of what happens in the chamber is still not clear but, using the semiconductor industry information we can understand something more [18].



Figure 11: SiH₄ plasma

One of the first samples has been obtained following the recipe written in Table 3. The process temperature is 850° C, the SiH₄ pressure is 10^{-2} mbar (mixing with Ar to reach 10^{-1} mbar) and the silanization time is 60 minutes (10 minutes x 6). The annealing has been performed for 2 h at 850° C.

The superconducting properties are: $T_c = 15.7$ K, $\Delta T_c = 0.2$ K. We are going to modify the used parameters to try to improve the films quality. A 6 GHz cavity will be coated too but it is necessary to build a correct shape electrode [18]: the distance cathode-anode is fundamental to be able to control the hydrogen presence into the film.

6 GHz CAVITIES

A large amount of samples can be produced but it is difficult to measure their RF resistance.

6 GHz seamless cavities, obtained by the spinning technique, can represent the solution. The following is a 6 GHz cavity photograph.



Figure 12: A 6 GHz resonant cavity

They are made from larger cavities fabrication remaining material, they don't need welding (even for flanges) and finally they can be directly measured inside a liquid helium dewar.

Nb cavities

The Nb bulk cavities are processed with:

- Mechanical polishing (ZrO₂)
- Chemical polishing (1:1:2)
- Electro-Polishing
- H_2O_2 washing

After every step, the cavity is heat-treated essentially to get rid of oxygen and hydrogen.

V cavities

Due to the spinning process, material mechanical stress is considerable and the damages caused by the mandrel are particularly evident.

The initial internal surface is extremely rough so, before the chemical treatment described in the case of samples (180" in a HNO₃ 65%, H₂O, NH₄F (25:12:1) solution), a mechanical polishing has been performed. We used a commercial tumbler and the chosen media are SiC and ZrO₂.

RF Test

The following image represents a cavity fixed to the stand built to test its RF properties (Figure 13). It is very simple, fast to use and it gives us the opportunity to make such small resonators become our samples.



Figure 13: A 6 GHz cavity mounted on the 6 GHz measurement system

CONCLUSIONS

*Nb*₃*Sn by Tin Thermal Diffusion*

The annealing procedure has been carried out in three different ways, in order to improve the coatings quality. The "hybrid process", consisting in a first annealing in presence of Sn vapour, followed by a second heating treatment in its absence seems to be the most promising. 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).

The best recipe has been used to coat a 6 GHz Nb cavity but Sn droplets are still present in the bottom part of the cell while it is not possible to see Sn traces on the cut-offs surfaces.

Presently we are going to modify a little bit the process to adapt it better to the more complicate cavity shape before performing RF tests.

In the meantime the "Double Furnace" System has been built to avoid air contaminations of the films due to the vacuum chamber opening.

The mechanical plating technique could be applied to the production of superconducting cavities. We filled a 6 GHz Nb cavity with Sn pieces: it has been tumbled, annealed for 1h at 950° C and measured.

Unfortunately the Q value of the cavity is the one of pure Nb. In the next trial we are filling the resonant structure with Sn powder and zirconia balls hoping to increase the Sn deposited and its adherence on the surface.

Nb₃Sn by Multilayer Deposition

The purpose of this research is to demonstrate it is possible to get Nb_3Sn thin films depositing Nb and Sn

by magnetron sputtering and then annealing the obtained multilayer structure.

Preliminary results are very promising as the maximum T_c is 17.9 K and the transition are usually very sharp. XRD characterizations confirm this result. We are going to apply this technique to RF superconductive cavity as soon as possible.

In the framework of the multilayer deposition, we use a magnetron sputtering technique in a post magnetron configuration too.

The Nb-Sn cathode is fixed at the bottom of a cylindrical vacuum chamber. The substrates (or the 6 GHz cavity) are fixed to a substrate holder connected to a linear feedtrough to move them up and down. An external coil is mounted in the sample zone: The first film obtained is 700 nm thick and its superconducting properties are $T_c = 17.3$ K and $\Delta T_c = 0.13$ K (four contacts measurement).

The next step will be the deposition inside a 6 GHz cavity.

V₃Si

The Si thermal diffusion trough bulk vanadium to form the A15 V_3 Si has been introduced and studied at INFN-LNL a dozen of years ago. We are trying to push ahead that work changing the substrate heating system, the samples process temperature, silanization time and annealing time in vacuum.

Encouraging preliminary results have been obtained: for the most of the coatings T_c is around 16 K ($\Delta T_c \approx 0.2$ K).

We obtained a V₃Si 6 GHz cavity: it has been silanized for 4 h at 850°C ($p(SiH_4) = 5 \times 10^{-3}$ mbar) and annealed for 6 h at the same T. The first RF test was unsuccessful: the possible explanation is the presence of unreacted Si on the cavity surface. We decided to measure it several times after a heating treatment in vacuum progressively longer (6h, 12h, 36h, 84h at 850°C).

The cavity quality factor progressively improves but it is still lower than the pure Nb one.

Now we are studying the plasma role during the silanization process.

The superconducting properties of one of the first samples are: $T_c = 15.7$ K, $\Delta T_c = 0.2$ K. We are going to modify the used parameters to try to improve the films quality. A 6 GHz cavity will be coated too but it is necessary to build a correct shape electrode: the distance cathode-anode is fundamental to be able to control the hydrogen presence into the film.

6 GHz cavities

We are perfectly aware that having satisfactory results with samples doesn't mean obtaining good superconducting cavities with ease. That's why we decided to look for the best way to overcome this problem. Our solution is to work directly with cavities. Obviously it would be foolish to use 1.5 GHz bulk Nb resonant structure: it would be too onerous both for the material cost and the cryogenic expense. The idea is to build 6 GHz cavities. They are made from larger cavities fabrication remaining material, they don't need welding (even for flanges) and finally they can be directly measured inside a liquid helium dewar. It is possible to perform more than one RF test per day.

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