Nb_3Sn FILMS BY MULTILAYER SPUTTERING

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Abstract

The common limitation of systems conceived for the RF characterization of samples consists in the difficulty of scaling the measured results to the real resonator. Our idea consists in the production of small resonators (6 GHz cavities Figure 6) completely equal in shape to the real scale model. Performing RF tests on a large amount of cavities it is possible to study alternative thin film superconducting materials, traditional and innovative surface treatments.

In this framework Nb_3Sn films have been deposited at first on planar samples and then on the internal surface of 6 GHz cavities through the multilayer sputtering method.

The UHV magnetron sputtering technique in planar configuration has been used to deposit multilayer of niobium and tin on planar samples.

Then a UHV magnetron sputtering technique in a post magnetron configuration has been used. A Nb-Sn cathode is maintained in a fixed position, while the 6 GHz resonator can be moved up and down thanks to a linear feedtrough. The external coil is mounted in the cathode zone. The cavity is then annealed at 960°C for a few hours (UHV) to obtain the stoichiometric A15 phase. The surface resistance is evaluated through the cavity quality factor measurement at 4.2 K.

SAMPLE DEPOSITION BY PLANAR MAGNETRON

The aim of this experiment is to demonstrate that it is possible to obtain Nb_3Sn thin film depositing and heating multilayers of Nb and Sn by magnetron sputtering [2].

The multilayers depositions are performed in a cylindrical stainless steel vacuum chamber 280 mm high and with a diameter of 100 mm, equipped with two planar balanced magnetrons sources (Figure 1). On top source is mounted a 2 inches niobium target and on the bottom source a 2 inches tin target.

The sample holder is in the centre of the chamber and it composed by an aluminium disk connected to a rotary feedthrough. In this way we can rotate the samples, and every 180° rotation we expose the sample to Nb plasma before, and Sn plasma after.

The distance between targets and samples is 60 mm and during the sputtering we use Argon as process gas (P = $2.2 \cdot 10^{-3}$ mbar). No substrate heating systems were employed. The sample annealing is performed after the deposition, ex-situ, in another chamber similar to this one connected at the same pumping system, but with a heater, which is composed by an Inconel cup with inside an infrared lamp that permits to heat the sample up to 1000 °C.

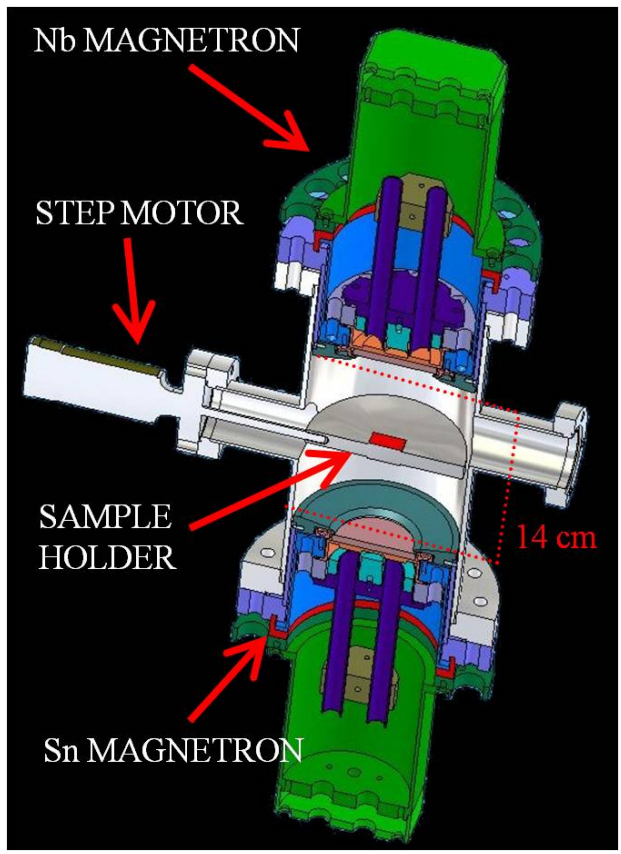

Figure 1: Sketch of the UHV multilayers deposition system.

Deposition Process and Annealing

During the deposition the angular velocity of the sample is constant, in particular it makes a complete round in 13 second.

The samples used are sapphire with dimension of 10x10 mm.

During multilayers deposition the exposition time of sample at Nb plasma and Sn plasma is equal and so it is important to find the correct current value that must be supplied to each magnetron source. To obtain, after annealing, A15 structure of Nb_3Sn it is very important that stoichiometry of Nb a Sn is respected during

deposition. In particular we calculate that thin film thickness of Nb must be 4.5 times the thickness of Sn film.

Before the multilayers deposition the chamber is evacuated at a base pressure of 1.5×10^{-9} mbar and during process Ar is used as process gas at a pressure of 4.0×10^{-3} mbar.

To obtain Nb/Sn multilayers, with a single deposition run, in a first step is deposited Nb buffer layer for 2 minutes in order to eliminate Oxygen contamination in the chamber. After we deposit simultaneously Nb and Sn for 30 minutes and in the last step we deposit another time an overlayer of Nb in order to have a "thick" Nb film as last layer.

This is important because Sn as a high vapour pressure and during post annealing if we don't have Nb film may happen that Sn evaporate and stoichiometry is not respected.

Sn magnetron is supplied with 0,22 A and Nb magnetron with a variable current from one run deposition to another as summarized in Table 1. For each run we deposited four samples.

Table 1: Sputtering deposition parameter.

Run	1	2	3	4	6	7	8
I(Sn) [A]	0,22	0,22	0,22	0,22	0,22	0,22	0,22
I(Nb) [A]	0,8	1	1,2	1,4	1,6	1,8	2

After sputtering deposition we proceed with annealing in vacuum for 3 hours at variable temperatures: 600-700-800-930°C as summarized in Table 2.

Table 2: Annealing parameter.

Annealing Process	1	2	3	4
T [°C] Annealing	600	700	800	930
t [h] Annealing	3	3	3	3

Each annealing process consists of 8 samples from the 8 different deposition runs.

Before heating vacuum chamber was evacuated at $9\cdot10^{-8}$ mbar in order to reduce oxygen contamination.

Results and Discussion

Multilayers thin films are analysed with X Ray Diffraction (XRD) after annealing.

The diffraction pattern (Figure 2) obtained after the annealing process at 930°C highlights the presence of the Nb_3Sn phase. The peaks shown on the plot are the A15 phase ones.

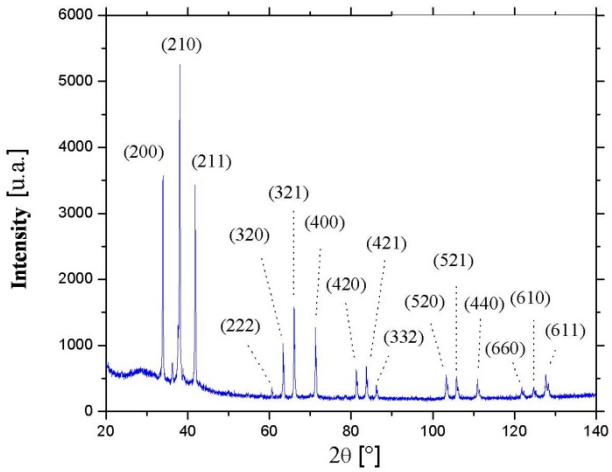

Figure 2: X Ray diffraction pattern of one of the best sample deposited with high ratio I(Nb)/I(Sn) and annealed at 930°C.

After this we measured superconductive characteristics of thin film, as can see in the plot of Figure 3, for each sample with the four point contact method.

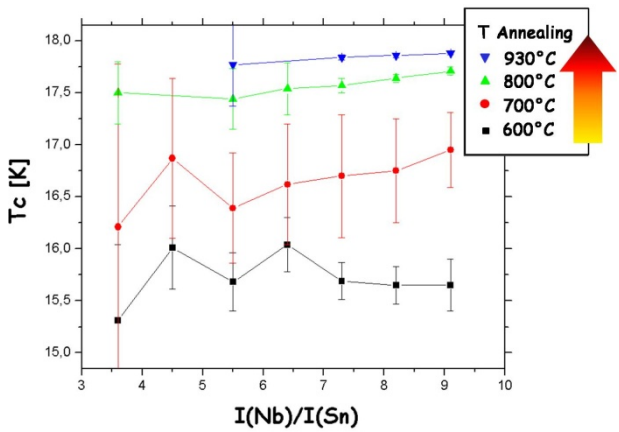

Figure 3: Superconductive transition on Nb_3Sn thin film obtained by multilayers deposition of Nb and Sn by sputtering and diffusing.

The superconductive transition T_C, for the best sample is at 17.88 K with a ΔT_C of 0.02 K and RRR of 3.58.

Preliminary results are very promising as the T_C of the film is 17.88 K (close to the theoretical one 18 K) and the transition is very sharp. XRD characterization confirms this result.

6 GHz CAVITIES – MINI LAB

The idea, to perform a high numbers of RF tests on superconducting materials and at same time to reduce research budget, is to build 6 GHz cavities. RF measured samples will never be comparable to a real large cavity. It is always an indirect measurement. On the contrary 6 GHz cavities are at the same time easy to handle as a sample but they are "real" cavity.

They are made from larger cavities fabrication remaining material using spinning technology [1], they

don't need welding (even for flanges) and finally they can be directly measured inside a liquid helium dewar. While 1,3 – 1,5 GHz cavities need for the RF test no less than 1 week time preparation. With 6 GHz cavities it is possible to perform more than one RF test per day.

With a tool like this it is possible to study traditional and innovative surface treatments and to perform RF tests on a large amount of cavities with a research budget much lower than the one necessary to treat and tests real cavities. One can study traditional surface treatments on Nb like: mechanical polishing, buffer chemical polishing, electro polishing, high pressure rinsing (HPR), alcohol rinsing and thermal treatments. It is also possible to study new thin film superconducting materials grown for example by sputtering or thermal diffusion.

In this paper is shown how is possible, with a reduce budget, to set up a Mini-Laboratory able to perform all the traditional treatments necessary on niobium high gradient large cavities and the deposition of Nb_3Sn films by multilayer sputtering inside it:

- A reduced size mechanical polishing bench
- A chemical/electrochemical minilab for BCP EP
- An inexpensive cryogenics and quick RF measurements

Figure 4: On the right a scrap, of a large Nb cavity, from which are obtained 4 small 6 GHz resonators from the 4 corners.

Geometry and Fabrication Technique

6 GHz cavities are made trough the spinning technology fig... that represents a valid alternative to electron beam cavities production, also for real resonators.

The 6 GHz cavities produced by spinning are obtained using larger cavities fabrication remaining material "scraps" as shown in Figure 6, they don't need any kind of electron beam welding (even for flanges) as shown in Figure 7, finally their production require a short fabrication time, a half day per cavity. For these reasons it is possible to control the costs production and realize a large amount of cavities with a low research budget.

6 GHz cavities are 97 mm long and have a 45 mm diameter cell, an *electrical length* of 25 mm and the same *geometrical factor* of a large resonator, 287. They have two large flat flanges at the ends. For each of them the available surface to ensure the vacuum sealing is equal to 7 cm^2.

Reduced Size Mechanical Polishing Bench

The spinning process implies material surface defects, stress and dislocations. The cavity cell is characterized by the presence of evident vertical scratches due to the used mandrel. Obviously the internal surface finishing of a resonant structure is directly correlated to its performance, especially at high fields. Moreover the lubricant, necessary for the metal mechanical processing can contaminate the used material. The idea is to make the surface smooth and free from contaminants.

In order to remove surface roughness and contaminations introduced during the spinning process, we use a compact and portable tumbler in spite of the bigger and complicated designed ad hoc for large cavities.

Chemical/Electrochemical Minilab for BCP EP

Following the classical and well known surface treatments general protocol of large cavities, after the mechanical polishing the procedure counts a chemical polishing. Chemical treatments are performed to smooth further on the cavity surface, to remove the possible niobium sub-oxide and contaminants.

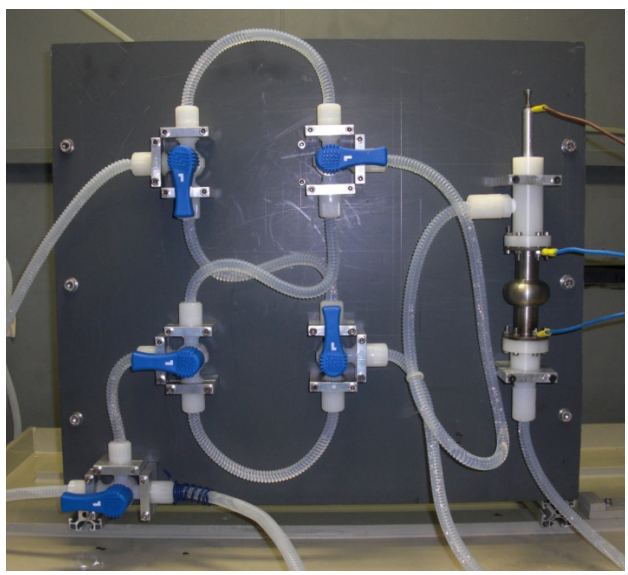

Figure 5: In this picture is visible the mini-chemical system set up for BCP and EP. In particular can be seen on the right a 6 GHz cavity installed in vertical position, equipped with special flanges for EP. The acid flux is directed from the bottom to the top of the cavity in order to evacuate the hydrogen, produced during the process, quickly. The 3-way valves are useful to invert the flux direction.

Chemical treatments for large resonators require tens of litres of sulphuric acid, hydrofluoric acid, phosphoric acid and nitric acid handled with big and complex

infrastructures. On the other hand, just a few litres of acids (2-3l) are needed for 6 GHz cavities using a simple and easy to handle infrastructure. This means a drastic reduction of acids and materials costs and also a considerable reduction of human risks and time processing.

To perform the traditional surface chemical treatments, buffer chemical polishing (BCP) and electrochemical polishing (EP), on a 6 GHz cavity can be used a small system as the one reported in Figure 5.

For electrochemical polishing the aluminium cathode used, that enters inside the cavity, is shaped to maintain constant the cathode – internal surface cavity distance.

For electrochemical polishing and buffer chemical polishing are used simple holed PVDF (poly-vinil-dene-fluoride) flanges. The flanges are expressly designed to obtain the highest acid flow through the cavity to allow hydrogen bubbles, produced during the oxi-reduction reaction, to escape freely.

The evaluations of damaged layer thickness produced by the spinning process could vary from 150 to 250 μm. For this reason it is convenient to remove with BCP/EP at least 300 μm: in average this thickness corresponds to about 30 g of removed material.

During a mechanical or a chemical treatment, for each cavity, it is easy to stop the process and monitor the internal surface finishing with the help of a miniature camera insertion device.

CRYOGENICS AND RF MEASUREMENTS

To perform the 6 GHz cavities RF tests has been designed and built a compact and user friendly cryogenic infrastructure. It has been conceived to enter a 450 or 250 l liquid helium dewar.

Such an infrastructure permits to reduce the cryogenic power consumption, avoiding any loss during a helium transfer into a cryostat for common large cavities: a measurement performed at 4.2 K requires roughly 20 litres of liquid He. The procedure to cool down and to warm up the system is very fast.

To oversimplify the system assembling, the bottom part (Figure 7) of the stand has been made completely independent. The small resonator can be mounted, in clean room, without the presence of the complete stand that would make the work complicated.

The bottom part consists of two main elements that close the cavity: the Coupler Flange and the Pick-up Flange. Coupler Flange is a stainless steel disc with 8 holes for M4 s.s. screws and a CF 16 knife edge on one side. It is equipped with a 8 mm pumping line (Coupler-Pipe) and a bellow that permits the motion of the coupler antenna. The last is connected to a CF 16 - SMA coaxial feedthrough. Four teflon coated vertical s.s. bar lines preserve the system alignment and prevent blockage problems due to the freeze-over of the bellow upper flange during the liquid helium insertion step. Pick-up Flange is again a stainless steel disc with 8 holes for M4 s.s. screws and a CF 16 knife edge. It is equipped with a 8 mm diameter pumping line (Pick-up-Pipe) and a Swagelock assembly for the pick-up antenna insert (SMA ended).

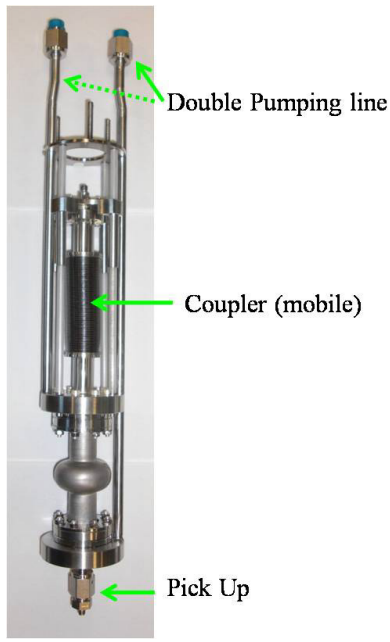

Figure 6: The bottom part completely mounted.

The cavity is closed at both sides through the Cp/Pk Flanges with a particular double system. The vacuum between the Pick-up Flange and the Middle Flange (equipped with 8 threaded holes) is guaranteed using a CF-16 copper gasket using 8 s.s. screws. Vacuum between the Middle Flange and the cavity flat borders is guaranteed using a 1.5 mm diameter indium wire squeezed with four stainless steel half round rings pressed through 8 nuts screwed to the same screws or with a new type of vacuum seals made of Kapton as reported in Figure 8.

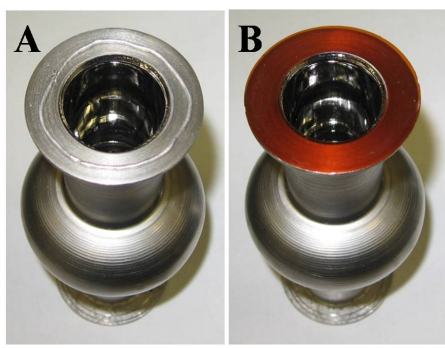

Figure 7: A classical Indium gasket. B a new Kapton Gasket, it is cleaner and easier to remove than Indium.

The stand is slowly inserted into the helium dewar using the overhead travelling crane and cooled at the temperature of the liquid helium 4,2 K. The surface resistance value of the superconductor under test is

obtained through the measure of quality factor of the cavity using the equation

$$R_S = \frac{G}{Q}$$

where, as mentioned in the first chapter, G is the geometrical factor. It is the same for 1,5 GHz and 6 GHz cavities and it is equal to 286.

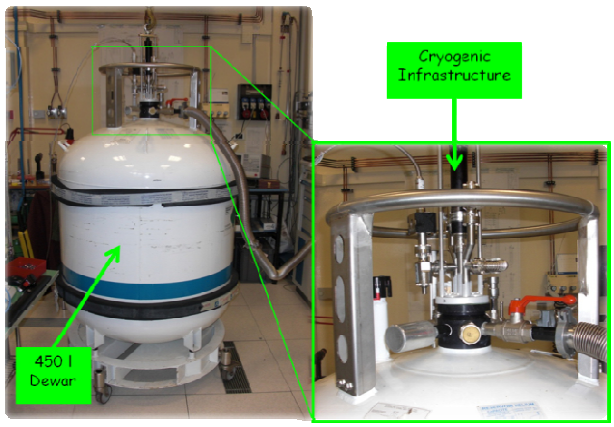

Figure 8: The 450 l helium dewar containing the cryogenic infrastructure during the cooling procedure before the RF test.

CAVITY DEPOSITION BY CYLINDRICAL MAGNETRON

With 6 GHz resonators it is possible to study any kind of superconducting material that could be deposited inside it. From this point of view it is possible to build systems like the two depicted in Figure 9.

This miniaturized sputtering system allows to coat the inner surface of a 6 GHz cavity with multilayers of Nb and Sn. The resonator is fixed to a linear feedthrough and during the deposition is moved up and down toward the cathode to assure the complete and homogeneous coating.

As shown in Figure 16 and 17 the cathode consists in a fix long stainless steel cooled tube ended with a composite Nb/Sn target. It is, except for the target, completely screened with a grounded second external tube. The cathode diameter is 17 mm while the diameter of the cavity is 20 mm so it can be moved coaxial to the cathode without touching it.

The target, that is equipped with two Nb wings, is positioned in the centre of a strong axial magnetic field produced by the coil. The wings along with the magnetic field enhance the sputtering rate. Sn/Nb cathode area ratio is decided on the basis of sputtering yields to obtain multilayers films with different percentage of tin.

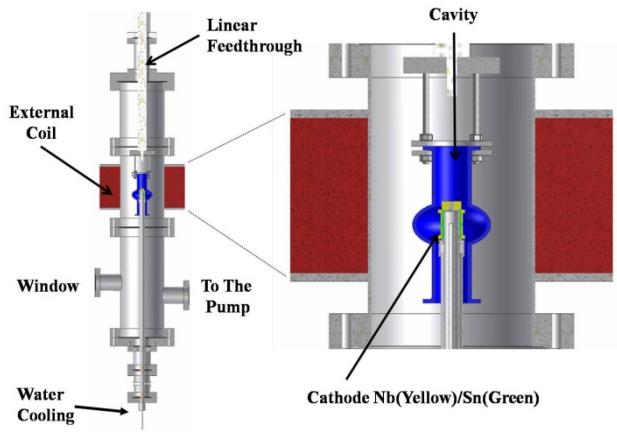

Figure 9: Sketch of the system built to coat 6 GHz cavities using the post magnetron sputtering technique. On the left the complete vacuum chamber is shown, on the right the detail of the deposition zone. The coil (red) is out of the chamber.

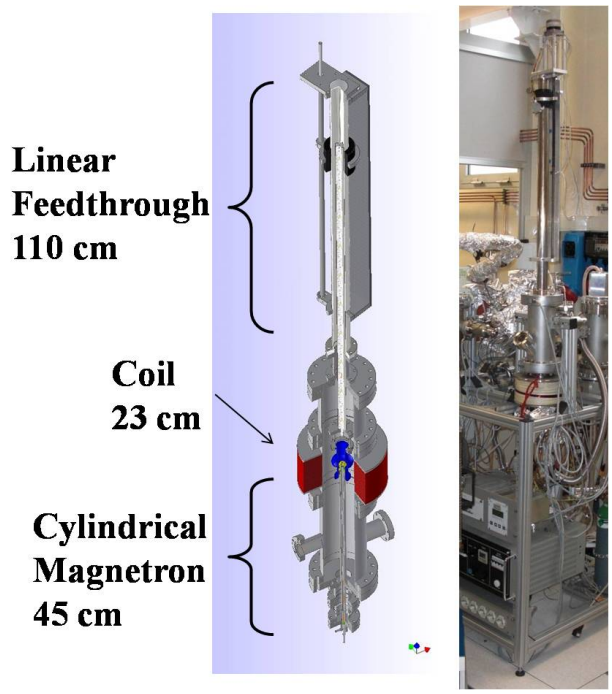

Figure 10: The sputtering system.

Deposition Process and Annealing

Before the multilayers deposition the chamber is evacuated at a base pressure of 1.5×10^{-9} mbar.

The cavity is moved up and down until the film formation, 5 hours for each cavity deposition with a thickness film of some micron. Then the cavity is removed from the vacuum chamber and mounted in the annealing system which is a cylindrical chamber made of Inconel. It is divided into two zones: the cold one on the top (cooled by a water flux) and the hot one at the bottom.

Before the annealing the chamber is evacuated at a base pressure of 1.5×10^{-9} mbar. The annealing is performed at T=960°C using an external furnace for three hours. The cavity temperature is monitored through a chromel-

alumel thermocouple. At the end of the annealing step the cavity is rapidly moved up in the cold zone to reduce the probability to have spurious low T_c phases (Nb_5Sn_6, $NbSn_2$).

After the annealing the cavity is rinsed with HPR for 10 minutes and mounted in the cryogenic infrastructure in clear room to be rf tested at 4,2K as described before.

Results and Discussion

The first Q vs Eacc curves we obtained are depicted in Figure 11. The Q value is still lower than the pure Nb one ($Q_{BCS}(4.2K) \approx 3 \times 10^7$).

We are going to analyze the internal surface of the cavity we gained and to optimize the process parameters. With 6 GHz cavities we have the best tool to improve fast.

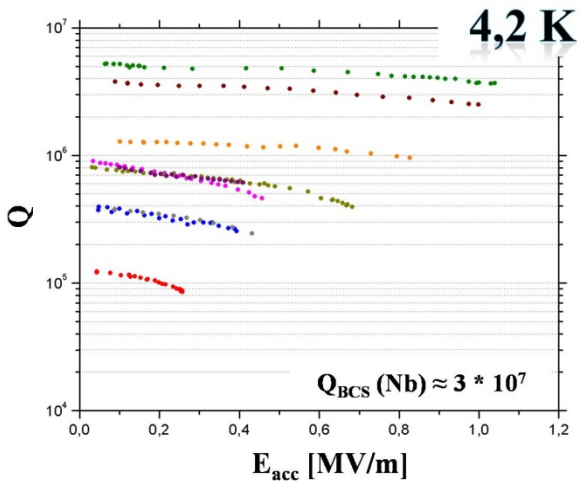

Figure 11: Q Vs Eacc curves.

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