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THE WAY OF THICK FILMS TOWARD A FLAT Q-CURVE IN SPUTTERED CAVITIES *

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Abstract

Thick films have bulk like properties. In this paper it is explored the possibility to sputter 70 micron thick films in order to get rid of the Q-slope in Niobium sputtered Copper Cavities. An innovative method based on the multilayer deposition of zero-stress single layers is reported. The deposition of zero-stress thick films into 6 GHz Copper seamless cavities, has shown the possibility to obtain straight curves for the Q-factor versus accelerating fields.

INTRODUCTION

This work will describe how the approach of thick films could be useful in order to solve the notorious problem of the Q-slope of Niobium thin films sputtered Copper cavities. At the actual point of technology, if the problem will be not solved, the film sputtering technology risks to be discarded from being adopted for next projects of accelerating machines.

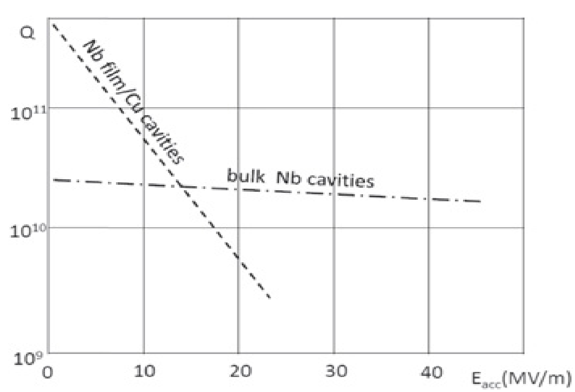

Figure 1: Classical behavior at 1.5 GHz and 1.8K of Nb thin film sputtered Cu cavities compared to Niobium bulk cavities [1].

However, it must be remarked that the slope of the Q-Factor versus the accelerating field, E_{acc} will certainly depend from the film deposition and not from the Niobium-Copper system. There is indeed experimental evidence that Niobium-clad Copper Cavities display a flat Q-factor and they reach accelerating fields up to 40 MV/m [2].

Niobium-clad cavities and Niobium sputtered cavities differ mainly for two factors: the Niobium thickness and the Niobium interface. For clad cavities indeed the thickness is normally hundreds of times higher than for films. Also the interface is incommensurably better for clad Cavities, since the explosive bonding makes the Niobium physically interdiffusing into Copper with almost a perfect joining at the interface.

So, how a high thickness of the Niobium film deposited onto Copper could affect cavity RF performances? One possibility is connected to the fact that a thick film has a value of the Residual Resistivity Ratio (RRR) higher than that of a thin film. The reason for that is at least twofold: The most evident reason is that a Niobium thick film will have larger grains than a thin film, so that the electron mean free path will be not limited by the thin thickness. Another possible reason is that if there is any impurity diffusing from the substrate, the inter-grain percolative path up to the film top surface is much larger in a thick film rather than in thin films.

Bulk Niobium sheets and also Niobium clad Copper sheets, can be purchased with RRR values in the range from 250 to 300, while thin films have often RRR values mainly around 30. Now a lower value of RRR could directly affect the Superconducting gap, and, in turn, the Rs value [3].

A further possible mechanism by which a thick film could perform better than a thin film, is the following: for a defect located at the Nb/Cu interface, in case of a thin film the produced heat flow will be transmitted unidimensionally from the film to the Cu substrate. In thick films instead the heat transmission occurs three-dimensionally, and the heat flux can easily shunt the defect at the interface [4].

Niobium Copper interface is by sure a crucial element in determining cavity performances and Q-slopes, and this has been first proposed in a previous paper by some of the authors [5]. A defected thermal contact at the Nb-Cu interface is by sure responsible of a Q-slope. And the quality of the interface depends from the film stress, that directly acts on the film adhesion to the substrate. The quality of interface is even more jeopardized in case of Niobium and Copper, because these two metals have no miscibility below 1080°C in any range of the phase diagram. Now, a thick film will be adherent to the substrate, only if it will be absolutely not stressed.

Therefore, a higher purity and a better Nb-Cu interface would push to explore thick Niobium films rather than thin Niobium films for the coating of Copper cavities.

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EXPERIMENTAL PROCEEDURE

A better Niobium interface would be achievable for a stress-free film. Stress can be tensile or compressive as shown in Fig. 2. As soon the film starts to nucleate on the substrate, opposite forces appear on the substrate.

If the film surface is smaller than that of the substrate, in order to grow, the film must adapt to the substrate under tensile forces. If instead the film surface is larger than the substrate, in order to grow, the film must adapt to the substrate under compressive forces.

A useful method to see if the film stress is compressive or tensile consists in depositing Niobium onto a thin substrate like extremely thin sapphires or onto a flexible substrate like a kapton foil. Then one must go to see in which direction the coated foil will bend. Of course a stress-free film will remain absolutely flat.

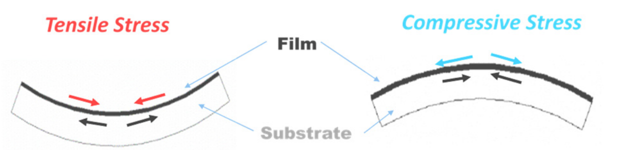

Figure 2: Tensile and Compressive Stress in a film sputtered onto a substrate. It is clear that a tensile stress in film, will correspond to a compressive stress in substrate surface and vice-versa a compressive stress in the film will correspond to a tensile stress in substrate surface.

The film intrinsic stress depends mainly on three parameters: the substrate temperature, the sputtering pressure, and the film thickness, even if other parameters like the target orientation, the bias potential, and the cathode atom/gas mass ratio should also be taken into account.

In any case, if we limit at the most crucial parameters, the role of substrate temperature is rather straightforward: the higher will be the temperature, the lower will be the film stress. Referring instead, to the role of the sputtering gas, the dependence is a bit more complex. As shown in Fig. 3, there is a critical pressure across which, the film intrinsic stress inside the film passes from compressive to tensile, because stress depends on the energetics of the deposition process.

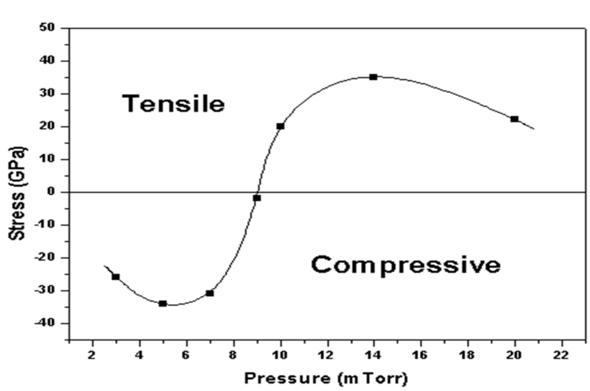

Figure 3: Typical behavior of the change in stress as a function of the gas pressure [6].

Indeed, tensile stress is generally observed in Thornton zone 1-type, porous films and it is explained in terms of the grain boundary relaxation model, whereas compressive stress, observed in Thornton zone T-type, dense films, is interpreted in terms of the atomic peening mechanism. At low pressures, the sputtered and reflected neutral atoms have much higher energies than the plasma gas between the target and substrate and arrive at the substrate with super-thermal energies. The sputtered atoms in this situation have a high surface mobility at the substrate. At higher sputtering pressures, the sputtered and reflected neutral atoms, are thermalized by the plasma gas, due to the increased number of collisions before arriving at the substrate. As a consequence, the sputtered atoms will have a lower surface mobility at the substrate.

A third important parameter determining film stress is the film thickness. There is a critical thickness where the stress passes from compressive to tensile (Fig. 4).

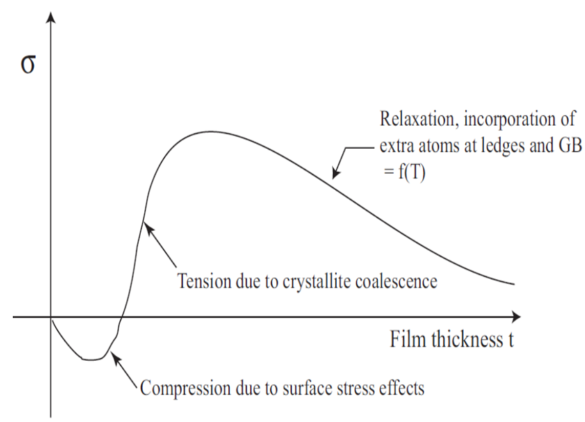

Figure 4: The intrinsic stress induced in a film depends on the film thickness.

A zero stress film must have the right thickness where there is the stress transition. This critical thickness is rather low, but if a multilayer of thousands thin layer at zero-stress will have also zero-stress. So, it is possible to deposit a very thick film, by

- depositing a thin layer of a few hundreds of nanometers;
- then stopping the process in order to give to the film the time to renormalize;
- and then depositing a new layer on the old one, and so one for thousands of times, in a similar way as it is done in Atomic Layer Deposition (ALD).

The depositions have been carried out on 6 GHz seamless Copper cavities produced by spinning technique. The internal surface was prepared as following:

- 1 hour mechanical grinding;
- degreasing in ultrasonic bath;
- vertical Electropolising (EP) with 3:2 ratio Phosphoric Acid : n-Butanol solution;
- chemical Etching: 5 minutes in SuBu5 solution at 72°C [7];
- passivation: 2 minutes in Sulphamic Acid 20 g/l;
- high pressure deionized water rinsing at 100 bar.

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The magnetron sputtering deposition set up is composed by a Nb post magnetron 10 cm long, covering from flange to flange the length of a 6 GHz cavity. The target is water cooled and an external coil is used to produce a magnetic field of 800 Gauss. A constant deposition temperature, of 550 °C, is obtained by a ring shaped IR lamp, placed in proximity of the cavity cell. It is well known that high substrate temperature improves the morphology of the growing film [8]. Indeed a similar substrate temperature was used with success for the ALPI QWR resonators deposition [9]. The pressure value has been optimized with several depositions on kapton substrates at different pressures. Looking at the foil curvature after the deposition, we are able to say if the film is affected by tensile or compressive stress. In our configuration the minimum stress has been obtained at $5*10^{-2}$ mbar. At the moment, the film thickness has not been yet optimized; we fixed a standard value for all the depositions in order to compare the influence of the other deposition parameters. Total thickness of sputtered films is 70 µm on the cell and more than 100 µm on the cavity cut-off tubes. Deposition time is 9 hours. With this sputtering configuration we are able to growth films thick more than 1 mm (see Figure 5)

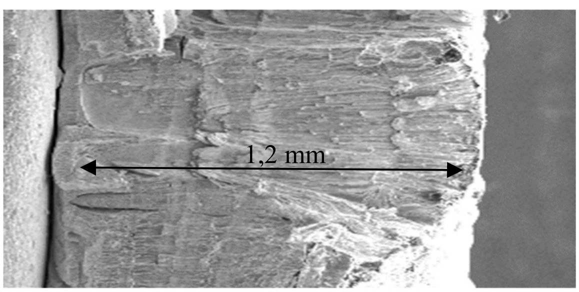

Figure 5: Nb film, deposited by magnetron sputtering, with a thickness of 1.2 mm.

The films are grown in two different ways: one shoot deposition and multilayer deposition.

In multilayer deposition, the power supply is switched on and off continuously, with a fixed duty-cycle of 0.7, as we can see in Fig. 6. The idea is that the pause time works as a sort of self-annealing, in which the adatoms have the time to rearrange on surface and reduce the film stress.

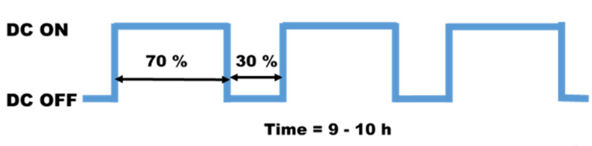

Figure 6: Schematic of the multilayer process. The Duty-cycle has been set to 0.7.

The deposition is carried out with a homemade software, written in Labview®, able to drive the power supply and to control all the other sputtering parameters. We have explored on cavities performances, the effect of different single layer thickness (100, 300, 400, 500 nm), keeping fixed the total film thickness. The single layer thickness should be a compromise between SRF properties (that increase with thickness) and stress (that could be reduced decreasing the thickness).

RESULTS AND DISCUSSIONS

Over 20 seamless 6 GHZ Copper cavities have been coated by Nb thick films. The cavities have been characterized by rf test at 4,2 k and 1,8 k, in order to see the behavior of the cavity quality factor Q_0 versus the gradient E_{acc}. An interesting aspect involves the Q-slope of the multilayer films. Figure 7 shows the Q vs E_{acc} curves at 4.2 K of 4 different cavities, all with 70 µm Nb thick film deposited in multilayer configuration, but with different single layer thickness. If we plot the slope value as a function of the single layer thickness, we obtain the interesting behavior visible in Figure 8. Further investigation is needed, but, from our preliminary results, the Q-slope can be modulated simply varying the single layer thickness.

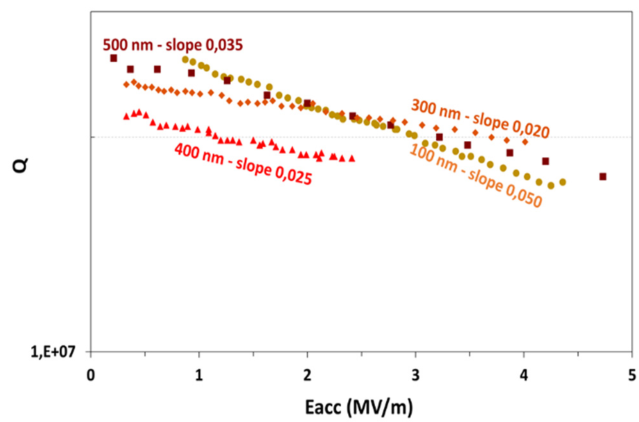

Figure 7: Q vs E_{curve} measured at 4.2 K and 6 GHz for different single layer thickness.

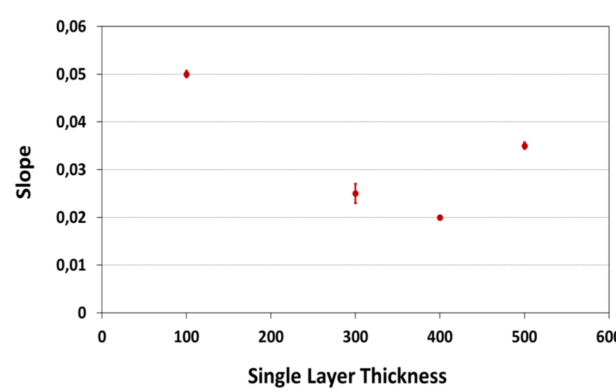

Figure 8: Curve slope in function of single layer thickness, referred to the measures of Figure for multilayered 6 GHZ cavities. Thickness units are in nanometers.

Three thick film cavities have shown a quite flat Q-value versus the accelerating field and are undoubtedly the 6GHz Nb/Cu cavities with the best performances ever

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obtained before (Fig. 9). For the first time a Nb/Cu sputtered cavity does not suffer the Q-slope problem.

Two cavities were sputtered in one shoot mode and one with the multilayer approach. For this last cavity, a 24 hours baking at 120°C was done with an improvement on the maximum accelerating field, a flatter Q-curve, but a sensible decrease of the Q-value at low fields.

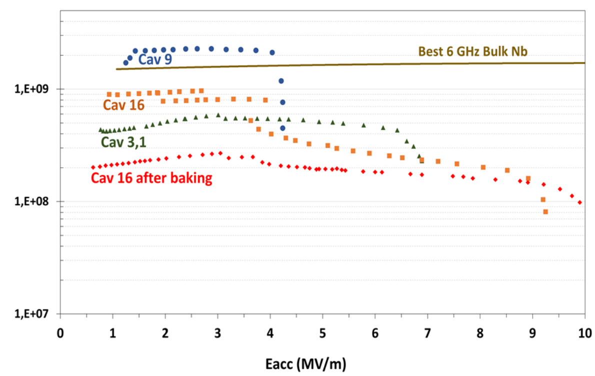

Figure 9: Q vs E_{curve} at 1.8K of the 3 best thick Nb/Cu 6 GHz cavities. Cav 3.1 and Cav 9 are sputtered in one shoot mode, Cav 16 are sputtered with the multilayer approach. On Cav 16 a 120°C baking was done for 24 hours.

CONCLUSIONS

Niobium Sputtered thick films are worthwhile to explore in order to solve the problem of Q-slope in thin film cavities. Depositing 70 micron multilayers made by hundreds of single zero-stress layers of 400 nm thickness, flat curves of Q-factor versus accelerating fields are obtainable.

Those reported are preliminary data, but very interesting, because they prospect a possible way toward the solution of the Q-slope problem in sputtered cavities.

A future strategy can consist in optimizing the results already got for 70 micron thick film cavities, in order to achieve the highest possible accelerating field. Once obtained that, a meticulous work for reducing the Niobium film thickness is mandatory. In this way, it should be possible to determine the minimum thickness required for having flat Q-curves versus E_{acc}.

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