INFLUENCE OF ELECTROPLATING PARAMETERS ON QUALITY OF LEAD COATINGS ON COPPER

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Abstract - We have investigated the role of deposition parameters as substrate temperature and plating rate on the superconducting properties of Lead. Cavities having a Q_0 up to 4 x 10⁸ and an accelerating field of 3 MV/m were prepared. Still room for improvements seems to exist. A simplified recipe for the passivation of plated surfaces is proposed.

Introduction

For such RF applications where not high level performances are required, in particular at low frequencies (BCS losses are low), the possess of Lead electroplating technology can allow the achievement of fast results, with modest equipment and in a relatively cheap way.

The low temperature RF performances of Lead Copper resonators are heavily influenced by the coating structure. The aim of our work was to try to find a correlation between the plating deposition parameters and the superconducting characteristics of the material. For Lead plating the amount of variables to control are paradoxically higher than for Niobium sputtering and our results are only preliminary. Nevertheless they pursued us, that by a deep control the deposition process there is still room for improvement.

The Lead plating

It is well-known that fluoborate solutions for Lead plating have efficiency close to 1. We coated the interior of a dummy resonator

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with several Copper samples, then after plating, the thickness of each sample.was measured. Fig. 1 reports the thickness distribution.



Fig. 1 Thickness profile of Lead coating in a dummy resonator equipped with Copper samples.

By means of small samples we also investigated the role of the substrate temperature, thickness and deposition rate on the Lead RRR. The depositions were performed in direct current without polarity reverse. From the data in fig. 2 and 3 it appears a net trend for RRR improvement at large thicknesses. Obviously large thickness means large grain sizes that means smaller resistivity.

Nevertheless the RF test performed indicate the opposite trend, the higher is thickness, the poorer are RF performances. In our opinion that is related to the fact that any lattice defect, microparticles on the surface are enlarged by large thickness and its effect on the resonator performances becomes amplified.



FIG. 2 Residual Resistivity Ratio values: a) versus deposition rate, b) versus substrate temperature.



Fig. 3 RRR values versus thickness for different substrate temperature.

By RF tests it was also seen a trend for lower deposition rates resulting in higher accelerating fields and higher Q-factors. Analyses by an electronic microscope show that lower deposition rates give rise to smooth and flat Lead surfaces.

Lead or Lead-Tin?

An important question that we encountered was the choice of the plating material. Lead has several stable oxides and hydroxides, so it is difficult to keep Lead surfaces free from oxidation. Lead-Tin alloys are on the contrary very stable and have been also successfully employed for low beta structures at Munich and at Stony Brook[1-2].

We coated several Copper samples by Lead, by Lead-Tin 2% and by Lead-Tin 4%. The classical Fluoborate bath was used The solution were Tin charged with the assumption that the plating efficiency for Lead and for Tin were equal i.e. that on the sample we could find the same percentage of Tin adopted in doping the bath. The plating parameters were kept rigourously the same.

After having electrolitically dissolved Copper, we measured the RRR and the critical temperature of the coating. We found that respect to the Tin percentage in solution, our films are richer in Tin. Moreover we found that the introduction of Tin in Lead coatings increases only slightly the film critical temperature Tc (fig. 4), but it makes RRR failing down from 50 to 24 for the sample with 2% of Tin and to 20 for the sample with 4% of Tin.

It can be shown that BCS losses decrease exponentially with Tc, but are proportional to the square root of low temperature resistivity in normal state. The slight increase in Tc does not compensate the loss in RRR. In our opinion once solved the problem of Lead surface instability, we can hope in achieving the same level of performances and reliability obtained by means of the Lead-Tin technology

Literature reports several recipes for Lead passivation, the guide line of them foresees a soak of chelating solution, a soak in ammonia and in acetone or ethanol. The resonator needs afterwards to be stored under nitrogen or in vacuum. We have seen that it is possible to passivate Lead surfaces in simple way. Our lead plated samples were exposed to the open air and do not show any sign of oxidation, for several days. The trick consists in carefully removing any trace of fluoborate by means of subsequent dilutions: if this is done, there is no need of any chelating solution.

The recipe consists in immersing the fresh-plated surface for five consecutive times in a different bath of pure deionized water,

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then as fast as possible the piece must be filled for 8 seconds with an acid solution, then it is rinsed five times in deionized water just as before with the only difference that the last passage is done with hot water. The piece is then dried with nitrogen. It must be avoided any ethanol or acetone rinsing, since this has the effect of dehydrating the Lead hydroxide making it becoming PbO, that has the well-known yellowish colour.



Fig. 4 The superconducting transition for a pure Lead film (cross), for a Lead film to which it was added a 2% of Tin (full square), a Lead film to which it was added a 4% of Tin (empty rhombus).

Hence in our recipe consists in only water rinsing and only one acid solution passage. This can be an acetic acid 1% water solution at the temperature of 90° C or in alternative a phosphoric acid 5% water solution at room temperature. It is worthwhile to remark that such a passivation treatment is not a kind of slight stripping, since its only effect is the dissolution of Lead oxides and the Lead complexation.

References

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