LEAD-TIN PLATING SPLIT LOOP RESONATORS

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Abstract

The twelve split loop resonators for the ANU LINAC have been re-plated with 96Pb/4Sn and their performance substantially improved. The resonators with cracked electron beam welds have been repaired using standard 40Pb/60Sn low temperature solder and brush plating with Ni followed by Cu. The re-plating with Pb-Sn was initially derailed by the appearance of dendrites on the surface plated using Schloetter MSA [1] solution. A new technique of mechanically polishing the unsatisfactory Pb/Sn surfaces and then re-plating, rather than chemically stripping the old Pb/Sn and hand polishing the Cu substrate, is enormously easier, faster and doesn't put at risk thin electron beam welds or repaired welds. Average acceleration field of 3.5 MV/m has been achieved off-line. The best resonator demonstrated 3.9 MV/m at 6 Watts on-line. Twelve SLRs are currently installed and operating in the ANU LINAC.

INTRODUCTION

Lead-tin plating provides fast adequate results with modest equipment and at relatively low cost. SUNY replated six high-beta SLRs with 2 microns of Pb-Sn using a modern, commercial, methane-sulfonate process (Lea Ronal Solderon MHS-L) and a simple open-air procedure [2]. This proven success motivated ANU to adopt MSA chemistry and to re-plate the first SLR in November 1998 followed by re-plating all twelve SLRs by November 2002. This increased the booster energy gain by almost 100%. A view of ANU LINAC comprising four module cryostats is shown in figure 1.

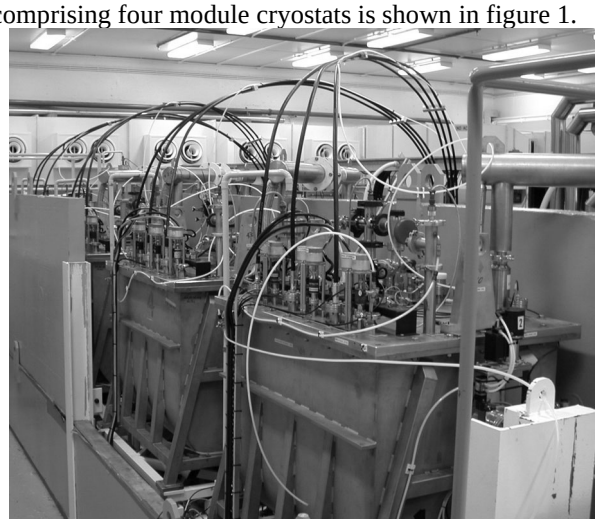

Figure 1: A view of ANU LINAC comprising twelve SLRs in four module cryostats.

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The Pb/Sn plating process at ANU initially employed Solderon MSA plating solution. A detailed account of this plating technology is given in [3]. The smoothest result was produced with a current density of about 5 mA/cm^2. However, the deposit contained crystallites with sharp edges, which probably compromises performance due to enhancement of magnetic and electric fields.

One strategy to reduce the sharp edges, pioneered at Caltech [4], was post-plating chemical polishing. This leaves a smooth mirror-like surface. Unpolished Solderon-plated SLRs have achieved remarkably the high average accelerating fields of 3.5 MV/m in spite of the high roughness [5]. Although the Solderon plating solution has a nominal shelf life of one year, it was employed at ANU for three and half years without noticeable degradation of its plating capability. After that, rapid aging turned the solution yellow, tin oxide precipitated and the throwing power decreased. In July 2002, the old Solderon solution was replaced with a similar MSA based solution from Schloetter, Germany [1]. Schloetter MSA solutions are readily available in Australia through the local electronics industry. In addition, the Munich cyclotron [6] and Mumbai heavy ion booster [7] successfully used Schloetter MSA chemistry in RF superconductivity applications.

LEAD-TIN PLATING SLRS AT ANU USING THE SCHLOETTER SLOTOLET K PROCESS

First trial plating with Schloetter MSA solution on samples was derailed by the appearance of dendrites - it was not possible to obtain a uniform matte deposit with good adhesion to the copper substrate. Instead, at current densities about 5 mA/cm^2, the deposit was loose and powdery. Increases above 5 mA/cm^2, encouraged the growth of large, separate crystallites and caused the growth of whiskers shown in figure 2.

Any form of mechanical stress seems to enhance the growth of whiskers. The stress could originate from: internal stress in the base material or in the coating itself, contamination of the coating by copper and co-deposition of organic components from the plating additive. The presence of hydrogen may also cause whiskers as discussed elsewhere [8].

Both poor adhesion and whiskers compromise the quality of a superconducting film. The whisker can be thought of as a long conducting wire on the surface with poor thermal anchoring to 4 K. In presence of RF, the superconducting whiskers will be heated rapidly above the critical temperature causing RF losses and heating the

adjacent superconducting surface ultimately leading to a quench.

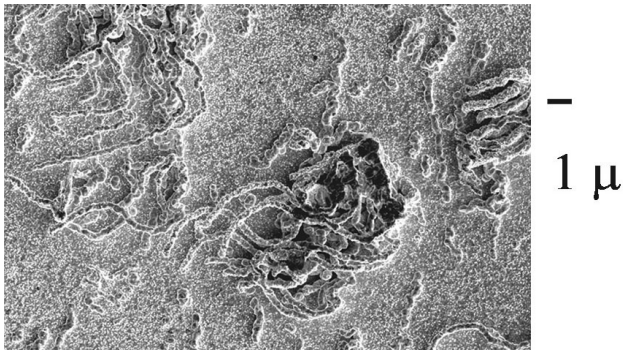

Figure 2: SEM image of lead-tin deposit electroplated onto a copper substrate using Schloetter MSA plating solution. At DC current density above 5 mA/cm^2, the growth of large, separate crystallites and whiskers is observed. The whiskers are about 1 μm in diameter and a few hundred microns long.

A method for obtaining deposit of high quality and free of whiskers was investigated and is discussed below.

Mechanical Polishing

The usual procedure for re-plating resonators is to strip the old Pb/Sn coating with acid followed by mechanical polishing and cleaning the copper. These processes inevitably result in the loss of some copper and so run the risk of breaching the thin cosmetic welds exposing cracks and voids in the deep electron-beam welds below. Such defects interrupt the conducting surface and retain contaminants spoiling the plating. The repair of the defects relies on thin brush-plated copper and so is also at risk if copper is removed. Finally, some copper always dissolves into the plating solution contaminating it. For all these reasons re-plating was attempted directly on the mechanically polished Pb/Sn surface.

The microstructure of the mechanically polished DC plated Pb/Sn film is seen in figure 3.

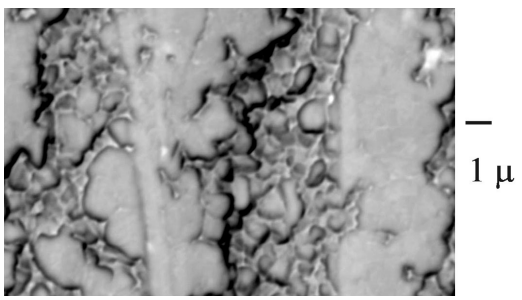

Figure 3: Microstructure of a 1.2 μ thick plated deposit using Schloetter lead-tin solution at 2.5 mA/cm^2 DC followed by polishing using DURAX wipers wetted with alcohol (ethanol).

In the procedure of hand polishing with ethyl alcohol (HPA), the alcohol-wetted wiper is drawn, across the specimen with an even, smooth stroke from one side to the other. It is important not to grind back-and-forth, because this will smear the surface and will increase the probability that loose particles will become embedded. In the initial stage of polishing, a ~30 nm layer of powdery deposit is removed leaving black stain on the wiper. During the entire process it is essential that the specimen and the wiper be frequently rinsed with alcohol to remove loose particles that might otherwise become embedded. Loose large crystallites are dislodged from the surface and removed. Small crystals in low areas that are not in contact with the polishing pad, survive polishing action. During polishing, uneven surfaces tend towards planer therefore the removal rate of material in the recessed areas remains low. The HPA procedure results in smooth mirror-like bright surface.

Chemical Etching of the Specimen after HPA

Even with the most careful polishing, a fairly thick layer of disturbed metal may remain on the surface. This layer can be removed by chemical etching using either lead stripping solution, lead-tin plating solution or by electro polishing. An adequate surface finish was achieved by a combination of: plating; hand polishing; and chemical etching in the plating bath with no current. This process can be compared to conventional chemical polishing involving the simple immersion of a metal specimen in an etching solution to obtain a metallographic polish. The etching rate is quite low in the plating solution without current and can be increased by introducing de-plating cycle. This electrolytic polishing is usually sufficient to remove the thin layer of disturbed metal.

The levelling action concerns the layer of viscous material present on the surface during electro polishing [9]. This anolyte layer is composed of products resulting from the reaction between the metal and the electrolyte. At areas of elevation on the specimen surface, this anolyte layer is thinner and offers less resistance to the flow of current than at areas of depression. The resulting higher current density at elevated areas causes the metal in those areas to dissolve more rapidly than metal in depressed areas.

Despite the fact that de-plating will adequately prepare the surface, reverse pulse plating offers further improvement.

Reverse Pulse Plating

In Reverse Pulse Plating (RPP), the cathode film is kept rich in metal ions and low in impurities. During the plating period, when the current is forward, the metal ions next to the cathode are depleted and a layer rich in water molecules is left. During the portion of the cycle when the current is reversed, the metal ions from the bulk of the plating solution diffuse into the layer next to the cathode. As well during the reverse part of the cycle, gas bubbles and impurities, absorbed on the cathode have a chance to

desorb. Then the process is repeated again. Forward time was 5 seconds and Reverse time was 0.5 seconds. Although it is desirable for some applications for a higher current density in the reverse (de-plating) stage than in the forward (plating) stage, the power supply available could only provide equal currents.

Optimum Plating Procedure

Different combinations of the steps described above, were investigated thoroughly using the SLOTOLET lead-tin plating bath.

The crucial steps were:

- Lead-tin film is deposited at 2.5 mA/cm^2 for a film thickness of 1.5 μ;
- Hand polishing of existing or freshly deposited lead with wipes and alcohol instead of stripping the lead;
- Water rinse thoroughly with de-ionized, high pressure water;
- 15 second soak in plating solution;
- De-plating at 1.5 mA/cm^2 for 30 seconds;
- Immediately reverse pulse plating at 2.5 mA/cm^2 for 7 minutes. Forward time is 5 seconds and Reverse time is 0.5 seconds.

An SEM image of deposit produced using this procedure is shown in figure 4.

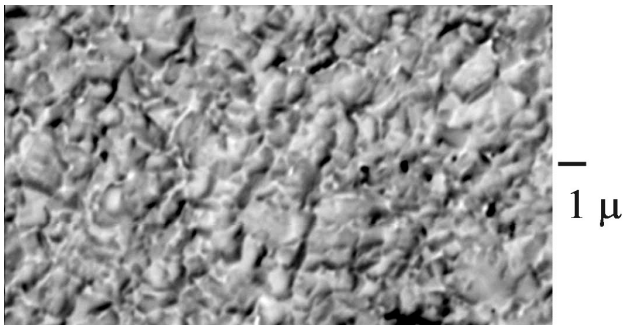

Figure 4: Microstructure of deposit produced using the adopted plating procedure.

This procedure has proven to be successful in re-plating three SLRs in November 2002. The new technique of mechanically polishing the unsatisfactory Pb surface and then re-plating, rather than chemically stripping the old Pb and hand polishing the Cu substrate, is enormously easier, faster and doesn't put at risk thin electron beam welds or repaired ones. Now it takes only one week to re-plate three SLRs, three times faster than the old process.

ON-LINE PERFORMANCE

The re-plating using the hand polishing surface treatment and reverse pulse plating technique has yielded resonators with accelerating field of greater than 3.5 MV/m at 6 W during on-line test - better than any we've previously produced. The Q's at 6 watts are at or above 10^8. The best resonator, achieved E_{acc} about 3.9 MV/m at 6 Watts on-line, figure 5.

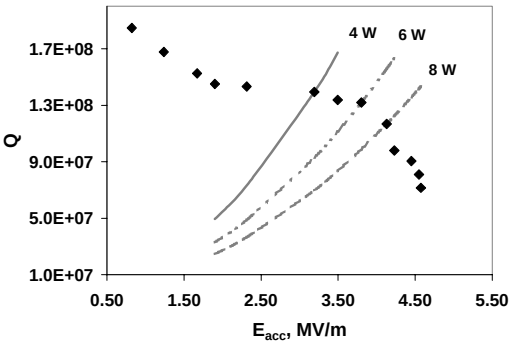

Figure 5: Q vs. E_a curves of the best ANU SLR R#306 re-plated using the Slotolet MSA process in November 2002.

The Q falls at low power levels from 0.2 to 2 Watts. It typically remains constant from 2 W up to 6-7 Watts following by another sharper decline caused by heavy X-ray emission. High Q at RF power level up to nominal 6 Watts is an important advantage of the hand polishing preparation technique compared to all previous procedures. During pulse conditioning, all hand polished re-plated cavities achieved peak magnetic fields of 65 mT and peak electric fields of 26 MV/m. This compares to typical values of 50 mT and 20 MV/m for previously plated cavities. During pulse conditioning, all cavities displayed a constant breakdown pattern and showed no signs further improvement.

The main advantages of the new surface preparation and plating technique is that it leaves a much finer grain structure compared to using the Solderon MSA and no hand polishing of the lead substrate. Solderon coatings contain crystals of 1-3 microns with sharp boundaries. However the new preparation technique might be as successful with the Solderon as with the Schloetter solution. This still has to be verified.

Surface roughness is expected to contribute to magnetic field enhancement. The field enhancement model suggests enhanced magnetic field on the boundaries between grains at different inclination. For surface roughness of 5 microns, a maximum enhancement factor of 2.5 was estimated in ref. [10]. For superconducting lead films operating near 0 K, the superheating magnetic field limitation is 120 mT. At 4.5 K, a magnetic field limitation of superheating field of 96 mT will occur. Assuming the surface roughness is the main limiting factor for ANU SLRs, the magnetic field enhancement factor is 96/50 = 1.9 for the previous coating technique. For the smoother films produces by the new process, the field enhancement is 96/65 = 1.5, 20% lower.

All SLRs were multipactoring conditioned off-line at room temperature, at liquid nitrogen temperature and then at 4 K. The first multipactoring conditioning usually involved processing of two or three multipactoring levels

at fields up to 1 MV/m for many hours. The best procedure was to maximize the absorbed RF power by adjusting coupling and frequency. Increasing absorbed power substantially reduced processing time. Luckily high field multipactoring need be eliminated only once, during initial post-plating conditioning, and does not reappear during subsequent cool down cycles.

The performance of the recently re-plated resonators is much superior to the rest of the LINAC with on-line accelerating field 50% higher. All three resonators had almost identical Q vs. E_{acc} characteristics indicating that the new surface preparation and plating technique provided consistent successful performance.

Three of the twelve SLRs have being re-plated by the new technique. The twelve have an average energy gain of 0.59 MeV/q per cavity. When the β matching and transit time effects are removed, the accelerating field on average is 2.82 MV/m.

The difference between on-line resonator performance and the performance observed off-line are likely to arise from two causes. First, the on-line test is done at 1.3 Bar helium gas pressure, 30% higher than for off-line operations. This raises the temperature from 4.2K to 4.48 K, increasing the BCS surface resistance of the superconducting layer by ~20%. Second, the off-line test always employs the best available RF amplifier and allows longer conditioning time. Thus it is possible to better condition resonators off-line with high power RF processing.

CONCLUSIONS

These investigations improved the understanding of Pb/Sn plated superconducting films resulting in the substantial improvement in the performance of the SLRs. This technology can lead to successful fabrication of LINAC resonators at low cost - crucial for university-based laboratories. At present, the ANU LINAC is able to provide an energy gain of ~ 7.1 MeV/q for beta 0.1 beams. The main advantages of the new Pb/Sn plating procedure are reliability, simple surface preparation, the possibility of solder repair of the substrate and it is inexpensive.

ACKNOWLEDGEMENTS

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