# $NB_3SN$ LAYERS ON HIGH-PURITY NB CAVITIES WITH VERY HIGH QUALITY FACTORS AND ACCELERATING GRADIENTS

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*Abstract*

We have optimized the vapor diffusion technique to coat high-purity Nb cavities up to RRR=1000 with a micron-thick $Nb_3Sn$ layer without loss of the thermal stabilisation of defects. Systematic measurements on samples have shown no change of RRR of the bulk Nb and homogeneously nucleated growth of the $Nb_3Sn$ layers. Rinsing of such layers just with high pressure water resulted in low field emission activity and residual surface resistance values in the n$\Omega$ range, i. e. comparable to the best Nb surfaces. Single-cell 1.5 GHz cavites provided $Q_0$ values up to $10^{11}$ at 2 K and above $10^{10}$ at 4.2 K, which stayed nearly constant up to peak electric surface fields of 10 MV/m but decreased to about $10^9$ at 20 MV/m. No field emission and no quench could be observed in these cavities up to the maximum achievable accelerating gradients of about 15 MV/m at 2 K as limited by the available rf power. The performance of the $Nb_3Sn$ cavities at 4.2 K exceeds the design value of the CEBAF Nb cavities at 2 K. First results on a five-cell cavity are promising.

## 1. INTRODUCTION

During the last decade, superconducting accelerating cavities made from bulk niobium have been improved a lot due to the systematic suppression of anomalous loss mechanisms [1]. Both the achievable field levels and quality factors gained from the enhanced thermal stabilisation of defects by use of high purity Nb as well as from reduced electron loading by means of advanced cleaning techniques [2]. Nowadays accelerating gradients $E_{acc}$ of more than 20 MV/m at $Q_0$ levels close to $10^{10}$ have been demonstrated even with multicell structures in the lower GHz range, which are appropriate for existing electron recyclotrons like S-DALINAC and CEBAF as well as for future linear colliders like TESLA [3-4]. For Nb, however, such performances can be achieved only at temperatures below 2.1 K, i. e. under very expensive superfluid He bath cooling conditions.

In order to allow higher operating temperatures and/or field levels, superconductors with critical temperature $T_c$ and superheating magnetic field $H_{sh}$ higher than those of Nb have often been considered but never been applied for accelerators. The simple reason is that higher $T_c$ is fundamentally combined with lower coherence length, i. e. stronger sensitivity to material imperfections. This is especially true for the high $T_c$ oxides, which provide much higher microwave losses than low $T_c$ superconductors [5]. Among the latter ones, $Nb_3Sn$ is most promising because of its phase diagram which favors phase-pure coating of Nb cavities by the Sn vapor diffusion technique [6]. This technique has been modified for uniform $Nb_3Sn$ coating of medium purity Nb cavities, but early results on a 3 GHz cavity still suffered from quenching [7]. Therefore, the systematic optimization of this process for high purity Nb samples resulting in a more successful $Nb_3Sn$ coating of 1.5 GHz cavities without loss of the thermal stabilization of defects will be presented in this paper.

## 2. $NB_3SN$ LAYER PREPARATION

The modified vapor diffusion technique is based on UHV furnaces with separate heaters for the cavity and for the Sn-source, which consists of a tungsten crucible in a long Nb tube underneath the cavity [8]. This allows an independent control of the growth temperature $T_g$ in the cavity and of the tin vapor pressure $p_t$ given by the source temperature $T_s$, which is important for both the nucleation and the final tempering of the $Nb_3Sn$ layers. After initial heating ($200^0$C) and evacuation of the Ti-shielded cavity, it is in situ closed with a Nb lid and serves as reaction chamber. Then both $T_s$ and $T_g$ are rised to about $500^0$C, where the nucleation of the $Nb_3Sn$ layer is intensified by use of high purity $SnCl_2$ [9]. After about 5 hours, slightly over-saturated growth conditions of typically $T_g$=$1100^0$C and $p_t$=1 Pa at $T_s$=$1200^0$C are reached and hold for 3 h. In order to avoid surplus Sn on the final $Nb_3Sn$ layer, the cavity heater is swiched off 30 min later than the source heater.

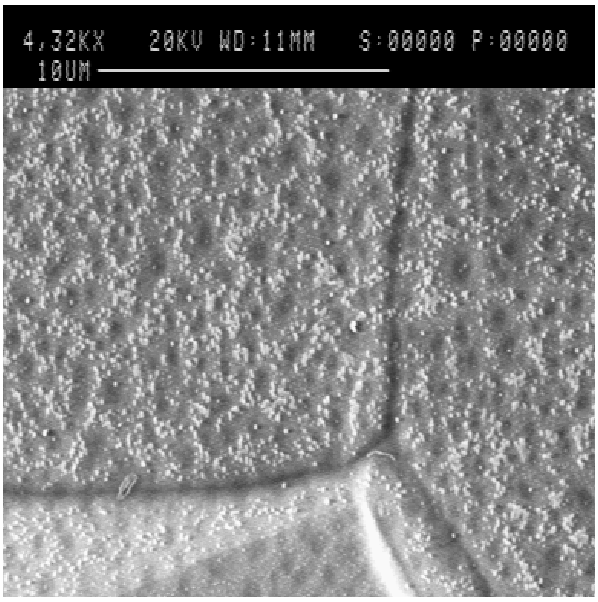


Fig. 1: SEM picture of a high purity Nb sample with Sn nuclei, which were formed by means of $SnCl_2$ at $200^0$C.

As demonstrated in Fig. 1, homogeneous Sn nucleation was achieved on coarse-grained Nb samples independent of the purity grade (RRR≤1000), crystal size and orientation. Contrary to former experiments, this result was obtained for optimized nucleation conditions even in case of non-anodized surfaces. This is very important for high purity Nb, since RRR values above 100 usually degrade during $Nb_3Sn$ growth by oxygen diffusion from additional $Nb_2O_5$ layers [7]. Accordingly, the conservation of the purity grade of non-anodized Nb samples with RRR values between 200 and 900 was confirmed by residual resistivity ratio measurements before and after the $Nb_3Sn$ coating. In comparison, the RRR of samples positioned between the cavity and the Ti shield improved slightly, while exposure in the UHV furnace led to a strong RRR degradation. Thus it can be assumed that the thermal stability of high purity Nb cavities is not affected by our $Nb_3Sn$ coating process.

The quality of the $Nb_3Sn$ layers grown on well-nucleated surfaces was investigated by several methods. At first, the average thickness $d$ of the $Nb_3Sn$ layers was measured gravimetrically, resulting in $d[\mu m]=(1.3\pm.1)t[h]^{(0.38\pm.04)}$ for time periods $t$ between 1 and 4h at $T_g$=1100$^0$C independent of the Nb purity grade. The stoichiometry as well as the depth profile of the layers were controlled by local EDX analysis. While the atomic Sn percentage varied laterally less than 1% around the nominal value of 25%, the Sn content decreased to 20% (10%) in 1.5 µm (2 µm) depth of a typical ($t$=3h) $Nb_3Sn$ layer. The homogeneity of the $Nb_3Sn$ surface was studied by means of scanning electron microscopy. As shown in Fig. 2, a typical $Nb_3Sn$ sample reveals a homogeneous layer with an average grain size between 1 and 2 µm, i. e. in the same order of magnitude as $d$. Moreover, no Sn residues were found on the surface for the given tempering conditions.

First attempts to reduce the granularity of the $Nb_3Sn$ layers by additional annealing at $T_g$=1250$^0$C for 24 h failed because of enhanced Sn diffusion into grain boundaries,

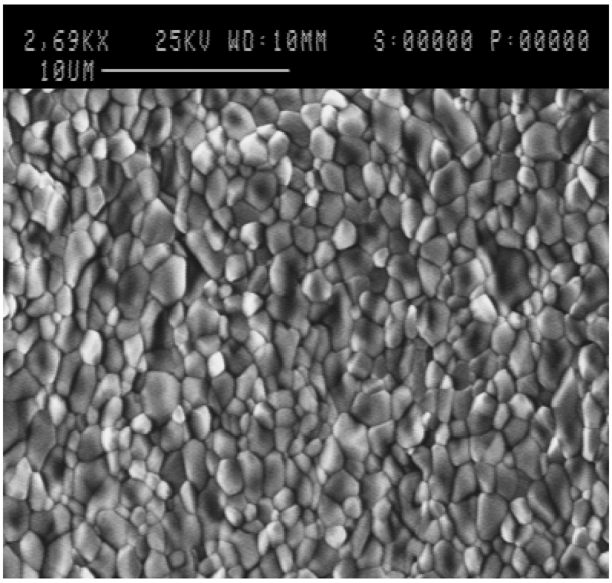


Fig. 2: SEM picture of a typical $Nb_3Sn$ surface as grown for 3h on a high purity Nb sample.

which was concluded from EDX line scans combined with the unchanged sample weigth. Nevertheless, an enlarged average size of $Nb_3Sn$ grains up to 5 µm was obtained, and further refinements of the preparation technique will be required to create homogeneous coarse-grained $Nb_3Sn$ layers on high purity Nb substrates.

## 3. MICROWAVE RESULTS

In order to qualify the described preparation technique for large cavities, the surface resistance $R_s$ of selected $Nb_3Sn$ samples was measured at 87 GHz by means of endplate replacement in a Cu cavity as function of temperature. Besides the correct $T_c$ value of 18.2 K, $R_s$ values of about 200 mΩ at 20 K and 1 mΩ below 10 K were obtained for all samples with exception of the postannealed one which showed a two-phase transition. Other slight variations of the measured $R_s(T)$ curves for differently prepared samples were very difficult to interpret because of the limited measurement sensitivity.

As first step towards the conversion of high purity Nb accelerating structures into $Nb_3Sn$ ones, two single-cell 1.5 GHz cavities of the elliptical CEBAF shape made from commercially available Nb with RRR=300 were successively $Nb_3Sn$ coated with optimized process parameters ($t$=2.7h, $T_g$=1100$^0$C, $T_s$=1200$^0$C, 30 min tempering). Since no Sn residues had to be expected, after receival at CEBAF the cavities were just rinsed with pure methanol and ultrapure high-pressure water. Then the cavities were

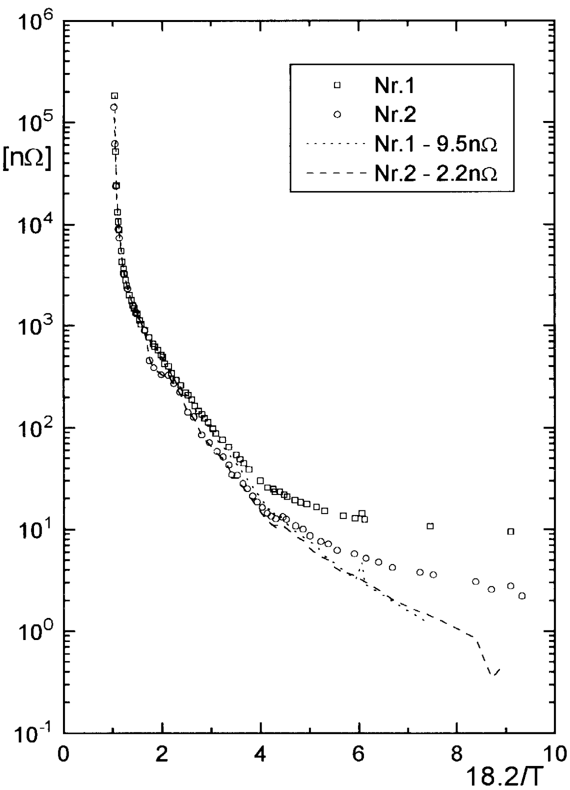


Fig. 3: Temperature dependence of the surface resistance of two $Nb_3Sn$ coated single-cell 1.5 GHz cavities. The lines result for substraction of the residual $R_s$ values at 2 K.

tested as usual, but with established precautions against thermocurrent-induced residual losses [8], i.e. slow cooling rates (1 K / 6 min) in the transition temperature range (25

- 15 K). The resulting $R_s(T)$ curves are shown in Fig. 3. For the first time, extremely low residual $R_s$ values of less than 10 n$\Omega$ have been reproducibly achieved for $Nb_3Sn$ surfaces. The better one provides 2.2 n$\Omega$ at 2 K, a value which is comparable to the record values for Nb at 1.5 GHz [5]. After substraction of a constant residual $R_s$ value, slope analysis for T > 4 K has revealed an reduced energy gap $\Delta/kT_c$ of about 2, which is about 10% lower than that obtained for $Nb_3Sn$ layers on low purity Nb cavities [8]. For temperatures below 4 K, however, a second slope is evident, which might be caused by traces of Sn. Nevertheless, it seems that the usual removal of some tens of nm by oxipolishing [6-9] can be avoided for well-tempered and cleaned $Nb_3Sn$ surfaces on high purity Nb cavities.

According to the low $R_s$ values, both single-cell cavities provide after $Nb_3Sn$ coating much higher low-field $Q_0$ values than before. While high purity Nb yields at 4.2 K and 1.5 GHz a $Q_0$ of about $3 \cdot 10^8$, nearly two orders of magnitude higher $Q_0$ values of more than $10^{10}$ have been achieved for $Nb_3Sn$ at this practical temperature, and even higher $Q_0$ values up to $10^{11}$ were obtained for one cavity at 2 K. As shown in Fig. 4, the $Q_0$ stays nearly constant up to a peak electric surface field $E_p$ of 10 MV/m at 4.2 K and 15 MV/m at 2 K. Then a gradual decrease of $Q_0$ to about $10^9$ at 20 MV/m and 30 MV/m sets in, respectively. This slope of the $Q(E)$ curve seems to be typical for our $Nb_3Sn$ surfaces, since it occurs similarly for both cavities but not for Nb. The onset field strength, however, depends not only on the temperature but also on the surface quality. A performance degradation of about 50% for $Q_0$ and the onset $E_p$ has been observed after oxypolishing (20 V) of the first cavity. It is remarkable that there is, if at all, only a very small $Q_0$ degradation at $E_p$< 2 MV/m for our $Nb_3Sn$ layers, contrary to former experiments where such initial slopes have been attributed to weak links or thermocurrents [8]. Therefore, temperature mapping [2] has to be applied to

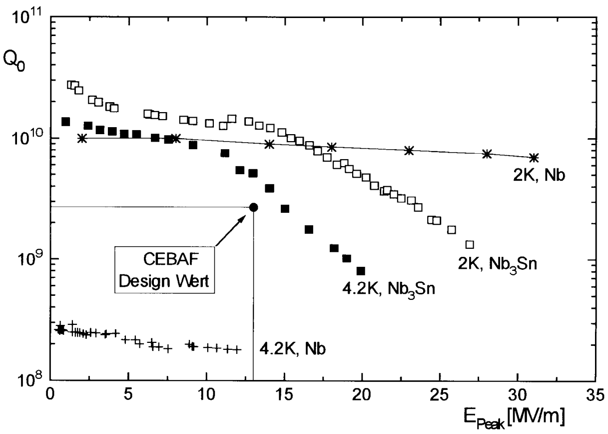


Fig. 4: Field dependence of the quality factor of one of the single-cell 1.5 GHz cavities as measured before (Nb) and after $Nb_3Sn$ coating at 2 and 4.2 K. The CEBAF design value is marked for comparison.

clarify the nature of these nonlinearities. The maximum achieved field leves ($E_p$ = 27 and 33 MV/m) were always limited by the available microwave power. Neither field emission nor quenching has been observed in any of these cavity tests. While the first is in accordance with improved surface cleaning techniques [10], the second demonstrates the benefits of high purity Nb cavities for the thermal stabilisation of $Nb_3Sn$ layers.

Stimulated by these promising results which are at 4.2K above the original CEBAF design values (see Fig. 4), we have coated a 1.5 GHz five-cell Nb cavity with $Nb_3Sn$. Because of the limited size of the furnace, a special cavity without waveguide couplers has been fabricated. Despite of using the same preparation techniques as for the single-cell cavities, first results show somewhat reduced and field-dependent $Q_0$ values in the $10^9$ range. As maximum performance, an accelerating gradient of 7 MV/m ($E_p$=16 MV/m) for a $Q_0$ of $8 \cdot 10^8$ has been achieved at 4.2 K. Therefore, it remains quite a challenge to transfer the single-cell $Nb_3Sn$ results to multicell accelerating structures.

## 4. CONCLUSIONS

For optimized parameters of the modified vapor diffusion technique, homogeneous $Nb_3Sn$ layers can be deposited on high purity Nb cavities now. The surface resistance of such layers is comparable to that of the best Nb cavities, but allows higher operating temperatures. Accelerating gradients up to 10 (6) MV/m with $Q_0$ values above $10^9$ have been demonstrated for 1.5 GHz single- (five-) cell cavities at 4.2 K. Further research on the nature of the losses is required in order to exploit the full potential of $Nb_3Sn$ for microwave applications.


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