

STUDY OF IMPURITY DISTRIBUTION OF ELECTROPOLISHED AND BUFFER CHEMICAL POLISHED NIOBIUM SAMPLES

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Abstract

High purity niobium is used in the fabrication of high accelerating gradient superconducting radio frequency cavities. These cavities undergo various chemical and thermal processing treatments. The main purpose of chemical processing is to remove the damage layer on niobium surface. Chemical and electropolishing treatments are also found to introduce or redistribute various impurities inside the top layer of niobium surface. On the other hand thermal treatments are done to remove dissolved gas like hydrogen and to uniformly diffuse the interstitial impurities like oxygen, on the top surface of niobium. The purpose of this study is to analyze these impurity distributions on the top layer of niobium surface. Secondary ion mass spectrometry (SIMS) was chosen as the technique suited for depth profiling and surface spectroscopy of various impurities on the surface and depth of niobium ~ 50 nm. In this technique the impurities are analyzed using a reflectron based time of flight analyzer (TOF). The study reports the distribution of various impurities on the surface and depth of niobium after being exposed to electropolishing (EP), buffer chemical polishing (BCP) treatment and their combinations. The various impurities profiled after each treatment include H, C, O, F, S, Cl and P. The study clearly shows that the effect of final electropolishing treatment is to reduce the impurity content on the top niobium surface layer.

INTRODUCTION

Over the last 2 decades newer surface processing techniques have been developed which led to improvement in the gradient of SC cavities. Among these processes, the two major ones are buffer chemical polishing (BCP) and electropolishing (EP) techniques apart from the low temperature baking. EP processing raises the accelerating fields by 7 MV/m with respect to BCP and these EP cavities suffer a clear degradation when subjected to a subsequent BCP [1]. But, a few BCP treated cavities have also reached fields as high as 40 MV/m [2]. Hence, a major concern in cavity performances is the lack of reproducibility even after applying controlled BCP and EP processing. It has also been reported that very high purity niobium has high BCS resistance which affects the cavity performance [3]. Moreover, the influence of chemical and annealing treatments on superconducting properties of niobium and its correlation to cavity performance have also been reported [4]. So the cavity performance can be linked to surface contamination apart from reduction in the surface

roughness. To investigate this, a study was initiated to focus on the distribution of various impurities that take place inside the top niobium surface after applying chemical treatments like BCP, EP and their combinations.

EXPERIMENTS

Procedures

Five small Nb samples were prepared from a sheet of 2.8mm thickness, having a residual resistivity ratio of 495. The concentration of H, C, N, O, Ta in the Nb sheet were 8, 65, 10, 40, 83 ppm respectively. These samples were initially degreased and ultrasonically cleaned for 2 hours after which four samples were given treatment as mentioned in table 1. One pristine sample, "Raw", was kept as reference. The four samples were identified as BCP, EP, BCP+EP and EP+BCP. The typical proportion of the acid mixture used for BCP was 1:1:2 (HF: HNO₃: H₃PO₄), while that of EP was 9:1 (vol/vol) mix of H₂SO₄ (96% wt) and HF (48% wt).

Table 1: The sequence of chemical treatments applied.

Treatments		Layer removed		Polishing condition
Step 1	Step 2	BCP	EP	
BCP	-----	110 μ m	-----	T= 20°C
EP	-----	-----	70 μ m	V= 12.5V, T= 28°C
EP	BCP	70 μ m	10 μ m	Same as above
BCP	EP	110 μ m	20 μ m	Same as above

Analysis process

Chemical impurity analysis was carried out by time of flight secondary ion mass spectrometry (TOF SIMS). In this technique the sample was alternately bombarded with a pulsed Bi⁺ ion gun having primary ion energy of 25 keV and Cs⁺ sputtering gun with energy of 0.5 keV. The secondary ions produced by the analysis gun were extracted to generate the mass spectrum. The mass spectrum was acquired for each spot in layer by layer fashion to construct the impurity profiles across the depth of the sample. The vacuum level was maintained at 9E-10 mbar. The total area of analysis was 100x100 μ m inside a sputtered area of 300x300 μ m. The surface roughness was slightly high in BCP treated and pristine sample.

RESULTS

The depth profiles of impurities such as hydrogen, oxygen, fluorine, phosphorous and sulphur are shown in figure 1 (Cl and C not shown). The x-axis represents the depth from top surface, while the y-axis represents intensity/counts of impurities. It was observed that Nb

samples undergoing EP treatment has lower O, C, F and P impurity levels when compared to samples undergoing BCP treatment. Although the impurity content of O is similar in the top 2 – 5nm layer, but beyond this depth, the contamination decreases by an order in EP processed sample. Similar trend was observed in case of carbon.

On the other hand the impurity level of H and S was lower in BCP processed sample when compared to EP treated sample.

The following observations were made when impurity distribution of pristine sample was compared with the other four chemically processed samples.

- a. The contamination of impurities like H, C, O, Cl decreases when either BCP or EP treatments or their combinations were applied on pristine Nb samples.
- b. But the trend was not so clear in case of impurities like F, P, and S. It was found that BCP processing introduces extensive P contamination (in the form of

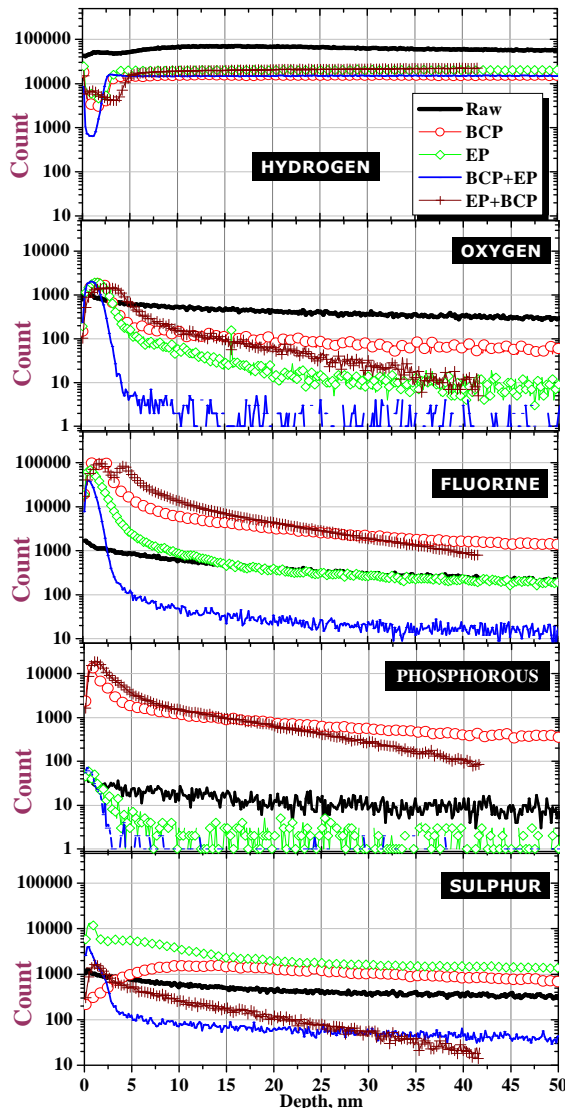


Figure 1: Impurity distribution of H, O, F, P and S in the top 50 nm layer of each of the 5 Nb samples.

either P or PO_4^{2-}) when compared to pristine sample. The incorporation of POx ions at the oxide layer interface due to the use of ortho-phosphoric acid has been reported elsewhere [5]. In case of fluorine, it was found that only the BCP+EP sample had lower contamination than pristine sample. Finally in case of S, the contamination increased in either BCP or EP treated sample when compared to pristine sample.

Finally the most remarkable feature observed in this analysis was that, whenever a combination of initial BCP and final EP treatment was applied, the lowest contamination level of all the impurities was achieved except for S and H.

CONCLUSION

It is clear from this analysis that the impurity content in the top 50 – 100 nm layer of niobium is extremely sensitive not only to the processing techniques, but also to their combination and the sequence. This also sheds light to the fact as to why a final BCP processing might degrade cavity performance. Moreover this technique can be extremely useful to pinpoint the impurities that actually affect the cavity performance. Finally it can be concluded that a combination of initial BCP and final EP can lead to extremely low levels of impurities inside the top 50 nm of niobium surface.

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