

CHEMICAL STRUCTURE OF NIOBIUM SAMPLES VACUUM TREATED IN NITROGEN IN PARALLEL WITH VERY HIGH Q_0 CAVITIES.

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Abstract

X-ray photoemission spectroscopy (XPS), Auger electron spectroscopy (AES) and X-ray diffraction (XRD), are used for the surface analysis and bulk phase characterization of nitrogen treated samples processed parallel with superconducting radio-frequency (SRF) cavities. We investigated the surface chemistry of samples treated with nitrogen in order to understand this treatment effect on SRF cavity performance for several baking temperatures and durations in order to find cost efficient post-furnace chemistry-free procedures to enable high Q -values.

INTRODUCTION

The combination of surface treatments applied to an SRF niobium cavity completely define its performance by means of altering the value of the cavity's surface resistance. Extensive research and cavity testing enabled the achievement of quality factor values (Q_0) on the order of 10^{10} at a peak magnetic field of ≈ 160 mT, which corresponds to accelerating gradients of ≈ 40 MV/m. However consistent improvement of cavity performance is limited by field dependent rf losses, such as Medium Field Q -slope (MFQS). MFQS is a gradual degradation of the quality factor which is observed between 20 mT and 80 mT. MFQS poses prohibitive economical costs for future particle accelerators, such as ProjectX, by restricting desired values of accelerating gradients. The search for an optimal, cost-efficient combination of surface treatments at FNAL is intended to overcome MFQS, in a reproducible way, and understand its causes.

A recently found combination of surface processing, which includes high temperature nitrogen treatment [1], demonstrates a reverse of Q_0 field dependent degradation. Reported values of Q_0 are two to three times higher than those obtained after the typical standard treatment sequence. Such an improvement in cavity performance was achieved by combination of: 1) reacting bulk niobium cavities with nitrogen gas at high temperatures in a UHV furnace, 2) material removal via electropolishing (EP) and high-pressure water rinsing (HPR). Figure 1 [1] shows the results of RF characterization of the cavities, which were subject to the described sequence with various experimental parameters. Non-typical improvement in the performance of these cavities (as compared to the cavity which had undergone standard surface treatments) is obvious.

Our characterization work was initiated in order to gain a better understanding of the surface changes of SRF cavities with applied surface treatments. Our investigations

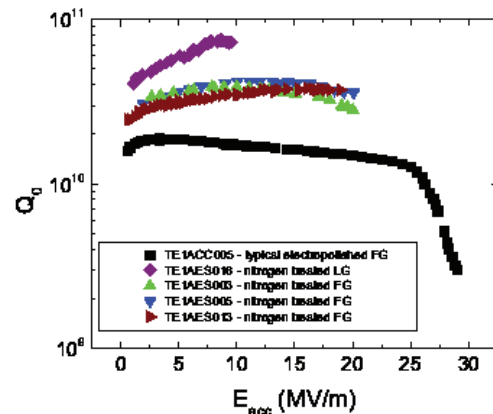


Figure 1: XPS Nitrogen 1s peaks taken from nitrogen treated samples.

consist of tracking material features induced by individual steps of the surface treatment and corresponding them to the results of RF cavity testing. Characterization of surface changes of the samples processed in parallel with SRF cavities gives insight into the processes responsible for particular RF performance of the cavity. XPS and AES were used to characterize compositional changes of the samples surface. Phase characterization of the samples was done by XRD.

EXPERIMENTAL METHODS AND DISCUSSION

Thermo Scientific ESCALAB 250 Xi instrument with Al $K\alpha$ source was used for collection of XPS spectra. Resolution of the instrument is ≈ 0.4 eV at pass energy 20 eV. Pananalytical/Philips X'pert² Material Research Diffractometer (with crossed-slit collimator, parallel plates collimator, flat graphite monochromator and proportional detector) was used for XRD. AES was performed with Physical Electronics PHI 660 instrument.

Combination of the surface characterization and RF testing were used to study the effect of nitrogen treatment at different experimental parameters and its changes after "light" material removal via EP. The first step of the surface treatment consists of baking a 1.3 GHz niobium SRF cavity in the high temperature (800°C-1000°C) furnace with nitrogen gas (partial pressure of $N_2 \approx 2 \cdot 10^{-2}$ Torr) for various periods of time. RF characterization of nitrogen treated cavities showed very poor performance (Q on the order of 10^7). Presence of nitrogen was detected by XPS

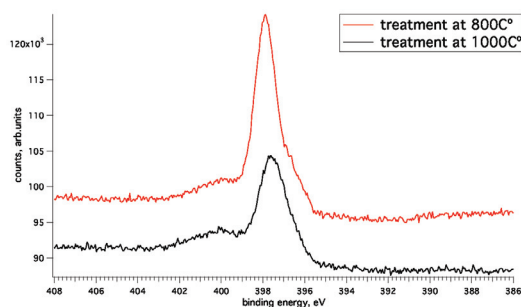


Figure 2: XPS nitrogen 1s spectra collected from nitrogen treated samples.

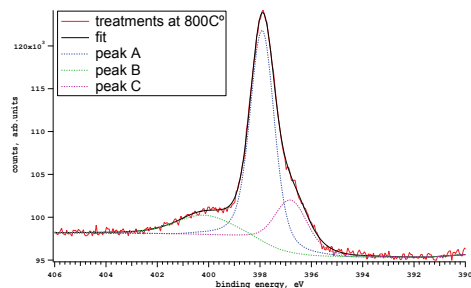


Figure 3: Nitrogen 1s peak fit to the spectra collected from niobium sample treated at 800°C for 10 min.

on niobium samples treated at 800°C for 10 minutes and at 1000°C for 1 hour. Figure 2 shows nitrogen 1s peaks collected from the surface of niobium samples treated with nitrogen. Preliminary fit of N₂ 1s peak collected from 800°C treated sample (Figure 3) shows that it consists of three contributions (at 397.9 eV, 400.1 eV, and 396.8 eV). The peak located at 397.9 eV could be assigned to nitrogen in NbN, and the peak at 400.1 eV could be assigned to the presence of nitrogen in oxynitride (NbN_xO_y) [2]. The approximate concentration of nitrogen on the sample surfaces was found to be of the order of 20 at.% for different temperatures of nitrogen treatment. Figure 4 demonstrates 3d niobium spectra collected from the same nitrogen treated samples. Three doublets give the best preliminary fit to the Nb 3d peak collected from the 800°C treated sample (Figure 5), which indicates the presence of a mixture of niobium nitride phase, some form of oxynitride, and niobium oxide (Nb₂O₅). Binding energies for Nb 3d_{5/2} doublets obtained from the fit are: 203.4 eV, 204.0 eV, and 207.2 eV. Doublets at 203.4 eV (doublet A) and 207.2 eV (doublet C) agree with the reference values for NbN_x phases [3] and Nb₂O₅ [2–4], respectively.

Additional surface chemical characterization was performed by AES. AES spectra were taken for several grains in the niobium sample treated with nitrogen at 800°C for 10 min (Figure 6). Qualitative analysis of the spectra shows that some grains reveal stronger oxygen peak than others, which indicates variation in oxygen concentration among the grains.

Phase characterization of the sample treated with nitro-

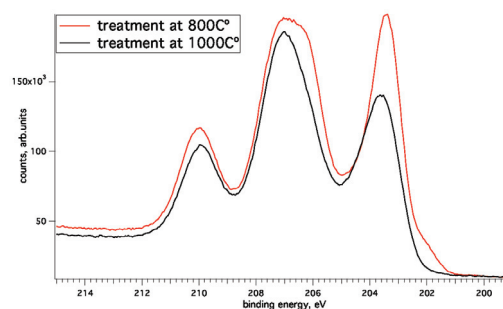


Figure 4: XPS Nb 3d spectra collected from nitrogen treated samples.

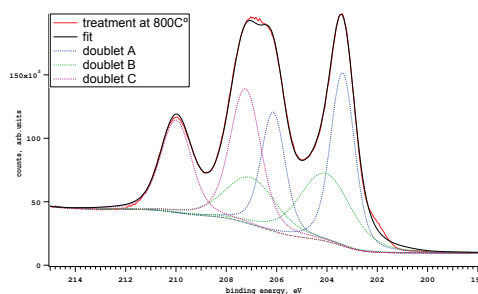


Figure 5: Nb 3d peak fit to the spectra collected from niobium sample treated at 800°C for 10 min.

gen at 800C for 10min was performed by XRD (Figure 7). Peaks from niobium nitride phase (marked in blue) were detected along with peaks from niobium substrate (marked in red). Niobium nitride phase corresponds to the hexagonal NbN_{0.5} (PDF 04-003-1490). Some preferred orientations of the diffracting grains can be noticed from the spectra. Lattice constants were calculated from the fit: a=3.05434Å, c=4.966Å. Approximate grain size from the peak fit was estimated to be 280Å. Cavity testing results and surface characterization of the first step of the surface treatment used for the reversal of MFQS indicate formation of an unwanted mixture of niobium and nitrogen phases, which are not beneficial for the surface superconductivity.

The second step of the surface treatment is EP which removes the material from the surface of the niobium cavity

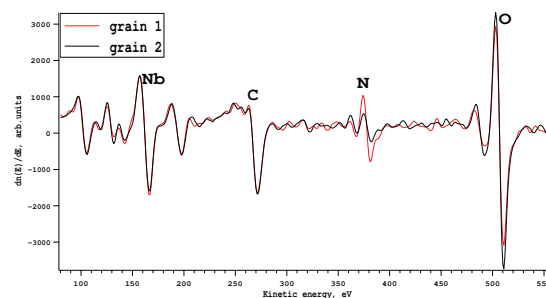


Figure 6: AES spectra collected from niobium sample treated at 800°C for 10 min.

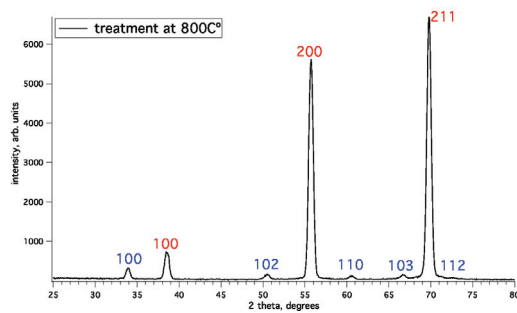


Figure 7: XRD spectra collected from niobium sample treated at 800°C for 10 min.

that had been heat treated with nitrogen. Various amounts of material removal, followed by HPR, have been characterized by cavity testing. The best performances have been found for the following combinations: 5 μm was removed from the sample that had been treated at 800°C for 10 min; 80 μm was removed by EP from the sample treated with nitrogen at 1000°C for 1 hour.

Niobium samples that had been treated with nitrogen, followed by EP and HPR, were characterized by XRD and XPS. No nitrogen presence was detected. XRD indicates only niobium peaks. This shows that no stoichiometric niobium nitride phases are forming after the material removal. The absence of a well defined nitrogen peak on XPS spectra might suggest a low concentration of nitrogen, beyond the detection limit. The presence of some nitrogen in the interstitial sites of niobium lattice was suggested as an explanation of anti-Q slope and much higher values of Q in nitrogen treated cavities. The thickness of removed material corresponding to $\approx 1/4$ of the diffusion length of nitrogen in niobium (for various experimental parameters) was found to provide the highest Q-values and strongest anti-Q slope [1]. When a larger thickness was removed, MFQS was found from RF testing of such cavity. Those facts support the presence of interstitial nitrogen, which could act as trapping sites for hydrogen. Further investigations, such as Secondary Ion Mass Spectroscopy, are needed in order to confirm this assumption.

CONCLUSION

XPS and XRD studies of individual steps of the surface processing that lead to non-typically high performance, suggest the beneficial effect of nitrogen doping. Additional surface studies are needed in order to quantify low concentrations of nitrogen and fully understand the mechanisms of the reverse of MFQS and non-typically high Q values appearance.

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