Nitrogen Infusion in a dedicated sample furnace

The so-called “Nitrogen Infusion” process applied to 33Gohm-TEA-type cavities was reported to achieve higher Q-values compared to the standard surface treatments. The recipe consists of a heat treatment at 800°C for 3h under vacuum conditions followed by a ramping down to 120°C. During the hold time of 48h at 120°C a partial pressure (3x10⁻² mbar) of nitrogen is applied. It is our goal to find the key parameter for a stable and reproducible recipe. An extensive sample study is carried out and the results are presented here.

Residual Gas Analysis (RGA) spectrum at different steps during a 120°C Nitrogen Infusion run.

Residual Resistivity Ratio (RRR)

- A 4pt contact method was selected to investigate the influence of nitrogen infusion on the RRR value of samples.
- The higher the RRR, the fewer defects and impurities in the material. Thus the RRR characterizes the purity of a metal.
- Temperature cycle: Ramp to 800°C for 3h under vacuum; ramping down to desired temperature (120°C - 400°C) for 48h, during which nitrogen is introduced (3x10⁻² mbar).
- Total of 15 fine grain (100 μm grain size) niobium samples.
- 5 of which were subjected to a nitrogen infusion in a temperature range between 120°C and 400°C.
- 5 samples are subjected to the same temperature treatment, but without nitrogen, i.e. under vacuum.

Surface composition for 400°C N infusion

Nitrogen InfusionSAMPLE R&D AT DESY

Christopher Bate *1,2, Arti Dangwal Pandey 1, Alexey Ermakov 1, Brian Foster 1,2,3, Wolfgang Hillert 1,2, Thomas Keller 1,2, Detlef Reschke 1, Jörg Schaftran 1, Sven Sievers 1, Hans Weise 1, Marc Wenskat 1,2, DESY, Hamburg, Germany.
2 Universität Hamburg, Hamburg, Germany.
3 Physics Dept., University of Oxford, Oxford, UK

ABSTRACT

Many accelerator projects, such as the European XFEL or upgrade of the ILC, would benefit from cavities with reduced surface resistance (high Q-value) while maintaining high accelerating gradient. A possible way to meet the requirements is the so-called nitrogen-infusion procedure on niobium cavities. However, a fundamental understanding and a theoretical model of this method are still missing. The approach here is the infusion (150 μm RRR) of small samples, with the goal of identifying all key parameters of the process and establishing a stable, reproducible recipe. To understand the underlying processes of the surface evolution that give improved cavity performance, advanced surface analysis techniques (e.g. SEM/EDX, TEM, AFM, TOF-SIMS) are utilized and several kinds of samples - such as cavity cut-outs and samples treated together with cavities - are analyzed. Furthermore, parameters such as RRR and the surface critical magnetic field denoted as $H_{c2}$ have been investigated. For this purpose a small furnace dedicated to sample treatment was set up to change and explore the parameter space of the infusion recipe. Results of these analyses and their implications for the R&D on cavities are presented.

Surface critical magnetic field $H_{c2}$ by SQUID

AC-susceptibility measurements of niobium samples with a superconducting quantum interference device (SQUID) were carried out to observe the dependence of surface treatments on $H_{c2}$.

Samples used for SQUID measurements. Sample U is a raw untreated sample.

Obtained values for the surface nucleation field $H_{c1}$ from susceptibility measurement. An error of roughly ±10% is estimated for the determination of $H_{c1}$. Sample 67 was cut in half to compare the surface treated upper half of $H_{c2}$ and the untreated lower half 67l.

Comparison of sample U with the differently treated samples shows that the treatment of the samples visibly increases the surface superconductivity critical field $H_{c2}$.

The proportionality factor obtained for sample E2+ = 3.6 ± 0.18 is much larger than was predicted by St. James and De Gennes $\nu = 1.675$ and also exceeds the factors obtained from St. Coehoorn et al. [2] with $\nu = 1.85 ± 0.03$ for $H_{c2}$ and $\nu = 2.1 ± 0.03$ for EP samples.

Conclusion

- RRR measurements show different dependency on the N infusion temperature to the same heat cycle without nitrogen. A maximum in the deterioration of RRR occurs at 260°C regardless of nitrogen introduced or not. The deterioration is higher for the N infused samples except at 120°C.
- It is assumed that a redistribution of the oxygen in the bulk play a role here and the nitrogen infusion also has an influence on it.
- SIMS measurements showed an NbN signal on an N infused fine-grain sample at 400°C. No such signal is observed on a large grain sample and another fine-grain sample that was not N infused at 120°C. However, oxygen contamination was present on these.
- Superconducting effects can be detected even with magnetic fields $H > H_{c2}$ and from the superconducting properties of a surface layer within a thickness of order the coherence length can be inferred.
- The currently used niobium material shows extremely high $H_{c2}$ values compared to [2].

Acknowledgement

The author would like to thank the collaboration colleagues Dieter Eisterer and Florian Semper from the TU Wien who carried out the SQUID measurements.


References

Poster SUPFDV001

NITROGEN INFUSION SAMPLE R&D AT DESY.

Poster ID: SUPFDV001

Christopher Bate *1,2, Arti Dangwal Pandey 1, Alexey Ermakov 1, Brian Foster 1,2,3, Wolfgang Hillert 1,2, Thomas Keller 1,2, Detlef Reschke 1, Jörg Schaftran 1, Sven Sievers 1, Hans Weise 1, Marc Wenskat 1,2

¹ DESY, Hamburg, Germany.
² Universität Hamburg, Hamburg, Germany.
³ Physics Dept., University of Oxford, Oxford, UK

28 June 2021 – 02 July 2021