## FLUX EXPULSION VARIATION IN SRF CAVITIES \*

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#### Abstract

Treating a cavity with nitrogen doping significantly increases  $Q_0$  at medium fields, reducing cryogenic costs for high duty factor linear accelerators such as LCLS II. Ndoping also makes cavities more sensitive to increased residual resistance due to trapped magnetic flux, making it critical to either have extremely effective magnetic shielding, or to prevent flux from being trapped in the cavity during cooldown. In this paper, we report on results of a study of flux expulsion. We discuss possible ways in which flux can be pinned in the inner surface, outer surface, or bulk of a cavity, and we present experimental results studying these mechanisms. We show that grain structure appears to play a key role and that a cavity that expelled flux poorly changed to expelling flux well after a high temperature furnace treatment. We further show that after furnace treatment, this cavity exhibited a significant improvement in quality factor when cooled in an external magnetic field. We conclude with implications for SRF accelerators with high  $Q_0$  requirements.

#### BACKGROUND

In the last several years, there has been rapid progress in technology for high  $Q_0$  applications. Nitrogen doping was discovered and recipes were developed to dramatically reduce both BCS and residual surface resistances ( $R_{BCS}$  and  $R_{res}$ ) at peak fields on the order of 70 mT [1]. Furthermore, researchers observed the importance of cooldown on residual resistance in the bulk dressed niobium cavity prepared by BCP [2], attributing the effect to additional magnetic fields generated by thermocurrents [3]. Subsequently, the importance of the cooldown conditions on the amount of trapped flux even for the same ambient field was discovered in bare cavities of various surface treatments [4] showing the dramatic impact of spatial temperature gradient at transition on the residual resistance. Studies showed that N-doping increases the sensitivity of the residual resistance to trapped magnetic flux [5]. In addition, the effect of material preparation on tendency to trap flux (i.e. percent of external flux not expelled during cooldown) was studied in bulk niobium samples [6,7].

Building on these studies, in this paper we study the effect of preparation and cooldown conditions on the tendency to trap flux in single cell 1.3 GHz cavities.

# FLUX EXPULSION

Cooling N-doped bulk niobium cavities through transition with a spatial temperature gradient reduces residual resistance from external magnetic fields. This has been shown both in vertical test [4] and in horizontal test [8]. The exact mechanism is not well understood, but it is likely that thermal forces on pinned vortices play an important role. We offer a picture of how this could work in Fig. 1.



Figure 1: Schematic of a cross section of a bulk N-doped cavity wall, showing layers of different materials: N-doped niobium at the inner surface, high purity niobium in the bulk, N-doped niobium at the outer surface, and NbN compounds at the outer surface. Isotherms during cooldown are also indicated schematically, along with the corresponding thermal force  $f_T$  on a vortex.

During cooldown, a temperature gradient will be present not just from the bottom to top of the cavity, but also from outside wall to inside. It has been shown that spatial temperature gradients create a force on vortices, pushing them towards cooler regions [9] (see [10] for another SRF cavity application of this). In the geometry from Fig. 1, there is a component of the force pushing the vortices away from the RF surface and out of the cavity. If the force is large enough, it can depin flux and expel it.

The required depinning force—and hence the required thermal gradient—would depend on the strength with which magnetic field lines were pinned. Sample studies suggest that grain boundaries and dislocations act as pinning centers [6, 7]. In addition to these bulk properties, surface properties may play a role. A N-doped cavity will have a thin layer of nitrogen-rich material at its interior and exterior surfaces. Immediately after doping, it can also have a layer of poorly superconducting niobium nitride phases on its interior and exterior surfaces, though the interior nitrides

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are generally removed with electropolishing (EP). A cavity treated with 120°C baking will have oxygen-rich material on its inner and outer surfaces.

If grain boundaries and dislocations act as pinning centers, it should be possible to change flux expulsion in a cavity by treating it with high temperature baking. If the interior or exterior layers act as strong pinning sites, it should be possible to change flux expulsion with chemical removal. One of the goals of this study was to determine the effect of these two treatments on flux expulsion.

If flux expulsion could be improved, it could be possible to reduce the requirements on magnetic shielding and on thermal gradient during cooldown for cavities in high  $Q_0$ machines. For example, in [8], it was found that vertical gradients on the order of 20 K were required to minimize  $R_{res}$ , even with a double layer of magnetic shields.

### **APPARATUS**

For this study, flux expulsion and surface resistance were measured for a number of cavities prepared in various ways, under different cooldown conditions. Flux expulsion was measured using the method from [11]. An axial magnetic field on the order of 10 mG was applied to the cavities using external field coils. A number of fluxgate magnetometers (generally three) were spaced around the equator of the cavities, parallel to the axis, as shown in Fig. 2. The magnetic field was measured before  $(B_{NC})$  and after  $(B_{SC})$  transition to the superconducting state, as shown in Fig. 3. When the external field was completely trapped in the superconductor, the field distribution remained approximately the same (predicted ratio  $B_{SC}/B_{NC} = 1$ ). When the external field was completely expelled by the superconductor, the fields outside the superconductor are enhanced by approximately 80% (predicted ratio  $B_{SC}/B_{NC} = 1.8$ ).





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Figure 3: Example measurements of flux expulsion during cooldown.  $\Delta T$  is measured from iris-to-iris when the bottom iris reaches 9.2 K.  $B_{NC}$  and  $B_{SC}$  are measured before and after transition. The x's mark which values are used. The magnetic field ratios show that in the top example, flux is largely trapped, and in the bottom, flux is largely expelled.

The fine grain 1.3 GHz single cell cavities used in this study were prepared in various ways, sometimes for other studies, from which parasitic flux expulsion measurements were made. The cavities were cooled in a vertical test dewar with liquid helium filling from the bottom. Temperature sensors were placed at the top and bottom iris and at the equator. The spatial temperature gradient was measured from the bottom to the top iris when the bottom iris reached 9.2 K. The temperature gradient was varied by beginning the cooldown at different starting temperatures. If RF results were to be measured during cooldown, liquid would continue to be added to the dewar-otherwise, once all sensors read 5 K, the helium flow was stopped and the dewar was warmed up. By performing a series of warmupcooldown cycles to only 5 K, flux expulsion could be characterized for a cavity with modest helium usage.

#### RESULTS

The results of the flux expulsion measurements are overviewed in Fig. 4.

The first measurement was performed on cavity AES011, which received a 5 micron external BCP after nitrogen dop-

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Figure 4: Flux expulsion ratio  $B_{SC}/B_{NC}$  of cavities prepared in various ways as a function of iris-to-iris temperature difference during cooldown. A ratio of 1.8 represents perfect flux expulsion, while a ratio of 1.0 represents full flux trapping. Some cavities fail to expel flux even for  $\Delta T$  on the order of 10 K, and they show enhanced surface resistance in RF measurements. Uncertainty in expulsion ratio is approximately 0.1 and in  $\Delta T$ , it is approximately 1 K.

ing with the 2/6 recipe (2 minutes in N<sub>2</sub> gas at 800°C with 6 minute anneal and EP). These results were presented in [11]. This cavity expelled flux fairly well, achieving close to full expulsion for  $\Delta T \gtrsim 2$  K. In subsequent tests, it received a 90°C bake and a 3 micron electropolish. Neither treatment affected the expulsion appreciably.

The next cavity that was tested was ACC002. This cavity was heavily doped (20 minutes in nitrogen at 800°C), and its flux expulsion was characterized before any external chemistry (AES011 was RF tested before its external BCP treatment, but the procedure for flux expulsion measurement was developed later). It showed considerably smaller flux expulsion as a function of thermal gradient than AES011. After external BCP of 6 microns to remove the NbN phases, it seemed to show somewhat improved expulsion. Additional BCP of 24 microns seemed to result in similar expulsion to before the first round of BCP.

AES017, which was doped with a 2/6 recipe, also showed relatively poor expulsion. It showed no appreciable change after outside BCP of 5 microns. After the outside BCP, the cavity should have had a very similar inner and outer surface as AES011. Both received 2/6 doping followed by a light external BCP. The fact that they have strongly different behavior suggests that the inner and outer surface treatment is not a dominating factor determining flux expulsion in these cavities. This is supported by the relatively small effect of the outside BCP on ACC002, as well as it having an expulsion characteristic intermediate to that of AES011 and AES017 in spite of having a heavier level of N-doping.

One interesting feature of AES011 is that in spite of being fabricated from material with grain size on the order of 50 microns, its surface shows very large grains, as shown in Fig. 5, suggesting significant grain growth over its history. However, logs of the treatment of this cavity show furnace treatments at 800°C, but not at higher temperatures. One possible reason for significant grain growth at such low temperatures would be a high RRR value of the material [12]. Material reports from these cavities show that the material had RRR values of approximately 480. Other cavities from this batch of cavities also seemed to expel flux well, such as AES014. On the other hand, the material from the batch with AES017 came from a different vendor, with reported RRR values of approximately 350. AES018, also from this batch, showed relatively poor expulsion as well.

The hypothesis that flux expulsion characteristic is strongly related to the lattice of the bulk material is supported by the last test of AES017. In this test, the cavity was given a heat treatment at 1000°C for 4 hours before reducing the temperature to 800°C and doping the cavity again with 2/6 recipe (to make up for nitrogen diffused into the bulk). After this treatment, the cavity appeared to have millimetersized grains, and it showed greatly enhanced flux expulsion, similar to that of AES011 and AES014.

#### DISCUSSION

The results suggest that there are two factors that significantly contribute to good flux expulsion: thermal gradients and heat treatment to affect crystal structure. The results do not distinguish between the effects of grain growth (re-



Figure 5: Significant grain growth observed in AES011.

duction in total number of grain boundaries) and the effects of recrystallization and dislocations. However, sample studies suggest both play a role: improved flux expulsion is observed both when going from polycrystalline samples to single crystal samples and when going from single crystal samples without heat treatment to ones with heat treatment at 800 and 1200°C [6].

The positive impact of larger grains would also be consistent with previously reported results suggesting improved quality factors in large grain cavities compared to fine grain cavities [13].

In Fig. 6, we show that the factors being studied in these experiments have a significant impact on cavity performance. Both sets of Q vs E curves come from AES017, one before furnace treatment at 1000°C, and one after. In both cases, the cavity was treated with a 2/6 N-doping, giving it a low-field  $Q_0$  of approximately  $3 \times 10^{10}$  at 2 K when the cavity is cooled in the absence of magnetic fields. However, when cooled in an external magnetic field of ~10 mG with a modest thermal gradient, the values are starkly different. The cooldowns for these curves are shown in Fig. 3. Before furnace treatment, the cavity shows a low field  $Q_0$  on the order of  $1.5 \times 10^{10}$  at 2 K, while after, it is on the order of  $3 \times 10^{10}$ . These measurements show that the improvement in flux expulsion directly translates to an improvement in  $Q_0$  in the presence of an external field.

Note that the *Q*-slope observed in the *Q* vs *E* curve post 1000°C treatment is characteristic of an overdoped cavity. It is suspected that the N-content in the cavity surface is higher than desired, possibly due to leftover nitrogen from the first 2/6 doping before doping again after the 1000°C treatment. The cavity will next be retested after a 3 micron EP. Heavy doping is not expected to strongly influence flux expulsion based on the results of ACC002.

Preliminary measurements of a cavity manufactured from large grain material also show very good flux expulsion.



Figure 6: Q vs E curves of AES017 cooled with thermal gradients on the order of 4 K in a ~10 mG external field. Before 1000°C furnace treatment (top), the cavity expels poorly and the  $Q_0$  is strongly suppressed relative to the zero-external-field value. After 1000°C treatment (bottom), the cavity expels well and the  $Q_0$  is close to the ideal value. The corresponding cooldowns for these Q vs E curves are shown in Fig. 3.

## **CONCLUSIONS**

In this paper, we presented results from a study of tendency to trap magnetic flux in 1.3 GHz single cell niobium cavities. It was found that expulsion of magnetic flux was significantly enhanced after furnace treatments at high temperatures and a modest spatial temperature gradient during cooldown through transition-on the other hand, various surface treatments of the cavity had little impact. This agrees with previous sample studies, but additional experiments should be performed to study the connection between furnace treatment and improvement in flux expulsion. If additional experiments confirm this connection, then these results may be important for high  $Q_0$  machines such as LCLS II. Before production begins, representative quality control cavities could be fabricated using the planned material and production method. The cavities may show good expulsion as-manufactured. However, if poor expulsion is observed, the study presented here suggests that the addition of a high temperature furnace treatment could prevent significant residual resistance due to trapped flux. This may represent a simpler solution than additional magnetic shielding, larger thermal gradients, or additional cryogenic capacity.

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#### REFERENCES

- [1] A Grassellino et al., Supercond. Sci. Technol., 26 102001 (2013).
- [2] O. Kugeler et al., Proceedings of the Fourteenth Conference on RF Superconductivity, TUPPO053 (2009).
- [3] J.-M. Vogt, O. Kugeler, and J. Knobloch, *Phys. Rev. ST-AB*, 18, 042001, (2015).
- [4] A. Romanenko et al., Journal App. Phys., 115, 184903 (2014).
- [5] D. Gonnella and M. Liepe, Proceedings of the Fifth International Particle Accelerator Conference, WEPRI063 (2014).
- [6] S. Aull, O. Kugeler, and J. Knobloch, *Phys. Rev. ST-AB*, 15, 062001 (2012).
- [7] A. Dhavale et al., Supercond. Sci. Tech., 25, 065014 (2012).
- [8] D. Gonnella et al., Proceedings of the Sixth International Particle Accelerator Conference, WEPTY072 (2015).
- [9] R. P. Huebener, Magnetic Flux Structures in Superconductors (Springer, Berlin, 1979).
- [10] G. Ciovati and A. Gurevich, Phys. Rev. ST-AB, 11, 122001 (2008).
- [11] A. Romanenko et al., App. Phys. Lett., 105, 234103 (2014).
- [12] H. Padamsee, private communication.
- [13] W. Singer et al., Phys. Rev. ST-AB, 16, 012003 (2013).

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