# BITTER DECORATION STUDIES OF MAGNETIC FLUX PENETRATION INTO CAVITY CUTOUTS

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# Abstract

Magnetic flux penetration may produce additional losses in superconducting radio frequency cavities. All the existing models for flux penetration are based on the formation of Abrikosov vortices. Using Bitter decoration technique, we have investigated magnetic flux distribution patterns in cavity cutouts at perpendicular magnetic fields of 10-80 mT in field cooling regime and up to 120 mT in zero-field cooling regime. At low fields <20 mT the magnetic field penetrates in the form of flux "bundles" and not Abrikosov vortices, the situation characteristic of type-I superconductors. With the increase of the magnetic field up to  $\sim 30 \text{ mT}$ "bundles" first merge into a connected structure and then break up into individual Abrikosov vortices at ~60 mT and a well-known intermediate mixed state is observed. Such magnetic field driven transition from type I to type II superconductivity has never been observed before in any existing superconductor. For the case of flat samples, we have observed a coexistence of both "bundles" and Abrikosov vortices in one experiment. Our results show that high-purity cavity grade niobium is a "border-line" material between type-I and type-II superconductors.

## **INTRODUCTION**

Superconducting radio frequency (SRF) cavities are the primary particle accelerating systems in many modern accelerators and also a technology for future projects. The main advantage of SRF cavities is an extremely low surface resistance in the superconducting state and hence a very high quality factor. A lot of studies have been produced recently in an attempt to overcome limitations encountered in niobium cavities, such as high-field Q-slope (HFQS) and quench. Nevertheless, the physical mechanisms of SRF losses are not yet fully understood. Among the possible sources for SRF losses is magnetic flux penetration into the superconducting layer. Using high resolution Bitter decoration technique we have studied magnetic field distribution patterns in cavity cut-outs with different SRF losses and also in cavity grade niobium samples at perpendicular magnetic field. The field orientation was different from SRF geometry but still we hoped to extract important information about superconducting properties of the material.

#### **EXPERIMENTAL**

Using the temperature mapping system at Jefferson Lab, we identified locations exhibiting strong and weak RF

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losses. Circular samples of 12 mm diameter were extracted from the selected locations using the automated milling machine. We also used flat samples cut by wire electron discharge machining from fine grain RRR  $\sim 300$  niobium sheets.

The decoration was performed by means of sputtering of iron in helium atmosphere at pressure ~0.1 Torr onto the sample surface at temperatures below  $T_c$ . The tiny magnetic particles (several nm), formed directly near the sample surface during the sputtering process, reached the sample guided by interaction with the magnetic field and concentrated at the areas where magnetic field penetrated the surface. The temperature was measured by a resistive thermometer fixed to the substrate near the sample. After decoration, the sample was warmed up to room temperature and transferred to a scanning electron microscope (SEM) to study the distribution of the magnetic particles.

The experiments were carried out in both field-cooling (FC) and zero field-cooling (ZFC) regimes at temperatures 5,5-6,5 K and magnetic fields up to 80 mT FC/120 mT ZFC directed perpendicular to the sample surface.

#### RESULTS

Successful imaging of flux distribution patterns was achieved in the field-cooling regime at magnetic fields of 10, 32, 60 and 80 mT for hot (with high SRF-losses) spots and 10 and 60 mT for baked (with low SRF-losses) spots. At those magnetic fields where both hot and baked spots were decorated, we did not see any difference between the results for different samples.

At 10 mT, decoration patterns reveal a very interesting feature, namely, no Abrikosov vortices are observed. A typical SEM microphotograph is shown in Figure 1. White are the regions with iron particles where magnetic field penetrated the sample, while dark ones are the regions without magnetic particles, i.e. Meissner ones. If magnetic field penetrated in the form of Abrikosov vortices, their characteristic size would be several tens of nanometers and intervortex distance would be half a micron. But we see only much larger structures - "bundles" of a typical size of more than 1  $\mu$ m. Magnetic field distribution inside such a bundle seems to be homogeneous (see Figure 1b).

The magnetic flux structure changes drastically when external magnetic field is increased. The pattern at a field of 32 mT is presented in Figure 2. Still there are alternating regions with magnetic field and Meissner ones, but their topology is completely different from that at 10 mT. Single bundles are now merged into a "connected" structure. Still

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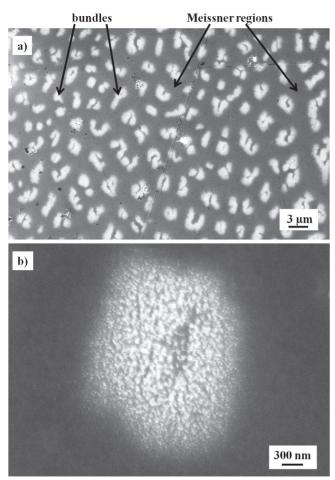


Figure 1: a) Decoration pattern at the magnetic field 10 mT reveals coexistence of "bundles" and Meissner regions. Each bundle carries 80 magnetic flux quanta in average; b) zoom-in of a single bundle.

no individual vortices are seen at high magnification.

At 60 mT there is still a coexistence of alternating magnetic and Meissner regions, but the internal structure of magnetic regions is different - now they consist of individual Abrikosov vortices (see Fig. 3b). Six clearly seen sharp maxima in the Fourier transform pattern reflect the long-range order in the vortex structure. The vortex density corresponds to a magnetic induction  $B_0$  of 760±50 Gs for a hot spot and 740±50 Gs for a baked spot.

At 80 mT, Meissner regions disappear completely and we see vortex lattice everywhere.

We have also carried out several experiments in the zerofield cooling regime, where magnetic field was introduced after cooling a sample down below critical temperature. The maximal value of the magnetic field was 60 mT for hot spots and 120 mT for baked spots. We did not observe any penetration of magnetic field into samples in these experiments.

To investigate if magnetic flux distribution is affected by sample geometry, we have carried out two experiments with differently treated flat samples in FC regime at 8 <u>а</u> <u>р</u> <u>а</u> <u>а</u>

Figure 2: Magnetic flux structure for a hot spot at the magnetic field 32 mT - "bundles" merge into a connected structure : a) general view ; b) zoom-in; c) highest-magnification image shows that no individual vortices are resolved.

mT. One sample was just mechanically polished and 800° baked for 3 hours at high vacuum and the other was subjected to the same treatments plus 20  $\mu$ m buffered chemical polishing material removal. We have found no difference between these two samples but there is a significant difference in comparison with cavity cut-outs. For the case of flat samples, we observe coexistence of regions with "bundles" and regions with Abrikosov vortices right next to each other (see Fig. 4) in one experiment.

### DISCUSSION

Low-kappa ( $\kappa\approx 1)$  superconductors like pure niobium have a specific magnetic structure. At magnetic fields H

300 nm

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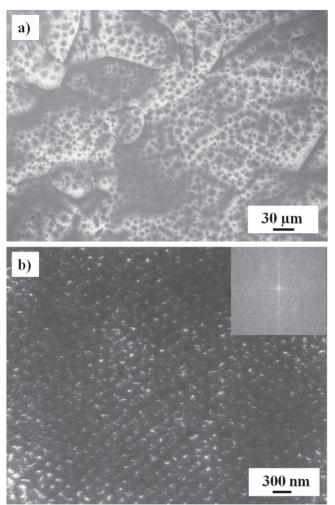


Figure 3: Magnetic flux structure at the magnetic field 60 mT: a) general view - coexistence of magnetic and Meissner regions ; b) individual vortices are resolved inside magnetic regions at high magnification. Insert: Fourier transform pattern.

satisfying  $H_{c1}(1 - D) < H < H_{c1}(1 - D) + DB_0$ where  $H_{c1}$  is a lower critical field,  $B_0$  - material-dependent constant and D - demagnetization factor, an intermediate mixed state (IMS) consisting of alternating Meissner (with zero magnetic inductance) and Shubnikov (with magnetic inductance  $B_0$  ) phases [1] is formed. The reason is the nonlocal character of the electrodynamics of low- $\kappa$  superconductors, often referred to as "type II/1" superconductors (in contrast to 'type II/2" superconductors with  $\kappa \gg 1$ ). This behavior in some respects resembles type-I superconductors with the difference that there are Shubnikov regions with individual Abrikosov vortices instead of normal domains.  $B_0$  plays the role of  $H_c$  for type-I superconductors.

What we observe at high fields starting from 60 mT is a typical IMS. However structures at low fields are different and to our knowledge have never been observed before.

According to magnetic flux conservation law, magnetic inductance inside fluxoids at 10 mT must be  $\approx 400$  Gs

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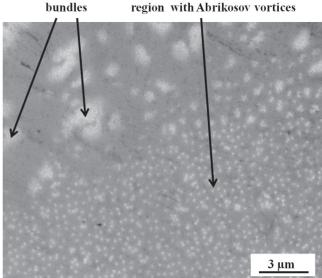


Figure 4: Decoration pattern for a flat sample at the magnetic field 8 mT reveals coexistence of regions with bundles and Abrikosov vortices.

(flux-free regions occupy 75 % of the sample surface). The strange point to be mentioned is that this value is significantly lower than both the thermodynamical critical field  $H_c$  (2000 Gs for Nb) and  $B_0$  at high fields (750 Gs). The average number of magnetic flux quanta per bundle is  $\sim 80$ . If the bundle consisted of individual vortices, the intervortex distance would be  $\sim 200$  nm. Since we clearly resolve vortices at 60 and 80 mT, insufficient spatial resolution cannot be the reason for observation of bundles.

Our interpretation of results at low fields < 60 mT is that the magnetic field distribution inside bundles is uniform, as is typical of type-I superconductors with  $\kappa < 1/\sqrt{2}$ . Indeed, very similar structures consisting of "bundles" carrying many magnetic flux quanta were observed for the case of a type-I superconducting lead [2]. Another possible, but in our opinion less realistic, scenario is that individual vortices are extremely mobile within the bundles, and the spatially uniform field is the result of averaging through the time of decoration process (several hundreds milliseconds). In both cases there must be something that prevents vortices from leaving the bundle.

Hence we can conclude that there is a field-driven transition from "type I" to "type II" superconductivity. Usually such results are explained by change of  $\kappa$  from below  $1/\sqrt{2}$ to above  $1/\sqrt{2}$ . For example, temperature dependence of  $\kappa$  [3] was assumed to be the reason for "type I"-"type II" switch. But in classical models,  $\kappa$  does not depend on external magnetic field. The reasons for the field-driven transition are unclear for us and should motivate further theoretical studies.

Both characteristic size of bundles and "interbundle" distance are of the order of several microns. This coincides with the characteristic scale of inhomogeneities in

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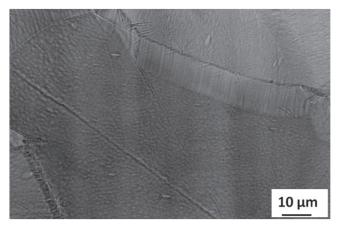


Figure 5: Sample surface after buffered chemical polishing.

the distribution of dislocations over the sample obtained by EBSD-based microscopy. The latter can also be roughly estimated from sample microphotographs after buffered chemical polishing (see Fig.5) - regions with different dislocation densities have different etching rates. The coincidence of the sizes of bundles and dislocation tangles allows us to suggest that vortex interaction with dislocations may be of importance.

Results observed on flat samples demonstrate that  $\kappa$  in cavity-grade niobium is indeed very close to  $1/\sqrt{2}$ . The energy difference between "bundle" and "individual vortices" configurations is very small and even slight spatial variations of superconducting parameters can make either of them preferable. Any correction to Ginzburg-Landau model, neglected under any other conditions, may become important. The big difference in results observed on flat samples and cavity cut-outs can be attributed to different type of interactions via stray-fields.

We have obtained the same results on hot and cold spots. We think that for the geometry used in the experiments the total magnetic energy is dominated by the bulk and we can not make conclusions about surface superconducting properties. Magnetic field parallel to the samples still may penetrate differently in hot and cold spots.

Zero-field cooling experiments demonstrate that pinning must be taken into account when considering magnetic field penetration into the sample. For the case of pinningfree samples of such geometry, a magnetic field must have already penetrated the samples earlier [4]. Another result indicating importance of pinning is that intervortex distance in Fig. 4 corresponds to an external magnetic field  $H_{applied}$ , not to  $B_0$ .

## CONCLUSION

Using high-resolution Bitter decoration technique we have investigated how magnetic flux penetrates cavitygrade niobium samples at different applied fields perpendicular to the surface.

Zero-field cooling results show the importance of pinning. It prevents magnetic field penetration deep into the

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sample up to magnetic fields of at least 120 mT.

We have observed field-driven phase transition from "bundle-type I" to "intermediate mixed state - type II" superconductivity in both hot- and cold-spot cutouts. For the case of flat samples we have observed coexistence of "type-I" and "type-II" regions in one experiment.

All these features – importance of pinning, nearly vanishing or even attractive intervortex interaction, influence of stray fields – must be taken into account to build a correct model for SRF losses due to flux penetration. Simple theoretical models, taking only Abrikosov vortices into consideration, are not satisfactory.

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