CHARACTERIZATION OF SRF MATERIALS AT THE TRIUMF μSR FACILITY

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Abstract

μSR is a powerful tool to probe local magnetism and hence it can be used to diagnose flux penetration in Type-II superconductors. Samples produced at TRIUMF and with collaborators in both coin shaped and ellipsoidal geometries have been characterized by applying either transverse or parallel fields between 0 and 300mT and measuring flux entry as a function of applied field. Samples include Nb treated in standard ways including forming, chemistry, and heat treatments. Further, Nb samples have been doped with Nitrogen and coated with a 2 micron layer of Nb3Sn by collaborators from FNAL and Cornell respectively and measured in three field/geometry configurations. Analysis of the method in particular the effects of geometry and the role of pinning will be presented. Results of the measurements will be presented.

INTRODUCTION

μSR (muon spin rotation) is a powerful condensed matter technique to understand superconductors in terms of their magnetic-phase diagram and penetration depth, as well as characterize impurities based on muon diffusion. In the early 1970's new high-intensity, intermediate-energy accelerators were built at PSI, TRIUMF and LAMPF. These new "meson factories" produced pions (and therefore muons) several orders of magnitude more than previous sources - and in doing so, ushered in a new era in the techniques and applications of μSR. Since 2010 the SRF group at TRIUMF has been using the μSR technique to characterize materials and processing techniques typical for the SRF community using the TRIUMF surface muon beam [1]. Typical samples have been prepared from RRR Nb either as coins (flat and formed) and in a machined ellipsoid geometry. The samples are then treated with a number of surface and bulk techniques and subsequently tested with μSR to determine characteristics of the superconducting state. This paper gives details on the samples and preparation, geometrical effects and measurement results.

μSR TECHNIQUE

Surface muons are emitted from a production target 100% spin polarized with momentum and energy of 29.8MeV/c and 4.1MeV respectively and are implanted one at a time into the sample. When the muon decays (half life=2.2μsec) it emits a fast decay positron preferentially along the direction of its spin at the time of the decay. By detecting the rate of emitted positrons as a function of time with two detectors placed symmetrically around the sample the time evolution of the spin precession of the muon and therefore the magnetic field properties experienced by the muon can be inferred from the time dependent asymmetry in the positron decay.

The samples are placed in a cryostat surrounded by field inducing coils. Field penetration measurements are primarily done by cooling the sample to below T_c (2K is common) in zero field and then applying a static magnetic field perpendicular to the initial spin polarization to see if field is in the sample. Specifically the asymmetry signal gives information on the volume fraction of the host material sampled by the muon that does not contain magnetic field.

This signal can be used to characterize the superconducting state, particularly the transition from Meissner state to mixed state. When completely in the Meissner state there is no field in the sample and the asymmetry is maximized. As the field increases flux will eventually enter the superconductor as it enters the mixed state and the asymmetry signal will be reduced. Further the rate of de-coherence of the asymmetry signal gives a measure of the non-uniformity of the sampled field.

The muon beam has a Gaussian transverse distribution with a physical half-width of approximately 8mm. The spin-polarized muons are implanted into the sample, and quickly stop at interstitial sites in the bulk. The energy of the surface muons means that they are deposited about 150 μm into the sample so the measurement represents a bulk as opposed to a surface probe.

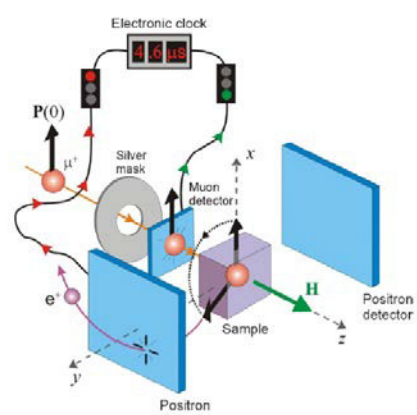

Figure 1: TF-μSR setup with the initial muon spin polarization perpendicular to the magnetic field. The silver mask in front of the sample restricts the muon implantation to the central region of the sample.

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The geometry of the transverse field (TF) measurement is illustrated in Fig. 1. The muons travel down the beam line and pass through an initial muon counter (scintillator) that starts an electronic clock. The muons then pass through a silver mask with an 8mm diameter hole in the centre used to restrict the measured muons to the centre of the sample. The muons are counted one by one with the positron decay stopping the clock.

A new beam-line has been added to test the samples with a field parallel to the beam direction up to 300mT [2]. The field that is used to probe the sample also bends the muons so that an upstream steering magnet is used to pre-steer the muons off-axis and the applied field at the sample bends the muons back to the sample. For coin samples the muon is delivered to the front face. A future plan is to place ellipsoids in this parallel geometry and deliver muons to the equator. A summary of the various samples, muon applications and field directions for our μSR studies are shown in Fig. 2.

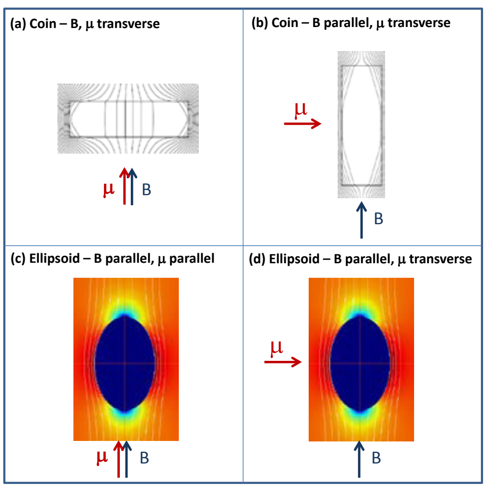

Figure 2: Four generic arrangements of sample (coin and ellipsoid), muon and field direction for the sample characterization studies at TRIUMF.

SAMPLES

Three generic sample types are used. Coin samples of 3mm thickness and 20mm diameter are cut by water jet from flat sheet of RRR Nb material. Similar coins were cut by wire EDM from a 1.3GHz half-cell formed from 3mm RRR material at a location 45º from the equator as rotated toward the iris. A third sample was machined out of bulk RRR Nb in the shape of a prolate ellipsoid. The dimensions of the ellipsoid are semi-major axis of 22.9mm and semi-minor circular cross-section of 9.0mm radius. Moreover, along the major axis, at one end there is a 21mm deep 1/4-20 threaded hole which was used to hold the sample. Figure 3 shows all three geometries.

The samples were subjected to a variety of different treatments typical for SRF cavity processing. These included heat treatments in vacuum such as 120C bake for 48 hours and 800C degassing for 4 hours. Vacuum heat treatments at 1200C and 1400C for 4 hours each were also employed. Surface treatments include surface etching using both buffered chemical polish (BCP) and electro-polish (EP) with various removals.

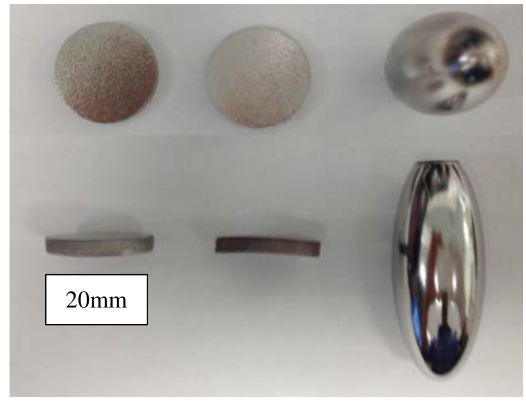

Figure 3: Example of the samples used in the experiment: plan view and side profile for flat coin, formed coin and ellipsoid.

EFFECT OF GEOMETRY

Consider first the coin sample with field applied perpendicular to the face (Fig. 4). When in the Meissner state surface currents will be set up to cancel the field in the bulk. The magnetic field will be enhanced at the edges of the coin by a factor related to the demagnetizing factor N by $H_{edge}=H_{applied}/(1-N)$ where $H_{applied}$ is the applied field. For Type II superconductors when the applied field is such that the enhanced field at the edges reaches H_{c1} the field will break into the edge such that the local field is reduced due to the rounding of the flux line. As the field increases the flux lines will cut further across the corner and eventually join at the centre of the sample edge. This corresponds to $H_{applied}|_{entry}$ and is higher than $H_{c1}*(1-N)$ due to the so called edge boundary [3]. The flux line now crosses the full sample width and is driven inwards due to interaction with the surface currents.

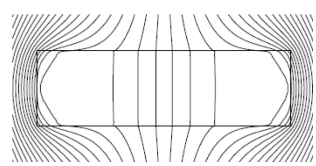

Figure 4: Flux applied to a thin circular disk transverse to an applied field where $H_{applied}>H_{applied}|_{entry}$.

In a pin-free sample the flux will move to the centre since this represents the lowest energy position (minimum line tension). As the flux increases and vortices multiply the vortex currents will repel so that the flux lines will redistribute and fill from the centre to the outside edge. In our case for the standard coin geometry, with diameter a=20mm and thickness b=3mm, the demagnetizing factor is $N=0.77$ meaning that $H_{applied}|_{edge}=0.23H_{c1}$ while

$$H_{applied}\big|_{entry} = \tanh\left(\sqrt{0.67\frac{b}{a}}\right)\cdot H_{c1} = 0.31 H_{c1}. \quad (1)$$

For a sample with pinning the pinning centres act as additional barriers adding 'resistance' to the mobility of vortices moving from the edges to the centre and increasing $H_{applied}|_{entry}$ compared to the pin free case.

For the ellipsoidal geometry the edge boundary is eliminated. The inward directed driving force on the vortex ends by the surface screening currents is compensated by the vortex line length that increases for fluxoids that are closer to the ellipsoid axis – so pin-free ellipsoidal samples produce a uniform vortex flux density in the mixed state. The Meissner state is supported by screening currents that augment the field at the equator and reduce the field at the poles. When the flux at the equator reaches $H_{nucleate}$ (H_{c1} or H_{sh}) fluxoids will nucleate at the equator and redistribute uniformly inside the superconductor due to vortex repulsion for a pin free sample as illustrated in the cartoon shown in Fig. 5.

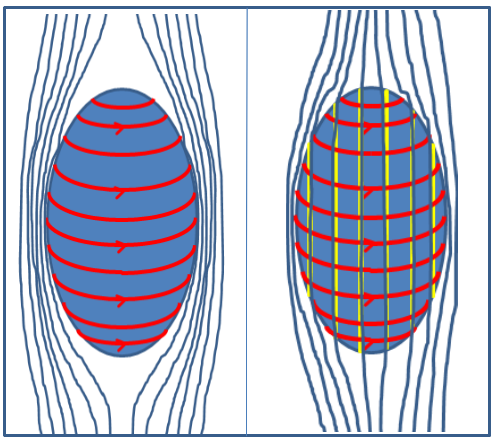

Figure 5: Schematic showing the magnetic flux just before and after flux nucleation at the ellipsoid equator.

In our geometry the demagnetizing factor is N=0.13 with $H_{applied}|_{entry}=0.87H_{nucleate}$ [3]. In the case of samples with pinning the redistribution will be affected as the pinning centres will add a frictional component to the redistribution such that the fluxoids will tend to preferentially populate nearer the equator and will only gradually reach the poles as the applied field increases beyond $H_{applied}|_{entry}$.

In the parallel geometry (Fig. 2 b,d) the sample coin or ellipsoid is placed parallel to the applied field and the muons are applied to the coin face or the ellipsoid equator [2]. In a parallel geometry the coin has an estimated demagnetization of N=0.22 and $H_{applied}|_{entry}$=0.91$H_{nucleate}$ for the pin free case. In this geometry the volume sampled by the muons is less sensitive to pinning. Flux could still be pinned at the corners before linking at the centre (pinning enhanced edge boundary) but much less so than in the transverse geometry since no flux motion is required. The parallel field ellipsoid geometry with an application of muons at the equator (Fig. 2d) is fully insensitive to pinning and will be our preferred geometry going forward to look for H_{c1} vs H_{sh} limits.

RESULTS

Comparing Geometries

We present several results from different samples first to illustrate the effect of the geometry. In these and subsequent plots the field is normalized to H/H_0 where H_0 corresponds to the expected entry field for a pin-free sample with demagnetization and edge boundary considered assuming a generic $H_{c1}|_{0K}$=180mT or $H_{c1}|_{2.5K}$=165mT. Table 1 gives the estimated H_0 values for the three geometries.

Table 1: Geometrical normalizing factors for expected pin-free flux entry for the three sample types assuming $H_{c1}|_{0K}$=180mT)

Sample	N	$H_{applied}|_{entry}/H_{c1}$	H_0 (mT)
Transverse coin	0.77	0.31	51
Parallel Coin	0.2	0.91	150
Ellipsoid	0.13	0.87	144

Figures 6 and 7 show the normalized asymmetry as a function of applied field for three different sample/field arrangements with a transverse and parallel coin geometry (Fig. 2a,b) and an ellipsoid geometry with muons at the pole (Fig. 2c). In Fig. 6 the samples are heat treated at 1400C and in Fig. 7 at 800C. From these and other plots it's evident that a 4 hour treatment at 1400C is sufficient to greatly reduce the pinning in the material. Note that in Fig.1 there is good agreement between the expected and actual $H_{applied}|_{entry}$ with the sharpest signature from the parallel coin sample closely followed by the ellipsoid. The transverse coin geometry results show that over the region of the face sampled by the muons a field of up to a factor of two higher than $H_{applied}|_{entry}$ is needed to fully saturate the volume with field.

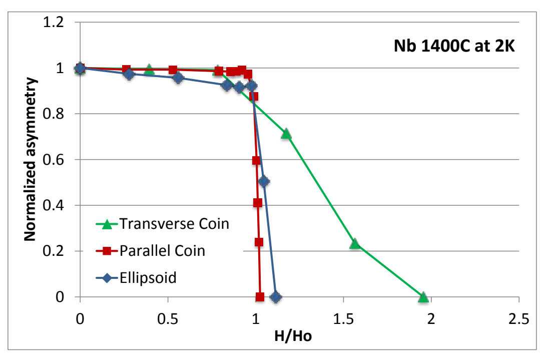

Figure 6: Normalized asymmetry plots showing fraction of sampled volume devoid of magnetic field as a function of applied field normalized to the expected $H_{applied}|_{entry}$ based on the demagnetization factor of the geometry. Shown are plots for samples annealed at 1400C to reduce pinning - a coin in transverse and parallel geometry and an ellipsoid with geometry as in Fig. 2c.

In comparison in Fig. 7 the samples treated to 800C are not fully annealed and both the ellipsoid sample and, more markedly, the transverse coin geometry show signs of pinning delaying flux entry.

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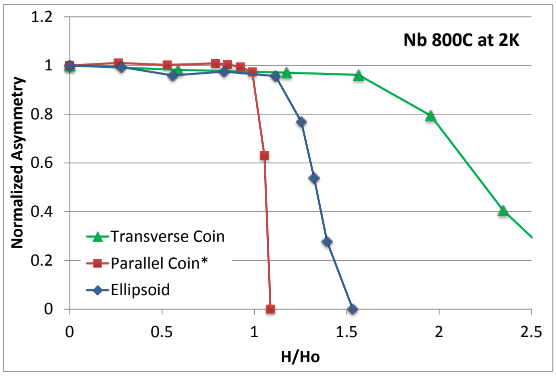

Figure 7: Similar to Fig. 6 but with samples treated at 800ºC (parallel coin is N-doped at 800C and then given an EP of 5µm) showing sensitivities of different geometries to pinning.

Transverse Coin Results

To get more information on how the flux breaks in for the case with pinning a series of measurements are taken with different masking foils: a standard 8mm aperture, an annular mask with radius from 4-6 mm and an annular mask with radius from 6-8mm. The results are plotted in Fig. 8 once again with fields normalized to the expected $H_{applied}|_{entry}$ based on the geometry. The results show that for the case where pinning dominates the field breaks in near $H_{applied}|_{entry}$ at large radii but does not migrate to the centre as would be expected for a pin free case. In this case the flux is not driven to the centre until the field reaches over 2 times the pin-free $H_{applied}|_{entry}$ and is not fully saturated until over three times $H_{applied}|_{entry}$.

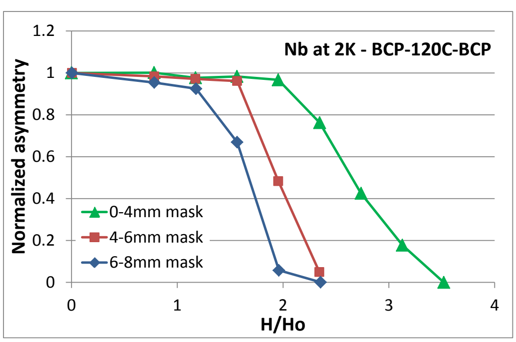

Figure 8: Field entry as a function of position on the sample for transverse coin geometry for a sample with a treatment of BCP-120C bake-BCP.

These runs show that the transverse geometry is especially sensitive to pinning in the sample. The following plots are chosen to show how various surface and bulk treatments can affect the pinning as demonstrated by the asymmetry response to the applied field in the transverse geometry. Figure 9 shows results from a thinner coin. An etched RRR sample with strong pinning is then heat treated at 1400C with a significant decrease in pinning. When the annealed sample is etched again the pinning does not return showing that the pinning is a predominantly a bulk rather than a surface effect.

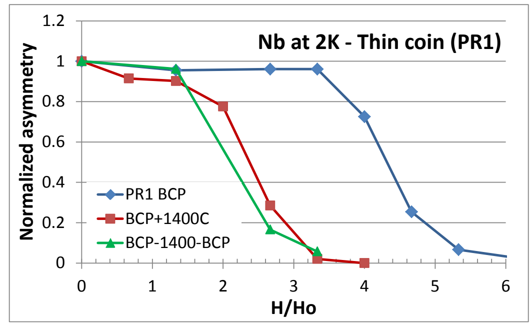

Figure 9: Results from transverse coin sample (PR1 – 0.8mmx16mm) after BCP treatment followed by annealing (1400C) followed by another BCP.

The results of Fig. 10 highlight that forming greatly enhances the level of pinning. Here formed samples with BCP, 800C bake are compared to flat samples with BCP and 800C treatments. Pinning in formed samples delay flux entry to three times higher field as compared to annealed samples. 800C treatment does relax pinning somewhat in both flat and formed cases.

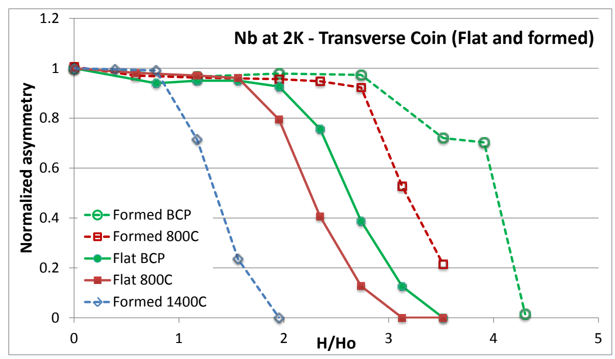

Figure 10: Results from transverse coins of formed and flat geometries. Formed samples are denoted with dashed lines. Red lines are samples treated at 800C for 4 hours; green lines are for samples after BCP; blue line is a sample annealed at 1400C.

Ellipsoid Results

The ellipsoid is positioned as in Fig. 2c with long axis coincident with the muon beam and aligned with the applied field. An 8mm diameter mask is used to confine the muons to the pole of the ellipsoid. The ellipsoids receive*d* various bulk and surface treatments as with the coins. In general pinning is less predominant in the ellipsoid due to the lower demagnetization factor. However pinning is still a factor as the fluxoids will nucleate at the equator and must overcome pinning to move to the pole. Heat treatments to 1400C are shown to be effective to strongly reduce pinning as with the transverse coins. Figure 11 shows results from three ellipsoids with four hour heat treatments of 1400C, 1200C and 800C respectively. In both 1400C and 1200C the field entry has a sharp threshold characteristic of uniform entry while the 800C sample shows entry of fields at the pole for higher applied fields with an extended tail to $1.5H_0$.

Another set of studies was done comparing N-doped and non-N-doped material. The N-doping [4] was done at FNAL. The doping involves heating a sample to 800C for four hours and injecting N_2 gas near the end of the treatment. The set of ellipsoid samples in Fig. 12 compares flux entry for samples with EP (30µm), with EP+N-dope, with EP+N-dope+EP (5 µm) and with EP+800C heat treatment. The results indicate that the 800C bake (including N-dope) in all samples reduces the pinning compared to the sample with only EP. Further the N-dope (a surface process) increases the pinning while the standard EP removal of 5 µm reduces the pinning back to the same levels after only 800C treatment.

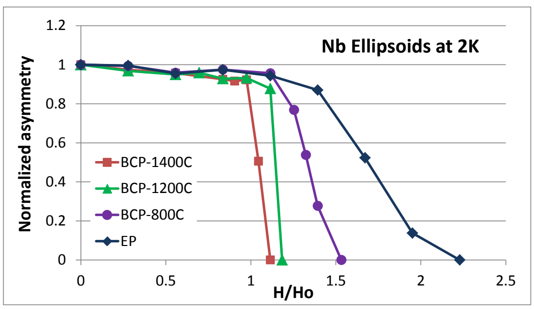

Figure 11: Ellipsoid samples heat treated at 1400C, 1200C, and 800C respectively for four hours each assuming $H_{nucleate}|_{0K}$=180mT.

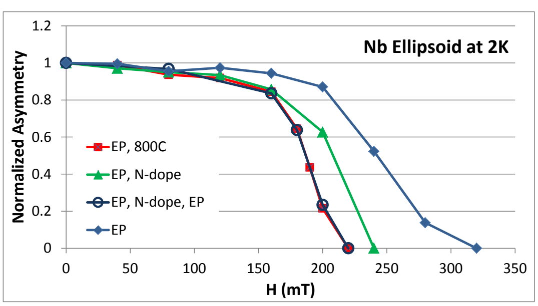

Figure 12: Ellipsoid samples treated at EP+800C and with N-doping compared to no heat treatment (Ep only).

Coatings

µSR has also been used to characterize Nb3Sn coatings. Collaborators at Cornell coated a standard Nb coin and ellipsoid with a 2µm coating of Nb3Sn using their standard recipe [5]. The samples coins were tested in both transverse and parallel geometries and the ellipsoid tested with muons applied to the pole as in Fig. 2c. The normalized results of all three data sets are summarized in Fig. 13. The transverse coin results indicate that the pinning in the sample is quite weak. This is understandable since the Nb3Sn application involves a heat treatment to 1100C. Also noteworthy is that the transverse results do indicate a low field flux entry. The ellipsoid compares closely to the parallel result also indicative of low pinning. What is interesting is that the coating has pushed out the field of first flux entry to about 1.3 times the standard Nb values meaning that $H_{nucleate}|_{0K}$ is enhanced to 230mT [6].

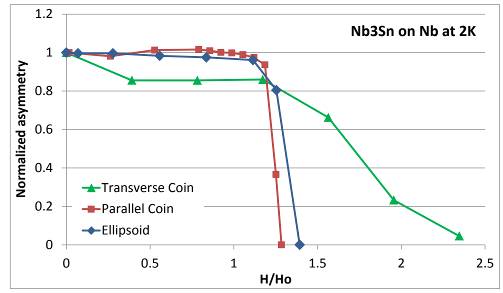

Figure 13: Coin and ellipsoid sample results at 2K for a 2µm coating of Nb3Sn onto Nb.

CONCLUSIONS AND FUTURE WORK

The various sample shapes and test configurations have been established and are crucial to the interpretation of the results. Bulk pinning in the material changes considerably depending on the bulk and surface treatments. A 1400C heat treatment virtually eliminates pinning and surface treatments like BCP do not erase the effect. However a sample with strong pinning can have the pinning enhanced with BCP and 120C bake. Pinning is an important parameter for SRF since background flux can be more readily trapped during cooldown.

Future work will involve testing ellipsoid samples by applying muons to the equator as in Fig. 2d. Here we expect to see results completely free from the pinning strength. Plans are underway to add a new spectrometer to the β-NMR facility at TRIUMF to allow testing samples in strong parallel fields within the London layer.

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