A study of vortex dynamics in patterned superconducting thin films

Jonathan George
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CERTIFICATION

I, Jonathan George, declare that this thesis, submitted in partial fulfilment of the requirements for the award of Master of Philosophy, in the School of Physics/Institute for Superconducting & Electronic Materials, Faculty of Engineering & Information Sciences, University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other academic institution.

Jonathan George
31 August 2016
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A STUDY OF VORTEX DYNAMICS IN PATTERNED SUPERCONDUCTING THIN FILMS

Jonathan George

A Thesis for Master of Philosophy

School of Physics/Institute for Superconducting & Electronic Materials
University of Wollongong

ABSTRACT

This thesis can be divided into two primary works, examining the effects of uniform and non-uniform pinning arrays of antidots in YBCO thin films, and optimising the deposition of Nb thin films. The ultimate goal was compare the poorly investigated effect of pinning arrays of large antidots in Nb and YBCO thin films, which have different intrinsic pinning properties and different vortex sizes.

The non-uniform graded pinning arrays etched into YBCO thin films such as graded, inverted and evenly spaced rings of triangular antidots indicated the success of novel artificial pinning arrays of large antidots could come from the suppression of interstitial flux channels between the edge of the film and the centre, and from some suppression of flux hopping. However, the success of these non-uniform arrays may also be attributed to the formation of a 'vortex vacuum' if vortices penetrate the thin film in a non-Bean like flux distribution because of the antidot array. This work is now published in Annalen der Physik in 2017 (J. George, et al., Ann. Phys. (Berlin), 1600283 (2017) / DOI 10.1002/andp.201600283).

To begin investigations into Nb, the first step was optimise the process of deposition to reliably produce consistent thin films, and then start investigating pinning arrays. Despite the obstructions in this stage it was determined that in order to deposit films via DC Magnetron Sputtering, the sputtering chamber needs to be exceedingly clean, and free from contaminants, and a deposition temperature of 350°C strikes an ideal balance between vortex pinning and current transparency. Unfortunately the system from Mantis Deposition Systems continues to remain excessively fragile and prone to
breakages and Nb thin films could not be consistently deposited for new research into pinning arrays of large antidots.

**KEYWORDS:** Superconductivity, vortices, large artificial pinning centres
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Thank you all, gg,
Jonathan
Chapter 1

Literature review

1.1 Introduction

The magnetic properties of superconductors are of great interest to researchers because they have a wide range of applications. This interest has unveiled a complex system of properties to be investigated, and this review will begin by differentiating some of them. Superconductors can be firstly classified into both type-I and type-II superconductors. Type-I superconductors are the simplest type and will exclude the magnetic field almost completely, below some critical field $B_c$ when it is in the superconducting state, regardless of the magnetisation of the sample. When the magnetic field is expelled as the material cools through the superconducting transition, it is known as the Meissner-Ochsenfeld effect, and is presented in Figure 1.1. Once above this field the sample leaves the superconducting state to enter the normal state. Some field will remain this point and penetrates a short distance $\lambda$ into the material. This distance is dependent on both the temperature of the sample and the properties of the sample itself\(^{45}\), it is known as the London penetration depth, where the sample remains in the superconducting state. A superconductor is superconducting when it is below some critical temperature $T_c$, which is $9.2K$ for Niobium\(^{45}\). The phase
diagram representing the transition between the normal and superconducting phases for a type-I superconductor is presented in figure 1.3 a).

Type II superconductors have a more complex response to the presence of a magnetic field, and as such are a great source of interest and challenges. When a type II superconductor such as Nb is placed in a magnetic field and is cooled below $T_c$, the field is completely excluded below some critical field $B_{c1}$. Above this threshold quantised bits of magnetic field enter the sample in the form of vortices. A quantum of magnetic flux surrounded by a superconducting current that penetrates a distance $\lambda$ into the superconducting space around it.

As the magnetic field increases, more flux lines or vortices penetrate the sample and more normal state regions appear, where at some higher second critical field $B_{c2}$, so many vortices have now entered the sample that vortex cores are touching and the average magnetisation of the sample is now equal to the external field leaving the interior in the normal or non-superconducting state. The surface may retain some superconductivity until the magnetic field reaches some third critical point $B_{c3}$ if the field is parallel to the surface.
The region between fields $B_{c1}$ and $B_{c2}$ is known as the Shubnikov phase, as shown in Figure 1.3 b), and it is here that vortices first enter the material. A vortex in a type II superconductor is a cylindrical region consisting of a normal core, containing a single flux quantum, with its axis parallel to the external magnetic field, and surrounded by a circulating supercurrent of radius $\lambda$, which is known as the London penetration depth. The radius of the core is given by the Ginzburg-Landau coherence length $\xi_{GL}$. Figure 1.2 presents a schematic of the vortex typically found in a type II superconductor.

Vortex motion defines the critical current of a superconductor. Given that each vortex contains a single flux quantum, if it is forced to move by an applied current, which is then known as the critical current, an electric field is generated in the same direction as the applied current and the sample enters a resistive state. If a superconductor is perfectly homogeneous then vortices experience no resistance to motion besides some viscous damping, rendering superconductivity an unusable phenomenon. All superconducting materials contain some defects, and these defects act as pinning centres, which are essential for superconductivity since they provide a region where it is ener-
1.1. Introduction

Temperature $T$ Applied Magnetic Field $B_c$
Superconducting Normal $B_{c1}$ $B_{c2}$
Superconducting Normal Shubnikov $T_c$
a) b)

Figure 1.3: Superconducting phase diagrams a) Type-I superconductor b) Type-II superconductor

Figure 1.4: Energy schematic of a potential well formed by a pinning centre

...getically beneficial for vortices to remain$^{48}$. This benefit comes from the reduction in the vortex length through the superconducting material where it passes through the defect, and a reduction in the length decreases the energy required to produce the vortex$^{54}$. This area of lower free energy, acts as a potential well or pinning centre, holding the vortex in place until some other force overcomes this.
1.1. Introduction

<table>
<thead>
<tr>
<th>Quantity</th>
<th>YBCO</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transition temperature</td>
<td>92 K</td>
<td>9.2 K</td>
</tr>
<tr>
<td>Coherence length, $\xi_{ab}$</td>
<td>1.5 nm</td>
<td>40 nm</td>
</tr>
<tr>
<td>Coherence length, $\xi_c$</td>
<td>0.2 nm</td>
<td></td>
</tr>
<tr>
<td>Penetration length, $\lambda_{ab}$</td>
<td>150 nm</td>
<td>40 nm</td>
</tr>
<tr>
<td>Lower Critical field, $B_{c1}$</td>
<td>10 mT</td>
<td>0.1 T</td>
</tr>
<tr>
<td>Upper critical field, $B_{c2}$</td>
<td>300 T</td>
<td>0.3 T</td>
</tr>
</tbody>
</table>

Table 1.1: Fundamental lengths of YBCO and Nb\textsuperscript{47}

1.1.1 Studied Materials

The type-II superconducting thin films used in this thesis are Niobium and YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7}. A low temperature superconductor like Nb is expensive and difficult to use, because of its low $T_c$, but it still has a number of applications and plays a critical role in understanding superconducting science. It serves as a base for superconducting alloys like Nb\textsubscript{3}Sn and NbTi that are used in MRI machines and particle accelerators. Historically Nb was also used to study the properties of type-II superconductors as Ginzburg-Landau-Abrikosov-Gor’kov theory\textsuperscript{12}. The relatively large coherence length of 40 nm for perpendicular applied fields makes it useful for the type of research outlined in chapter 3. A high temperature superconductor like YBCO is far easier to cool below its $T_c$ of $\approx$ 90 K. It was one of the early types of layered cuprates, and is now relatively simple to fabricate in thin films, bulk samples, and tapes. YBCO will crystallise in perovskite structure as shown in figure 1.5. YBCO presents a relatively novel case for the application of large applied pinning centres because of its small coherence length as mention in table 1.1. The investigation of artificial pinning arrays in both YBCO and Nb will provide not only crucial insight into the pinning mechanisms that take place for large antidots. Table 1.1 summarises the fundamental values that determine superconducting phenomena in each material. The Ginzburg-Landau parameter $\kappa = \lambda/\xi$ about 100:1 for YBCO:Nb is one of the sources of analysable vortex behaviour differences between the two superconductors\textsuperscript{45}.  


1.1. Introduction

Figure 1.5: YBCO crystallography include lattice parameters in a, b, and c directions\textsuperscript{44}

**Critical current**

Resistivity develops in type II superconductors in the Schubnikov phase when the transport current density $J$ exceeds a critical value. This is a significant limitation in the application of superconducting technology, constraining the extent of its benefit to society. The critical current value at which superconductivity is destroyed comes about because of the interaction between the transport current travelling through the plane of the material rather than just the surface, and the vortices oriented perpendicular to the plane. This interaction is the origin of the Lorentz force, acting on the vortices in the direction perpendicular to both the transport current and the fluxoid, as shown in Equation 1.1 and Figure 1.6.

$$F_L = J_c \times \Phi_0$$  \hspace{1cm} (1.1)

where

- $F_L =$ the Lorentz force;

---

\textsuperscript{44} Increase this to indicate a reference.
1.1. Introduction

Figure 1.6: Lorentz force diagram: the vortices pointing out of the page, experience a force down the page due to the transport current $I$ travelling across the page.

- $J_c$ = the critical current and;

- $\Phi_0$ = a fluxoid or single flux quantum

Vortices are affected not only by the Lorentz force but also by repulsive vortex-vortex forces, and vortex-pinning centre interactions that restrict motion. If the pinning force is larger than the sum of the Lorentz force and the vortex-vortex interaction, then the vortex cannot move. Hence, for an unchanging pinning force, the vortices will be forced to move if either the Lorentz force or the vortex-vortex interactions become too large\(^{45}\). The Lorentz force will increase linearly with the size of the transport current, and the vortex-vortex interactions will increase as the density of vortices increases with the applied magnetic field ($B_a$). Although at high currents, some flux creep will occur due to thermomagnetic instabilities\(^{48}\). Hence, manipulation of the pinning centres to control the pinning force is essential for obtaining greater critical currents with wider ranging applications. Raising the critical current of a superconducting material increases its applicability.
1.2 Pinning centres

In an ideal, homogeneous type II superconductor without defects in the Shubnikov phase the only force felt by vortices are the vortex-vortex repulsions within the sample, so they naturally arrange themselves so that the distance between them is maximised in a triangular array as shown in figure 1.54,45,48. In the presence of weak pinning centres that resist the triangular arrangement vortices will tend to naturally distribute in the same way at high enough fields when the vortex-vortex interactions over the pinning forces of the defects.

A defect serves to pin a vortex by reducing the vortex line energy and hence reduces the total energy of the superconductor. The control of the pinning force depends on both the type of defect, and the superconductor. Superconducting precipitates act as pinning centres when they have a low transition temperature54. All superconductors contain some naturally forming pinning centres like grain boundaries and dislocations,54,14 which can be quite strong or weak. Atomic defects act as pinning centres when they are distributed inhomogeneously; if they are spread uniformly then no particular region is energetically beneficial compared to other regions within the material54. For cuprates, line and point defects, and even weakly superconducting intermediate layers in the crystal structure will act as pinning centres when placed in perpendicular and parallel fields respectively, since the coherence length $\xi(T)$ is very small. Line and point defects can also be stimulated by bombarding the sample with heavy ions or protons respectively, where straight highly altered regions were created across the crystal54. Metallic superconductors such as Niobium and cuprates like YBCO have high potential for enhancing flux pinning through the use of artificial pinning centres that can be created from either magnetic dots or antidots (holes).
1.3 Current research

Much of the research around pinning effects now focuses on artificial pinning centres like antidots and magnetic dots, which can be distributed precisely allowing researchers to test the efficacy of more than randomly distributed atomic defects. An antidot is a region of hollowed out material in a sample. In the case of superconducting thin films it refers to the removal of some thickness of the material in some shape. This can mean removing the fill thickness of the thin film in the shape of 100 nm circle or a 5 µm triangle removed to only 30% of its total thickness. A dot is created by the deposition of a microscopic amount of material.

1.3.1 Antidots

Antidots have been used to test the effect of pinning centre shape, depth, size, distribution, and combinations of these different parameters. An easy example of this is the enhanced pinning of triangular and diamond shaped 1 µm blind antidots over circular ones in YBCO thin films\textsuperscript{29}. It has been further discovered that a full antidot is capable of producing much stronger vortex pinning than a hole that has not been fully etched\textsuperscript{9}. The hole is called a blind antidot when it has not been etched through to the substrate. Determining the optimum antidot size is not a simple question to answer since the optimal pinning size is field dependent and smaller antidots are optimal at smaller fields, and bigger antidots are optimal at higher fields. Furthermore, antidots with a diameter considerably larger than the coherence length $\xi(T)$ are efficient pinning centres, and larger antidots are able to stabilise multiquanta vortices\textsuperscript{26}. Different distributions of antidots have also been tested in various array configurations like regular uniform arrays\textsuperscript{3;42}, which produce peaks at matching fields. Quasiperiodic arrays that attempt to enhance the critical current $J_c$ outside of the first matching peak\textsuperscript{24;25;22;23}. Randomly diluted arrays show success in further enhancing the critical
current outside of the matching fields, attempting to optimise the number of pinning centres by randomly removing antidots from uniform pinning arrays\textsuperscript{41,16}. Conformal arrays of pinning centres display no commensurability peaks with matching fields at all but do display significant enhancement, which is attributed to the suppression of flux channelling\textsuperscript{39,52}. Graded pinning arrays also show notable success attributed to commensurability with the vortex lattice proposed by the Bean model\textsuperscript{27} but it will be shown in chapter 3 that this is not the case.

Magnetic dots have been used in a similar fashion to antidots to test the effect of different pinning centre parameters, where the strength of the pinning centre is controlled by varying the size and thickness of the dot which regulates the number of vortices the dot can contain, since the larger potential well can hold more vortices. A magnetic dot can be grown within or on top of a superconductor and will form a ferromagnet/superconductor hybrid in both cases. A result of this hybridisation is the proximity effect, where superconducting electrons enter the other conductor (in this case a ferromagnet) through the metallic interface, reducing the transition temperature of the superconductor\textsuperscript{5}

### 1.3.2 Regular pinning arrays

Early studies on pinning arrays largely focused on simple circular antidots in uniform periodic pinning arrays etched into low temperature superconductors (LTS). These arrays were typically square or triangular arrangements of antidots, and produced clear pinning enhancements over an unaltered superconducting thin film. The benefit of uniform pinning arrays arises when the lattice period of the vortex lattice and the pinning array are equal so that the repulsive vortex-vortex interactions work with the pinning forces applied by the pinning centres rather than against them producing a significantly enhanced pinning potential\textsuperscript{3}. 
The vortex lattice period \( a_v \) is controlled by the applied magnetic field \( B_a \) and is referred to as a matching field when the periods match up. It is represented by the equation 1.2 where \( \phi_0 \) is the single flux quantum within a vortex \( 2.07 \times 10^{-15} \) Wb. As the applied field increases more vortices enter the sample and the vortices press closer together. It is noteworthy that there is more than one matching field for any periodic array, the first matching field is typically denoted \( B_1 \) and the subsequent fields are, \( B_2, B_3, \ldots, B_n \), where \( B_n = n \times \frac{\phi_0}{a_v^2} \). Figure 1.7 displays the "filling" process as an increasing magnetic field is applied to a superconducting thin film, and vortices enter from the edges of the film. Figures 1.7(a) - 1.7(d) show the "filling" process as an increasing magnetic field is applied to a square arrangement of pinning sites and figures 1.7(e) - 1.7(h) show it for a triangular arrangement. The first matching field occurs when there is one vortex per pinning centre, and interstitial positions become progressively occupied as the field increases. At the second matching field, some interstitial positions are occupied by vortices, and as the field is raised further, more interstitial positions are filled and so the pattern continues, where the increasing number of vortices results in an increasingly complex dynamic situation. The square and triangular arrays in figure 1.7 present the vortex filling arrangement when each pinning centre is sufficiently small that it can only contain one vortex. A larger pinning centre would be able to hold more than one vortex and an array of these pinning centres would show a different filling process. The number of vortices that a pinning centre can hold is defined by the saturation number \( n_s \) as shown in equation 1.3 where \( r_h \) is the pinning centre (hole) radius and \( T \) is temperature.

\[
a_v \sim \frac{2\phi_0}{\sqrt{3}B_a} \quad (1.2)
\]

\[
n_s \approx \frac{r_h}{2\xi(T)} \quad (1.3)
\]
Figure 1.7: **Square and Triangular pinning arrays:** antidots are represented by open circles and each vortex is a dot; parts (a)-(d) show the first 4 matching fields in a square array, and parts (e)-(f) show the first 4 matching fields in a triangular array\(^{42}\)

Pinning enhancements derived from periodic pinning arrays of either antidots or magnetic dots\(^{49}\) are evident as peaks in the critical current density \((J_c \propto \Delta M)^{35}\), or drops in the magnetoresistance (magnetic field dependent resistance) at matching fields\(^{19}\). Figure 1.8 shows the peaks in magnetisation at different matching fields for square arrays of antidots, and it can be seen that the magnetisation loops differ significantly with lowering temperature. As \(\xi(T)\) reduces with decreasing temperature \((T)\), \(n_s\) increases, meaning more vortices can be trapped by a single hole, and this pushes the critical point at which flux flow begins up to higher matching fields. In figure 1.8 at 6 K and 6.5 K the filling process led to doubly quantised holes by the third matching field \(H_3\). Figure 1.9 presents the critical current \((I_c)\) enhancement of a conformal pinning array (explained later)(red curve), a uniform triangular array (blue curve) and a plain film (black curve), and shows there is a clear increase for both arrays compared to the plain film.

Early theories about the difference between square and triangular arrays predicted
that triangular pinning arrays would produce better matching effects, given that it more closely matches the vortex lattice, which is also triangular. However, it was observed that there was no significant enhancement from a triangular pinning array compared to a square pinning array, which indicated the elasticity of the vortex lattice gives way to the vastly higher pinning strength provided by the matching lattice densities. A stricter regulator of the enhanced pinning at matching fields is the temperature. The enhancement of $J_c$ only occurs when the interstitial pinning potential $U_{pi}$ is small compared to the antidot pinning potential. The pinning potential refers to the depth of the potential well that pins a vortex so a high potential pins the vortex strongly. Equation 1.4 shows that pinning potential is proportional to the pinning lattice period $a_p$, and inversely proportional to the penetration depth $\lambda(T)$, if $T$ is close to $T_c$ then $\lambda(T)$ is large and $U_{pi} \rightarrow 0$; however, if this is not the case then the pinning potential is relatively high, and this severely impacts the path through which flux enters the sample. 

Figure 1.8: Magnetisation loops in Pb/Ge multilayer thin films ($T_c \approx 6.9K$), with arrays of submicron holes, figures (a), (b), (c) show the matching fields at 6.8K, 6.5K and 6K respectively. 
Figure 1.9: Experimental results of a conformal array (red) compared to a triangular array (blue) and a plain film (black)\textsuperscript{52}
1.3. Current research

\[ U_{pi} \propto \frac{a_p}{\lambda(T)} \rightarrow 0 \text{ at } T \rightarrow T_c \]  \hspace{1cm} (1.4)

- \( U_{pi} \) = interstitial pinning potential;
- \( a_p \) = antidot lattice period;
- \( \lambda(T) \) = temperature dependent London penetration depth

If \( T \) is very close to \( T_c \) then observable matching effects have shown to be significantly reduced after \( B_1 \), but as the temperature decreases away from \( T_c \) the subsequent matching potentials become observable and increase relative to \( B_1^3 \). In a high temperature superconductor (HTS) like YBCO, if the temperature is sufficiently low then the antidot pinning potential is lower than \( U_{pi} \), and the antidots actually facilitate the movement of flux within the superconductor\(^{50}\).

The vortex dynamics in a regular pinning array (RPA) are heavily dependent on the saturation number \( n_s^{26} \).

As \( \xi(T) \) decreases with \( T \) the saturation number increases, facilitating the transition from a single vortex (\( n_s = 1 \)) to a multiquanta vortex configuration (\( n_s > 1 \)) or even a superconducting network (\( n_s \gg 1 \)) if \( n_s \) is large enough. The main deciders of whether a pinning array becomes a multiquanta vortex state or not, are the ratios \( r_h \) to \( \xi(T) \) and \( r_h \) to \( a_p^{326} \), where \( a_p \) is the antidot lattice period. The matching effects seen figure 1.8 occur for an antidot lattice where \( r_h \ll a_p \), which leads to case of single vortex and even multiquanta pinning at holes and interstitial sites. If \( r_h \sim a_p \) then this can lead to the formation of superconducting networks.

A disadvantage of regular periodic pinning arrays, is that the peak enhancement may sometimes only appear at matching fields, because of the formation of interstitial vortices, and it would be useful to extend this enhancement across a range of fields.
Research has shown that other types of ordered pinning arrangements such as quasiperiodic arrays, random dilutions of periodic arrays, conformal arrays, and graded arrays can boost this enhancement by adding more matching periods and suppressing flux channelling.

Flux channelling refers to the flow of flux lines or vortices into the sample. In superconductors with low intrinsic pinning, and strongly pinning antidots as might be the case for a Nb thin film patterned with a uniform array of antidots in the vicinity of $T_c$, channels can form in between the rows of antidots that allow vortices to enter the sample easily, producing resistivity $22$. In the case of a HTS like YBCO with strong intrinsic pinning at a distant temperature from $T_c$, so that antidot pinning strength is relatively low compared to intrinsic pinning, it has been observed that lines of antidots can then serve as agent for flux entry into the superconducting thin film $50$.

1.3.3 Quasiperiodic arrays

Possible strategies for enhancing the pinning capability of periodic arrays includes attempting to increase the number of peaks in $J_c$ or broadening the peaks by simply introducing more pinning sites but this idea has some faults. Firstly, too many antidots can reduce the quality of the sample like decreasing $T_c$ or reducing the sharpness...
1.3. Current research

Figure 1.11: Different QPAs (blue curve) compared to a five-fold Penrose array (red curve): (a) square-triangle QPA, (b) square-triangle nonpisot QPA, (c) Goodman-Straus QPA, (d) Nonpisot Penrose QPA.\textsuperscript{22} Pinning centres are located at the vertices of each tiling system.
of the transition temperature when it is favourable to have a high $T_c$ and a sharp transition. Endlessly increasing the number of antidots would also create a series of high and sharp peaks\textsuperscript{22} making it difficult to precisely predict the efficiency of a superconductor at a given field and reducing the practicality of such a superconductor. Quasi-periodic arrays (QPAs) have been shown through both simulation and experimentation to improve $J_c$ up to and slightly in excess of the first matching field $B_1$,\textsuperscript{24,17} compared to random pinning arrays, and RPAs. Quasi-periodicity refers to a type of periodicity that is regularly recurring but not in a simply defined pattern compared to a regular periodic array where a pinning centre will be located every 1 $\mu$m when moving along a row of antidots for example. The theory goes that pinning arrays that are incommensurate with the vortex lattice will reduce flux channelling and enhance $J_c$ over a larger range of fields\textsuperscript{22}. It has been found that peaks in $J_c$ can be accurately predicted for a 1D quasi-periodic chain such as the Fibonacci sequence and that peaks in $J_c$ are markedly increased when the ratio of successive lengths between pinning centres is equal to the golden mean 1.618\textsuperscript{25}. More complex quasi-periodic lattices have also been investigated and have decidedly shown that a Penrose configuration (Figure 1.10) of pinning centres is the most optimal of 2D QP arrangements, as is shown in Figure 1.11. It can be seen that only the Nonpisot penrose arrangement presented in figure 1.11 d) displays a broadened and raised peak $J_c$ above that of a fivefold Penrose array, which is shown by the red curve in parts a) to d) of figure 1.11. A visual representation of a Nonpisot Penrose array is shown in the inset of figure 1.11 d). Misko \textit{et al} propose that QPAs enhance $J_c$ in the case of single vortex pinning when each vortex contains a maximum of one vortex before and minimally after $B_1$ by suppressing flux channelling, and creating a space separation between pinning centres, which prevents the hopping of vortices between them.
1.3.4 Random dilutions

In similar efforts to suppress the interstitial flux channelling in superconductors in a single vortex pinning (SVP) regime, beginning with a uniform antidot array and randomly removing some proportion of them has been shown to be beneficial. Numerical simulations on randomly diluted arrays of pinning centres in the SVP regime have shown commensurability at higher fields than the first matching field and significant critical current enhancement over uniform arrays and randomly arranged arrays over a wide range of fields even when up to 90% of pinning sites have been removed.

Investigations into randomly diluted arrays of antidots in Nb thin films where \( n_s \geq 2 \) have also shown an increase in the critical current \( I_c \). Figure 1.12 shows the onset of the flux flow voltage that occurs for different dilutions of a periodic array. At \( P_d = 0.2, 1.0 \) the fraction of removed pinning centres is 20%, and 100% respectively. It can be seen that the plain film with no pinning centres and the undiluted array show the earliest onset of flux flow, with this onset steadily decreasing as the fraction of pins removed \( P_d \) increases.

Both simulations and Nb investigations propose that randomly removing pinning sites disrupts channels of interstitial vortices that periodic arrays intrinsically endorse. Random dilutions block the motion of these interstitial vortices, and increase the depinning threshold.
1.3. Current research

Figure 1.13: Conformal transformation applied to a hexagonal lattice; taken from\textsuperscript{39}

Figure 1.12: Onset of flux flow voltage due to different dilution fractions of a triangular array of antidots in a Nb thin film\textsuperscript{16}. The inset is a magnified portion of the graph between 0 and 1.5 mA.
1.3.5 Conformal pinning arrays

Attempts to design arrays that further enhance the critical current compared to a uniform pinning array relied on conformally arranged pinning centres. The conformal arrangements of pinning centres that have been produced for study have been created by applying a conformal transformation to a semi-annular section of a hexagonal lattice. Figure 1.13\textsuperscript{39} shows that this transformation yields a series of interlaced layers of concentric rings of pinning centres when applied to a triangular array.

Numerical simulations working in the SVP regime with strong demonstrate significantly enhanced pinning over a wider range of applied magnetic field than the enhancement shown for other pinning arrangements with a comparable number of pinning sites, such as triangular, square and randomly distributed arrays\textsuperscript{39}.

Experiments in MoGe thin films reveal more clearly that the benefit of a confomal antidot array over a triangular array is most pronounced at low temperatures, and when pinning centres are strong\textsuperscript{52}. Figure 1.9 reveals the comparison between conformal and triangular arrays of fully etched antidots and blind antidots between samples II and III respectively to show that triangular array even outperforms the conformal array at matching fields because of caging effects\textsuperscript{52}. The enhanced critical current in the conformal array in figure 1.9 a) matching fields greater than $H_2$ reveals that conformal array is particularly better at pinning interstitial vortices than the triangular array.

Both investigations in simulations and real thin films agree that conformal arrays strongly pin interstitial vortices compared to uniform arrays specifically in regards to the arches of antidots that suppress interstitial vortex flux channels, but the numerical work goes further in proposing that the preservation of the sixfold ordering in the conformal array plays a crucial role, and that some matching between the flux gradient of a sample under a transport current as predicted by the Bean model\textsuperscript{4} and pinning
gradient is important\textsuperscript{39}.

1.3.6 Graded pinning arrays

With the aim of further suppressing flux channelling, and extending $J_c$ enhancement beyond $B_1$, semi-periodic pinning arrays such as graded arrays have been investigated and show promising results. A graded pinning array (GPA) refers to a periodic arrangement of pinning centres that experiences a spacing gradient along some axis. Much like the QPAs mentioned above, graded arrays exploit the incommensurability of pinning sites with the vortex lattice unlike a uniform pinning array to both reduce the size of the thermomagnetic instability (TMI) region and extend the range of critical current maximisation compared to a plain film or uniform pinning array\textsuperscript{27,23}. Figure 1.14 displays the benefits of a GPA of antidots with the spacing increasing laterally inwards towards the centre, in a MoGe thin film with a $T_c$ of 7.10 K. The graded array shown in blue clearly exhibits a higher magnetic moment than the uniform array of holes, which corresponds to a ”presumably” enhanced critical current density. Figure 1.14(b) shows the same enhanced effects but at lower temperatures where the flux avalanches associated with the TMI region are evident, and are further suppressed by the addition of a Ag cap on top of the graded film. It can be seen though that even without the Ag cap, avalanches are somewhat better suppressed by the graded array compared to the uniform array.

However, the arrays presented by Motta et al are in need of more rigorous investigation given that a comparison is made between magnetic moment loops rather than magnetisation loops of the respective films. It is also suspect that the uniform film does not exhibit any of the usual matching peaks that are normally present at these fields, and there is no explanation of this phenomenon despite an obvious up-to-date engagement with the literature at the time.
Figure 1.14: A comparison of a GPA (GRAD - blue), an RPA (UNI - red), and plain film (black)\textsuperscript{27}
Research into other spacing-graded hole densities has shown that minima in magnetoresistance curves are evident at the additional matching fields in a graded pinning array, corresponding to the varying densities of holes. This study also reported producing a measurable ratchet effect in Nb thin films from the asymmetric geometry of the array of 200 nm holes, with \( n_s \sim 1 \) where interstitial vortices were rectified in the opposite direction to pinned vortices, and a change before and after the first matching field. It is proposed that the source of the rectification difference before and after the first matching field stems from the lack of interstitial vortices that have formed before the first the matching field, and the reversed flux flow voltage originates with the interstitial vortices. The measurement of a ratchet effect indicates potential for use in superconducting electronic devices.

1.3.7 Ratchets

Ratchet technology is a recent development in vortex flow research, and substantial investigations have already been made. A ratchet is any device which allows a current of any type to flow with less resistance in one direction than the other. Investigations into numerous types of ratchets have been conducted on bimolecular motors colloidal suspensions, molecules, electrons and vortices in superconductors. Studies in superconductors are concerned with how to best obtain rectified vortex flow and hence, a unidirectional current given an AC source. A rectified vortex flow comes about from a superconducting ratchet system, where vortices will preferentially move in the ‘easy’ direction under the influence of an AC current, meaning that onset of flux flow resistance is delayed in the ‘hard’ direction when the vortices flow against the structure of the ratchet.

Numerical simulations into SVP regimes has been undertaken and compares the
1.3. Current research

Figure 1.15: Arrays used in simulations to test the influence of a) a conformal array, b) a graded random array, c) a graded square array on the ratchet effect.

The ratchet effect in three differently graded arrays\(^4\). It compares a conformal array, a graded square array, and a graded random array as shown in figure 1.15. It is observed that the conformal pinning array generally produces the most significant ratchet effect especially above the first matching field \(B_{c1}\). The wide distribution of pinning sites in low density regions perpendicular to the gradient allows interstitial vortices to flow easily in the gradient direction. The square array presents no difference in the direction perpendicular to the gradient so that a small ratchet effect is only observed when interstitial vortices are present, and the graded random pinning array presents no difference in any direction to vortex motion, so no ratchet effect is observed.

Investigations into rectified vortex motion from the influence of collective ratchet pinning potentials have been conducting using arrays of \(\sim 1\mu m\) blind triangular an-
1.3. Current research

tidots in YBCO thin films\textsuperscript{29,43}. According to table 1.1 the size of $\xi$ in YBCO is approximately 1.5 nm, so a hole on the order of 1 $\mu$m leaves plenty of space for multiple vortices and presents a novel pinning arrangement. Both positive and negative rectification systems have been observed in these arrays to show that in positive systems ratchet effects are determined via the interactions between vortices and the asymmetric patterns i.e. pinned vortices, and in negative rectification systems, the ratchet effects are controlled by the strong collective interactions of pinned vortices and interstitial vortices.
Testing the effects of different antidot patterns on YBCO and Nb thin films is a lengthy process beginning with deposition of the film, measurement of its superconducting properties using a SQUID magnetometer, designing a pattern and applying it, etching the pattern, and making the final measurement to observe the effects produced.

2.1 Physical Vapour Deposition

Physical vapour deposition (PVD) is an umbrella term used to describe several types of deposition that all rely on the target material entering the vapour phase and being directed towards the substrate to form a film or coating\textsuperscript{20}. In this work Nb and YBCO thin films have been produced by dc magnetron sputtering and, pulsed laser deposition (PLD) respectively.

2.1.1 Magnetron Sputtering

Sputtering is a type of physical vapour deposition where atoms are sputtered or ejected from a target surface, entering a vapour phase and adhering to any surrounding surfaces. Physical sputtering is one of these types, where an inert gas is ionised, and then
2.1. Physical Vapour Deposition

forced to bombard the target surface, liberating the surface layers. DC magnetron sputtering is just one type of physical sputtering that creates a very localised plasma around the target, accelerating charges towards it and releasing atoms from the target.

In this work a DC magnetron sputtering system from Mantis Deposition Systems using a planar magnetron configuration has been utilised to deposit Niobium thin films as shown in figure 2.2 where the target material sits on the cusp connected to a high voltage source, and target material is accelerated towards the substrate that sits at the top of the chamber above the cusps. The planar configuration creates a magnetic field as shown in Figure 2.1, and produces an indented ring over time in the target surface by bombarding it with Ar$^+$ ions, shown as the two dips just outside the middle of the Nb target in the sputtering schematic. The Ar$^+$ ions liberate clusters of Nb atoms from the target, and direct them towards the substrate, where epitaxial growth can be achieved under the right conditions.
2.1. Physical Vapour Deposition

Figure 2.2: Schematic of DC magnetron sputtering provided by Mantis Deposition Systems

2.1.2 Pulsed Laser Deposition

Pulsed laser deposition also known as flash evaporation or laser ablation deposition, is a type of PVD where high energy laser pulses are fired at a target, vaporising the material, causing it to eject in a highly directed plume towards the substrate\(^{20}\). A number of parameters such as the laser energy, laser fluence, and pulse frequency among other things control the type of growth, and growth rate of the ejected material. When properly configured, epitaxially grown, high quality YBCO thin films can be deposited. The quality of YBCO is highly dependent on all of the individual deposition parameters, and the YBCO structure can be tuned by the pulse frequency\(^{10}\).

In this work, PLD was used to deposit YBCO thin films on $5 \times 5$ mm MgO substrates\(^ {10,34}\). A KrF excimer laser was used with a wavelength of 248 nm, and fluence was maintained at $\sim 3 - 4$ J/cm\(^2\), but had a realistic fluence of 2 J/cm\(^2\). The frequency varied between 1 and 8 Hz, and the YBCO target and the substrate were separated by 85 mm. The pre/post-deposition pressures were $10^{-6}$/0.3 Torr.
2.2 Magnetisation measurements

The superconducting properties of YBCO and Nb thin films in this work are evaluated by their supercurrents, measured using a superconducting quantum interference device (SQUID) that detects the magnetic fields associated with them. A SQUID is the most sensitive device available for measuring magnetic fields and is utilised by the Magnetic Property Measurement System (MPMS) from Quantum Design, to detect minute magnetic fields in many types of materials, including superconducting thin films. The system consists of a superconducting magnetic shield, a superconducting detection coil, and a superconducting magnet. The detection coil is a single wire wound in second derivative array where the top and bottom coils are wound in a counter clockwise direction and the two centre coils are wound in a clockwise direction, as shown in figure 2.3. The coil is wound this way to reduce noise from the magnet, and to minimise background drift from any relaxation of the field. The coil is inductively coupled to the SQUID and acts as a highly linear, current to voltage converter that is

Figure 2.3: Superconducting pick up coil, taken from the MPMS instrumentation booklet.\textsuperscript{21}

respectively, and the substrate temperature was 780°C.\textsuperscript{10,34}
proportional to the magnetic moment produced by the sample. This set-up is highly sensitive and samples produce magnetic flux in the system as small as one thousandth the order of a single flux quantum, so it is very reactive to any external fields meaning it has to be shielded. The magnetic field supplied by the system comes from a closed superconducting coil wound in a solenoidal fashion, allowing constant magnetic fields to be maintained by persistent supercurrents. The magnetic field is changed by heating a superconducting switch, so that it enters the normal phase, a power source alters the current, and the coil is re-cooled\textsuperscript{21}. The system is capable of cycling the temperature from 2 to 400 K, and the magnet control system ranges from -5 to 5 T. Utilising this system superconducting thin films can be easily characterised to determine their $T_c$ and magnetic hystereses at different temperatures.

### 2.3 Pattern application

The pattern application process requires the etching of a design into the superconducting material. In this work Nb and YBCO thin films have been dry etched using
ion milling, a mechanical process where Ar$^+$ ions are launched at the films attached to a rotating table, that is maintained perpendicular to the beam during the etching process. It operates under a similar principle to sputtering, where it relies on the bombardment effect to remove material.

To apply patterns to these films, arrays of triangular antidots were created using AutoCAD, and were then was applied using a Heidelberg $\mu$pg101 Micro Pattern Generator, which uses a UV laser to activate areas of a positive tone photoresist applied by spin coating, that will dissolve when developed, leaving behind a mask$^{28}$. Figure 2.5 displays the spin coater a) and lithography equipment b) used in this work. The open spin coater shows a central stand rising from the middle with a small black dot in the middle. The back of the sample is centred on the black dot and photoresist is applied. After spinning it is centred on the stage in the Heidelberg $\mu$pg101 Micro Pattern Generator where a design is loaded into the system from the computer, and a UV laser inside will apply the design to the photoresist coated sample. After developing the film will be etched using ion beam milling. A process where an inert gas such as Argon is injected into a vacuum chamber, and a plasma is then initiated by an RFM100 ion gun from the gas, creating Ar$^+$ ions. These ions are then launched towards the film by a pair of accelerating grids with a strong electric field between them, forming a collimated ion beam 5 inches in diameter in the case of this work. The etching properties of the ion beam are controlled by a number of plasma parameters. For etching YBCO and Nb thin films, the optimal plasma and chamber conditions for this Mantis system have been determined through repeated iterations, altering one parameter at time. These parameters are presented in table 2.1. Using these parameters, a 250 nm YBCO film will be completely etched in just 40 minutes, with minimal film damage.

While the simplicity of this process provides a huge advantage, the picture described above is the ideal case, and it still suffers from some disadvantages such as
2.3. Pattern application

Figure 2.5: a) Spin coater b) Heidelberg µpg101 Micro Pattern Generator

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base pressure</td>
<td>$\leq 10^{-6}$ mbar</td>
</tr>
<tr>
<td>Argon pressure</td>
<td>$\sim 3 \times 10^{-3}$</td>
</tr>
<tr>
<td>Plasma power</td>
<td>$\sim 80$ W</td>
</tr>
<tr>
<td>Accelerating voltage</td>
<td>300 V</td>
</tr>
<tr>
<td>Ion current</td>
<td>35 mA</td>
</tr>
</tbody>
</table>

Table 2.1: Etching parameters

...poorly defined feature edges like the rounded corners in figure 2.6 b) that may be caused by poor resolution, underexposure, extra scattering, edge diffraction, and improper lift off where not all photoresist is removed as desired during the development process$^{18}$. Part a) of Figure 2.6 displays an array of supposedly triangular antidots arranged in rings that become further spaced apart as they move inwards, showing that the overall design comes out mostly as expected, but part b) is an SEM image taken using a JEOL SEM, and shows that corners of the holes are rounded, which does not correspond to the original design, and is largely a feature of the 1 µm resolution limit of the optical laser.
2.3. Pattern application

Figure 2.6: Antidot with rounded edges as seen by an optical microscope a) and SEM b) in a YBCO thin film.
Chapter 3

Novel pinning arrays in YBCO thin films

3.1 Introduction

A series of artificial pinning arrays etched into YBCO thin films was investigated in this work. Previous investigations of regular artificial pinning arrays have returned peaks in $J_c$ at certain low matching fields in low temperature superconductors as described in chapter 1. Some success has been made in extending this enhancement across a wider range of applied magnetic fields, by quasi-periodic, conformal, and graded arrays but is still limited to small fields.

3.1.1 The Bean model

The Bean critical state model describes flux entry into a type II superconductor in the Shubnikov phase. The flux profile for a plain YBCO thin film is presented in figure 3.1. Beginning with a zero field cooled sample, the applied magnetic field is ramped up to 5 T, down to - 5 T, and if cycled continuously will follow the hysteresis in part
Figure 3.1: Schematic distribution of magnetic flux according to the critical state model according to Bean as the applied magnetic field \( (B_a) \) cycles from 5 T to -5 T in a zero-field cooled sample. Part a) magnetisation hysteresis, part b) cross sectional view of YBCO thin film of width \( w \), part c) flux front within the sample for increasing and decreasing field. Adapted from \(^{48,54}\).
3.1. Introduction

a), where different stages of the loop are identified by the applied magnetic field stage \((B_{a1} - B_{a10})\). Part a) is a complete magnetic hysteresis loop of a plain YBCO thin film in perpendicular magnetic field measured using a Quantum Design MPMS. Part b) reveals the cross section for a typical, plain YBCO thin film of width \(w\) used in this work. Part c) reveals the spatial distribution of the internal field and the applied magnetic field \((B_a)\) at each stage. Stage \(B_{a1}\) is the first point reached after first ramping up the field from the zero field cooled state. The field at the edges of the sample is equivalent to the applied field, but no vortices have penetrated the centre yet because of intrinsic pinning. As the field progressively increases, vortices penetrate inwards overcoming pinning effects and geometric barriers. At 10 K, \(B_{c2}\) can exceed 100 T for a YBCO thin film, which is too high for the MPMS so that is not shown.

As the applied field decreases from 5 T, vortices then leave the sample, leaving the internal field higher at the centre of the film than the edges because some vortices remain pinned in the middle as shown in stages \(B_{a1} - B_{a8}\). Decreasing from 0 T to -5 T \((B_{a9}, B_{a10})\) anti-vortices penetrate the sample from the edges, so the field at the edges now has a higher magnitude than the middle again. An anti-vortex is a vortex with opposite polarity to the vortex that originally entered the sample. The magnetic flux quantum inside is ‘upside down’ relative to the original vortex and the surrounding supercurrent flows in the opposite direction. When a vortex and anti-vortex meet they may annihilate bringing the internal field to zero at that location.

Ramping the applied magnetic field back up to 0 T anti-vortices begin to exit from the edges with the most again remaining pinned at the centre. The tendency of the flux front to lag behind changes in the applied field because of intrinsic pinning means that vortices are not distributed uniformly within the sample and will be most dense at the edges of the film on the positive ascending field branch, and most dense at the centre on the positive descending field branch.
3.1.2 Non-uniform pinning arrays

The 3 µm triangular antidots in these films utilise a type of edge pinning where the vortices get pinned along the wall of the antidot as has been demonstrated experimentally in arrays of large antidots in YBCO thin films\textsuperscript{30}. The triangular antidots in these films are significantly larger than ξ and λ, hence collective pinning effects are controlling the significant enhancement of almost all non-uniform arrays in these films. These antidots enable high tunability of $J_c$.

The benefit of graded pinning arrays comes from the broadening of $J_c$ peaks at matching fields corresponding to commensurability between the vortex lattice and the pinning array. This broadening effect has also been demonstrated\textsuperscript{25} by introducing quasi-periodic arrays with multiple commensurate matching states or even combining multiple matching states with graded arrays to force non-Bean like flux penetration through hyperbolic tessellating pinning arrays\textsuperscript{23}.

A linearly graded array was experimentally shown to be quite effective at enhancing pinning MoGe thin films by Motta \textit{et al}\textsuperscript{27}, however, the somewhat controversial explanation given assumed the strong dependence on the Bean-like flux distribution in the superconducting films.

The graded arrays in this thesis utilise much larger antidots in YBCO films with much smaller vortices than any other attempts to further enhance the matching fields produced by uniform arrays.

It is noteworthy that the uniform arrays in these YBCO films produced no pinning enhancement while the uniform arrays in single vortex pinning regimes do enhance $J_c$ at matching fields. The graded pinning arrays in this work do produce significant enhancement across a wide range of fields, indicating commensurability with the vortex lattice may be applied in much broader terms than just with respect to its lattice parameter.
3.2 Pinning enhancement

To examine the effects of arrays of large antidots, a number of YBCO thin films were etched with different antidot arrays by ion beam etching after the array pattern was applied using laser lithography of positive tone photoresist, as outlined in detail in chapter 2. The lithography process was optimised for this Mantis system, through numerous iterations controlling for one parameter at a time to find the ideal combination. Table 2.1 presents the optimal etching parameters, which will allow a 250 nm YBCO thin film to be fully etched in 40 minutes with no damage to the protected regions of the film.

3.2 Pinning enhancement

A summary of the array designs is presented in table 3.1. Samples YBCO11 to YBCO32 represent different arrangements of $3 \, \mu m$ triangular antidots in arrays, measuring a side length of 3 mm. This size was chosen to balance workability, and maximal signal strength. A uniform array in this work means a square array with a lattice period of $10 \, \mu m$, and the term graded array means a series of rings of triangular antidots where the spacing between each ring progressively increases by a factor of 1.618 working inwards, as shown in figure 3.3 b). The CAD drawing to lithographically apply this pattern is shown in figure 3.2. The rings are most dense at the edges of the thin film. The antidot parameters remain the same with a side length of $3 \, \mu m$ and a period of $10 \, \mu m$ along each ring. The initial ring spacing is $7 \, \mu m$ and the final spacing is $532 \, \mu m$, where the ratio of the $i^{th} + 1$ and $i^{th}$ spacings is the golden ratio 1.618. Samples YBCO20, and YBCO21, YBCO24, YBCO31, YBCO32 are described with an array type of 'inverted gradient' and 'spaced'. The inverted gradient still has a ring spacing that increases by a factor of 1.618 but working outwards this time, so that the rings are most dense in the centre as shown in figure 3.3 c), and all other parameters are the same as the graded array. The evenly spaced array is a regular but non-uniform array
Table 3.1: Summary of YBCO lithography patterns

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Antidot shape</th>
<th>Array type</th>
<th>Antidot direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>YBCO4</td>
<td>3 ( \mu m ) circles</td>
<td>2 mm uniform</td>
<td></td>
</tr>
<tr>
<td>YBCO6</td>
<td>3 ( \mu m ) circles</td>
<td>1 mm uniform</td>
<td></td>
</tr>
<tr>
<td>YBCO7</td>
<td>3 ( \mu m ) triangles</td>
<td>2 mm uniform</td>
<td>same</td>
</tr>
<tr>
<td>YBCO8</td>
<td>3 ( \mu m ) circles</td>
<td>3 mm graded</td>
<td></td>
</tr>
<tr>
<td>YBCO9</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm graded</td>
<td>same</td>
</tr>
<tr>
<td>YBCO11</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm uniform</td>
<td>same</td>
</tr>
<tr>
<td>YBCO12</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm graded</td>
<td>same</td>
</tr>
<tr>
<td>YBCO13</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm uniform</td>
<td>inwards</td>
</tr>
<tr>
<td>YBCO14</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm uniform</td>
<td>outwards</td>
</tr>
<tr>
<td>YBCO15</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm graded</td>
<td>inwards</td>
</tr>
<tr>
<td>YBCO16</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm graded</td>
<td>outwards</td>
</tr>
<tr>
<td>YBCO20</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm inverted gradient</td>
<td>same</td>
</tr>
<tr>
<td>YBCO21</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm spaced 140( \mu m )</td>
<td>same</td>
</tr>
<tr>
<td>YBCO24</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm spaced 80( \mu m )</td>
<td>same</td>
</tr>
<tr>
<td>YBCO31</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm spaced 20( \mu m )</td>
<td>same</td>
</tr>
<tr>
<td>YBCO32</td>
<td>3 ( \mu m ) triangles</td>
<td>3 mm spaced 40( \mu m )</td>
<td>same</td>
</tr>
</tbody>
</table>

Presented in figure 3.3 d), a series of equally spaced rings 140 \( \mu m \) apart, with the same parameters along each ring as the other samples. The samples YBCO31, YBCO32, and YBCO24 have rings that are spaced 20, 40, and 80 \( \mu m \) apart respectively.

Early attempts at determining the optimal pinning array were first concerned with the ideal antidot shape at this scale. This occurred alongside the investigation of ideal etching parameters so some of the results were clouded by these competing influences, but it was possible to conclude the 3\( \mu m \) triangles in a 3 mm array outperformed all other types. The 3 mm array struck a good balance between convenient array size and an acceptable signal/noise ratio.

Figures 3.4, 3.5, and 3.6 present the difference in \( J_c \) between post and pre lithography, at 4.2 K, 10 K, and 77 K respectively between 0 and 5 T. A \( J_c \) difference value > 0 indicates that \( J_c \) improved after having a pattern applied, and a value < 0 indicates that it was reduced. The abbreviations in the legend describe the array
3.2. Pinning enhancement

Figure 3.2: CAD drawing of a graded array of 3 μm triangles all pointing ‘upwards’. The inset in the centre of the design is a zoomed in portion of the top left corner of the array showing the triangular holes that make up the rings.
3.2. Pinning enhancement

Figure 3.3: SEM images of YBCO thin films YBCO11 a), YBCO12 b), YBCO20 c), YBCO21 d) after etching. d) is at a lower magnification to better observe the pattern.

![SEM images](image)

Figure 3.4: $J_c$ difference for pre and post lithography for samples YBCO11 - YBCO32. The legend describes the features of the array in the form array type/antidot direction. The measurements are taken between 0 and 5 T at 4.2 K.

![Graph](image)
3.2. Pinning enhancement

Figure 3.5: $J_c$ difference for pre and post lithography for samples YBCO11 - YBCO32 at 10 K.

Figure 3.6: $J_c$ difference for pre and post lithography for samples YBCO11 - YBCO32 at 77 K.
3.2. **Pinning enhancement**

Pinning enhancement type/antidot direction. The plots are presented in such a way as to make it obvious, which array types out perform which, how they perform across a range of fields, and how performance changes with temperature.

In these plots, the most significant feature is the clear difference between uniform and non-uniform arrays. The three uniform arrays consistently presented a $J_c$ difference $< 0$, while almost all of the non-uniform arrays presented a $J_c$ difference $> 0$ indicating that uniform and non-uniform arrays respectively worsened and improved the pinning mechanisms inside the film. The only difference between the three uniform arrays is the direction the triangular antidots point. In YBCO11 they all point the same direction (‘upwards’), in YBCO13 and YBCO14 they all point inwards and outwards respectively. This is to examine the influence of a ratchet effect in such an array and will be discussed later. It is very promising that the majority of these non-uniform arrays enhanced $J_c$ across all fields, since previous attempts at improving on the previously obtained matching fields such as quasi-periodic arrays, conformal arrays, and randomly diluted arrays have only produced enhancements up to a few multiples of the original matching field. The pinning arrays in this work present an enhanced $J_c$ from 0 to 5 T. Matching fields are not expected for any of the arrays in this work, since they are only observed when the saturation number $n_s \sim 1$ as described in chapter 1. The antidot pinning arrays in YBCO thin films where $\xi_{ab} = 1.5 \text{ nm}$, and the hole size is $3 \mu m$ gives $n_s > 200$, a completely different regime to that frequently examined in these types of arrays. If matching fields were to be present $B_1 \approx 0.2 \text{ Oe}$, but none could be observed at these low fields even with the high resolution of the MPMS.

It can be noted in figures 3.4 - 3.6 that most results for the non-uniform arrays are clustered in a fairly narrow range. These differences are examined more closely in figure 3.7, which presents the self field ratios for samples YBCO11-YBCO32 of
3.2. Pinning enhancement

post/pre lithography, so a value < 1 indicates that lithography was ineffective and a value > 1 indicates that pinning was enhanced within the sample. Figure 3.7 presents a comparison between the films in the low field region where the bulk of the research into antidot pinning arrays has been conducted and shows again that almost all uniform and non-uniform films respectively worsened and improved vortex pinning. The strengthened pinning forces can possibly be explained by the same reasoning applied to quasi-periodic arrays\(^2\), firstly, where the arrays in this work prevent flux channelling at high fields, because of the lack of alignment between antidots in the densest regions of the graded and inverted arrays. The lack of alignment means flux channels cannot stretch from the edge of the film to the centre without an interrupted path. The second benefit of quasi-periodic arrays comes from the suppression of ‘vortex hopping’, but it is not clear whether either of these effects are taking place in these YBCO thin films. In previous investigations that propose these mechanisms of enhanced pinning, simulations and experiments in real films take place in films with no or low intrinsic pinning. In YBCO films that naturally have relatively strong intrinsic pinning these effects of flux channel suppression and vortex ‘hopping’ restriction might be a native feature of the film rather than being an effect of the array.

In figures 3.4 - 3.7 it can be seen that the only non-uniform arrays that perform negatively are the regularly spaced but non-uniform arrays with ring spacings of 20 \(\mu m\) and 40 \(\mu m\), but once doubled to 80 \(\mu m\) quickly jumps to have a small positive enhancement of \(J_c\), equal in magnitude to the thin film etched with rings of antidots spaced 140 \(\mu m\) apart. This effect supports the idea of multiple pinning effects stemming from these arrays of antidots. These evenly spaced films would likely share the advantage of preventing flux hopping between antidots with graded arrays because of the large spacing between them working inwards, but they lose the lack of alignment between antidots, meaning that the rings of antidots create an environment in which
3.2. Pinning enhancement

Figure 3.7: Self Field $J_c$ ratio of post/pre lithography
3.2. Pinning enhancement

flux channels can easily form, especially in the closely spaced rings of 20 and 40 µm. This could explain the smaller enhancement of $J_c$ than the other non-uniform pinning arrays. Contrary to the rest of the samples, the spaced arrays all show a further decrease in $J_c$ at 77 K, when the relative pinning strength of the antidots to intrinsic pinning centres is higher.

Looking closely at figure 3.7 it can be seen by comparing the differences in the self-field $J_c$ ratio that the graded array with all triangles pointing ‘up’ performs the worst of all the non-uniform arrays at low temperatures but only by about 1-2%. The most efficient pinning mechanism is achieved with a graded array where all antidots point inwards, towards the centre, by just under 10% at low temperatures and almost 20% at 85 K. There is also a small difference between the uniform arrays with inward and outward pointing triangular antidots, but the differences are actually opposing in this case between high and low temperatures. It is possible that the 85 K measurement for the uniform array with inward pointing triangles is not accurate because of a poor signal to noise ratio.

It can be seen that the graded arrays enhance the critical current across the entire range for temperature and applied magnetic field. At low fields the individual or small number of vortices are pinned along the antidot boundaries and in interstitial regions. Given this combined pinning effect it is possible that a non-Bean like flux entry profile might be taking place. Rather than distributing according to the Bean model it is possible that vortices become jammed behind the first row of antidots in the graded array and as the field increases vortices gather behind the dots until they flood past and gather along the second row of antidots, and onto the third and so on as the field increases, creating a ‘vortex vacuum’ behind each row. It is further possible that any type of non-uniformity in these conditions would disallow the organisation of a Bean
like flux gradient, which would likely be controlled by the natural pinning mechanisms. This is somewhat similar reasoning to that used to explain quasi-periodic arrays and graded arrays with antidot size $\sim 2\xi(T)^{25,23,27,3,17,52}$ and might be a more fitting explanation than the suppression of flux channelling. It is possible that this vortex vacuum effect is just a different way of looking at the suppression of flux channelling.

This scenario explains the $J_c$ reduction in evenly spaced arrays with small spacings (20 $\mu$m and 40 $\mu$m). The smaller the spacing the more likely it would be to enter the Bean like vortex distribution. The larger spacings lean towards a non-Bean like flux gradient and explains the enhancement of the larger evenly spaced arrays at fields $> 0.8$ T.

The $J_c(B_a)$ dependences in this work were extracted from measured magnetisation loops where vortices enter and exit the film. The above scenario describes vortex entry so vortex exit would invert the scenario where vortices would gather and form ‘vacuum’ regions on the front face of the antidots. This explains the similar success of the inverted gradient array and comparative performance to the graded array. This scenario was not considered by Motta et al and although that work focuses on MoGe films with holes comparable to $\xi(T)$ it is possible this same scenario applies to their films in a very narrow field range as only a few vortices cannot participate in that case. Regardless of whether it does apply the success of graded arrays in MoGe thin films is not quite adequately explained by commensurability of the pinning lattice and the Bean profile proposed by Motta et al, which is completely opposed on the positive descending branch of the magnetisation measurements.

### 3.3 Ratchet effects

Based on the work of other collective vortex pinning systems$^{29,43}$, which demonstrate reversible rectification effects for micron sized triangular blind antidots in YBCO thin
Figure 3.8: Scaled moment difference between ascending and descending field branches at 10 K and 77 K.
films that stem from the anisotropic influence of the triangular shape of the antidot, it was assumed that a similar feature might be observable in this work.

Figure 3.8 allows us to examine the arrays for signs of any ratchet effects. The 'scaled moment difference' is the difference in the absolute value of the magnetic moment of ascending and descending field branches, which represent entering and exiting vortices respectively. A value $< 0$ indicates the ascending branch had a larger magnitude and vortex motion was resisted more at entry than exit. The 10 K results show a positive scaled moment difference for all arrays after $\sim 1$ T, indicating that vortices collectively have an easier time leaving than entering. Below 1 T, there seems to be multiple influences but it is clear that the inverted array makes it significantly easier for vortices to leave than the other arrays. The 77 K results are lacking complete clarity, even after some 'smoothing' but it can be seen that the inverted and graded arrays have slightly opposing effects, where the graded array displays marginally stronger pinning effects upon exit rather than entry, and vice versa for the inverted array. Below $10^{-1}$ T the results are chaotic, and possibly indicate the competition of multiple pinning mechanisms like bulk pinning effects, nano-wall pinning along the edges of the antidots, and geometrical barrier effects from the sharp edges of the film, which have been shown to produce resistance to flux departure.$^{31}$

It was proposed that additional ratchet effects might be stimulated by etching the triangular antidots to face different directions. Both the uniform and graded arrays were patterned with different orientations of antidots, all holes pointing inwards and all holes pointing outwards, so the effects could be compared for different arrays. All magnetisation measurements in this work start at 0 T, and the applied magnetic field is ramped up to 5 T. Vortices press inwards up to 5 T and try to exit again as the applied field decreases, with vortex motion being resisted by pinning effects as shown in figure 3.1. The resistance to vortex motion is indicated by the tendency of the film
3.3. Ratchet effects

magnetisation to lag behind the applied field as proposed by the Bean model\(^4\). The triangular fully etched holes present a barrier to vortices, where some will get pinned along the walls of the antidots thanks to nanowall pinning\(^30\). When filled, the walls of these antidots will then repel the approach of other vortices, impacting their motion. Pressing inwards or outwards, the motion of vortices should feel more impedance when they encounter the flat face of the triangle rather than the apex, since it will produce more resistance, just the like the nose of a jet is pointed to reduce air resistance. This work hoped to exploit antidot orientation dependent vortex motion, but figures 3.4 to 3.8 all show minimal performance differences based on antidot direction, compared to the array type. Figure 3.7 actually shows a large difference especially at high temperatures between inward and outward orientations of antidots for the graded arrays, but this result is suspicious, firstly because this difference is practically non-existent for the uniform arrays, and secondly because the effect disappears looking at the scaled moment difference in figure 3.8.

Another observed effect of patterning was a temperature dependent positive shifting of the central magnetisation peak, displaying no shift at 77 K, but significant lateral shifting up to \(9 \times 10^{-2}\) T at 4.2 K. Figure 3.9 is an excerpt from a granularity investigation\(^46\), which showed that demagnetisation will affect inter-granular currents, shifting the central peak. The figure is presented to illustrate the concept central peak shift. This indicates that the films in this work also experience some demagnetisation effects, perhaps from inter-antidot currents in this situation. The shifting may also just indicate the presence of modified currents within the film as a result of the implemented patterns.
Figure 3.9: Figure 5 taken from Shantsev et al.⁴⁶ presenting the temperature dependent shift of the central peak of a magnetic hysteresis loop in a YBCO thin film.
Chapter 4

Optimisation of Nb thin films

As stated previously Nb thin films were deposited in this work via dc magnetron sputtering, however, the process is being newly established, so was originally far from optimal, and the system required much adjustment and modification to successfully deposit films. The important task before novel research could be undertaken was to optimise the sputtering process for the new Mantis deposition system used in this work, which required finding the optimum configuration of the different controllable parameters. Deposition parameters can be highly interdependent, and renders the simple technique of simply manipulating one parameter at a time as heavily time consuming, since a number of configurations must be examined for their effect on film quality. It has been observed in other work that for ZnO thin film growth that the optimum combination of substrate temperature and deposition rate was in the high range for both, and combinations of low/high or high/low for the deposition rate and substrate temperatures respectively, caused a mixed orientation of the c-axis in sputtered films. This is just one example of such interdependency and can be extended to other parameters.

To assess the impact of different parameter configurations, film quality was assessed at a basic level by measurement of $T_c$, the transition width $\Delta T$, and $B_{c2}$ using a Quan-
Figure 4.1: Scaled moment plot to find $T_c$ and $\Delta T$

tum Design MPMS, as has been used in other works$^{6;8;15;38}$. For practical purposes it is desirable to have the highest $T_c$ possible, the sharpest temperature transition so the smallest transition width $\Delta T$, and the highest $B_{c2}$ since these all maximise the size of the environmental conditions in which the superconductor can be used. The transition width is defined in multiple works$^{6;15}$ by the temperature difference between 90% and 10% of its signal strength for either magnetisation or transport measurements. In magnetisation measurements made using the MPMS the lower temperature will correspond to 90% of the signal strength and 10% will be the higher temperature close to $T_c$ as shown in figure 4.1 where the dashed horizontal lines correspond to 90% and 10% of the signal strength and the intersecting vertical dashed lines reveal the temperatures at these points.

The sputtering parameters listed immediately below reveal the main values that need to be controlled for to tune the superconducting properties of Nb thin films to
optimise their quality.

**Sputtering parameters:**

- Substrate temperature
- Film thickness
- Preparation time
  - Baking time
  - Time at deposition temperature
- Deposition rate
- Deposition time
- Base pressure
- Deposition argon pressure

Starting from recommendations suggested by Mantis, some consensus has been obtained through repeated depositions but there is a significant problem with interpreting some of these results because they are clouded by the instability and malfunctioning of the system that was contaminated by the oxidising material that coats the walls of the main chamber from previous depositions, every time something had to be repaired. The system is ideally supposed to be maintained at an ultra high vacuum (UHV) of \( \leq 9 \times 10^{-8} \) mbar in a relatively unbroken state.

Tables 4.1 and 4.2 present the main deposition parameters that need to be controlled for, to adequately deposit Nb thin films via DC magnetron sputtering. The table headings are arranged in order of sample number; deposition temperature; thin film thickness; preparation time (the time spent baking the chamber beforehand to clean it, this happens before every deposition); base pressure (the chamber pressure
Table 4.1: Some deposition parameters. The blank areas were not recorded at the time.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Dep. temp.</th>
<th>Thick.</th>
<th>Prep time</th>
<th>P&lt;sub&gt;base&lt;/sub&gt;</th>
<th>P&lt;sub&gt;50°C&lt;/sub&gt;</th>
<th>P&lt;sub&gt;dep&lt;/sub&gt;</th>
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<tbody>
<tr>
<td>Units</td>
<td>ºC</td>
<td>nm</td>
<td>hours</td>
<td>mbar</td>
<td>mbar</td>
<td>mbar</td>
</tr>
<tr>
<td>Nb01</td>
<td>200</td>
<td>50</td>
<td>12+</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb07</td>
<td>400</td>
<td>300</td>
<td>20</td>
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<td>7.4 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
<td></td>
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<td>600</td>
<td>300</td>
<td>4</td>
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<td>1.4 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
<td></td>
</tr>
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<td>4.3 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
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<td>3.5 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
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<td>8.4 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
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<tr>
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<td>27.5</td>
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<td>7.9 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
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<tr>
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<td>27</td>
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<td>7.0 × 10&lt;sup&gt;-8&lt;/sup&gt;</td>
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<td>1.6 × 10&lt;sup&gt;-7&lt;/sup&gt;</td>
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</tbody>
</table>

Table 4.2: Remaining deposition parameters, T<sub>c</sub>, and ∆T. The blank areas were not recorded. sccm stands for standard cubic centimetres/minute.
Figure 4.2: Temperature dependence curves for valid Nb samples. Applied magnetic field is $2.5 \times 10^{-3} \text{T}$.

Before the sample is even loaded inside via the loadlock is a key indicator of cleanliness; chamber pressure after it has been baking at 50°C to clean the chamber by exciting oxidised materials off the walls of the chamber to be picked up by the turbopump for the majority of the preparation time; chamber pressure once the argon flow has been optimised; the deposition rate of Nb from the sputter target onto the substrate, which is measured using a quartz crystal microbalance that detects the mass of deposited material on a quartz crystal resonator by measuring the resultant change in resonance frequency dependent on the mass of material$^{13}$; argon flow; the plasma parameters of power, voltage, and current that control the plasma energy and particle energies involved; and finally the sample characteristics of superconducting transition temperature, and transition width that are used to judge the quality of the sample.

Figures 4.2 and 4.3 present the temperature transition curves and transition width for the listed samples, where a high transition temperature and a narrow transition
width are the most desirable. These figures reveal that samples Nb01, Nb07, Nb08, Nb09, Nb17a, and Nb17b, have the highest $T_c$ and smallest $\Delta T$ of all thin films deposited, and are of sufficient quality to be used for research where accurate inferences could be made on the impact of non-uniform antidot arrays. Figure 4.2 reveals the clear difference between films Nb01, Nb07, Nb08, Nb09, Nb17a, Nb17b where it can be seen that the curves for these samples are clustered up the far end of the graphs in the higher temperature region, also clearly displaying sharp temperature transitions compared to the other samples.

The high quality of thin films Nb01, Nb07, Nb08, and Nb09, can be attributed to the 'cleanliness' and deoxygenation of the deposition chamber prior to and during deposition. The chamber is cleaned/prepared for deposition by baking the chamber at 50°C for 12-24 hours depending on required cleanliness of the chamber prior to deposition, after the substrate has been loaded inside. After this baking period, the chamber is then heated to the deposition temperature, and left at this temperature for 2-6 hours to let the pressure return to its value prior to the latest temperature increase. The plasma is then ignited and maintained to deposit between 5 and 500 nm of target material. The preparation process is a critical part of the whole deposition procedure, since Niobium thin films are particularly sensitive to contaminants or oxidation, and a high quality thin film requires an exceedingly high standard of cleanliness. This dependence on cleanliness was revealed experimentally in this work from the inability to produce Nb thin films with $T_c > 4.2$ K via PLD, which cannot achieve an ultra high vacuum state. Nb08 reached the required standard because of the exceedingly high deposition temperature of 600°C, which took approximately 2 hours to reach, so the relatively short time spent at 600°C was sufficient to reach the required standard. Nb09 was deposited at just 400°C, and the preparation procedure was not quite as strict as Nb08 or Nb07 where the preparation time was shortened for convenience, and
its quality was reduced as result, having the lowest \( T_c \) and highest \( \Delta T \) of any of the early depositions.

Films Nb10 to Nb16, including Nb13 and Nb14 (not listed) experienced a severe decline in quality because of poor preparation conditions that failed to clear oxidants and other contaminants from the deposition chamber. Ultrathin Nb films on sapphire substrate display more sensitivity to substrate cleanliness than thicker films because any mismatch between the lattices of the substrate and Niobium will heavily influence \( T_c \) if film homogeneity is disrupted. Nb10 and Nb11 make this obvious from their relatively low deposition temperature, short preparation time, and low thickness with almost the worst results of any of the depositions. Samples Nb13 and Nb14 were excluded from the listing because they were not of sufficient quality to warrant inclusion with \( T_c < 4.2 \) K, however, their poor quality can be attributed to opening the deposition chamber for maintenance, which led to contamination but was cleared away by the heating cycles of subsequent depositions. Given that Nb15 is not of particularly high quality, alterations were made to the deposition procedure for Nb16, where the substrate was exposed to a small bias in an effort to increase cleanliness, and remove any oxides that may have formed on the surface.

### 4.1 Thin film microstructure

Figure 4.3 provides some measure of how ordered the microstructure is for the Niobium thin films in this work. A wide transition width typically means the sample is inhomogeneous, which indicates a less ordered microstructure. This in turn leads to a higher degree of intrinsic pinning than a well ordered microstructure, and increases the irreversible field value (the point where magnetisation loops become reversible) that is very close to \( B_{c2} \) for Nb. The upper critical field \( B_{c2} \) can be seen at the tail end of the magnetisation plots in figure 4.8 where the loops collapse in the high
4.1. Thin film microstructure

Figure 4.3: Comparison of $T_c$, $\Delta T$ and $B_{c2}$ across Nb depositions. The black, red, and blue dashed lines represent the averages for $T_c$, $\Delta T$ and $B_{c2}$ respectively.

Field region. The field at which the loops collapse defines the irreversibility field and is approximately equal to $B_{c2}$ for Nb. This is indicated in figure 4.3 by the similarity between $\Delta T$ and $B_{c2}$ values and their relationship to their own averages. It can be seen that for most of the samples that if the transition width for a sample is above the mean then $B_{c2}$ will also be above the mean.

Figures 4.4 and 4.5 present the $J_c(B_a)$ dependency of niobium films Nb-1 and Nb-2.

<table>
<thead>
<tr>
<th>Deposition parameter</th>
<th>Nb-1</th>
<th>Nb-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (nm)</td>
<td>450</td>
<td>450</td>
</tr>
<tr>
<td>$T_c$ (K)</td>
<td>8.4</td>
<td>8.9</td>
</tr>
<tr>
<td>Base pressure (mbar)</td>
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<td>$10^{-9}$</td>
</tr>
<tr>
<td>Deposition temperature (°C)</td>
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<td>500</td>
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<td>Argon pressure (mbar)</td>
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<td>$6.5 \times 10^{-3}$</td>
</tr>
</tbody>
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Table 4.3: Nb-1 and Nb-2 deposition parameters
4.1. Thin film microstructure

Figure 4.4: Critical current density dependence on magnetic field of Nb-1 thin film at $T = 4.2$ K, $dB_a/dt = 5 \times 10^{-3}$ T/s

Figure 4.5: Critical current density dependence on magnetic field of Nb-2 thin film at $T = 4.2$ K, $dB_a/dt = 5 \times 10^{-3}$ T/s
Figure 4.6: Comparison of deposition temperature, rate, and preparation time for $T_c$ (black squares), $\Delta T$ (red circles), and $B_{c2}$ (blue triangles). The coloured dashed lines represent the average of all values for each property.
4.1. Thin film microstructure

Figure 4.7: Comparison of plasma power, argon flow rate, and argon chamber pressure for $T_c$ (black squares), $\Delta T$ (red circles), and $B_{c2}$ (blue triangles). The coloured dashed lines represent the average of all values for each property.
4.1. Thin film microstructure

Figure 4.8: Magnetisation plots at 4.2 K, $dB_a/dt = 3.3 \times 10^{-3}$ T/s
Figure 4.9: \( J_c \) plots at 4.2 K, \( dB_a/dt = 3.3 \times 10^{-3} \) T/s
that were produced alongside the other Nb thin films in this work. Table 4.3 presents the deposition parameters of these films. These films were deposited to investigate the influence of vibration frequency and amplitude in a vibrating sample magnetometer (VSM) on \( J_c(B_a) \) which the plots in these figures are comparing. This is described in detail elsewhere\(^{11} \) and summarised in section 4.3. The effects of frequency and amplitude should be disregarded for the present because figures 4.4 and 4.5 also present clues on the effect of microstructure in Nb thin films. Film Nb-1 shows that \( B_{c2} \) is approximately 5 T and for Nb-2 \( B_{c2} \) is approximately 2 T, 2.5× smaller than Nb-1. The only difference between the two depositions was the deposition temperatures of 350 °C and 500 °C for Nb-1 and Nb-2 respectively. This tells us that a higher \( B_{c2} \) indicates a less ordered microstructure and \( B_{c2} \) is higher for lower temperature depositions. This less ordered microstructure also lowers \( T_c \) so it is a matter of picking the deposition parameters that will produce the desired superconducting properties.

Figure 4.6 a) builds on the result of figure 4.3 to present the influence of deposition temperature on thin film microstructure by measurement of \( T_c, \Delta T, \) and \( B_{c2} \). The black, red, and blue dashed lines represent the averages for each value respectively. It can be seen that samples deposited at 350°C have the least ordered microstructure, and exhibit the most intrinsic pinning, since all samples deposited at 350°C have an irreversible field value above the mean, and the same goes for almost all \( \Delta T \) measurements.

Strengthening the point made earlier about the interdependency of deposition parameters and their effect on thin film quality. It can be seen in figures 4.6 and 4.7 that only deposition temperature, and preparation displays signs of being critically responsible for controlling thin film properties. This is evident from the spread of points for each property \( T_c, \Delta T, \) and \( B_{c2} \), where it can be seen for each plot that there is no particular distribution around the average, so it looks almost random, indicating that
no single particular value for a parameter is ideal. Figure 4.6 b) is a plot examining the
effect of deposition rate on thin film quality and shows $T_c$, $\Delta T$, $B_{c2}$ are all distributed
almost evenly around their averages indicating there is no ideal deposition rate when
examined on its own. Most of the plots reveal a similar relationship, however, it can be
seen that there is an ideal range for some parameters, like the chamber pressure when
argon is flowing is on the order of $10^{-3}$ mbar. The exception to this is the effect of de-
position temperature on sample properties, where dependency can be clearly identified.

Figures 4.8 and 4.9 reveal highly differing magnetisation loops or vortex behaviour
for different samples. Since Niobium is a low $T_c$ superconductor it can be assumed
that thermal effects across pinning barriers are minimal\textsuperscript{47}, and vortex behaviour is de-
pendent on the microstructure since the measurement parameters are constant. These
plots were used to determine $B_{c2}$. All magnetisation measurements were made in the
MPMS at 4.2 K, and $\frac{dB_a}{dt} = 3.3 \times 10^{-3}$ T/s. Thin films Nb07, Nb08, Nb09, Nb17a,
and Nb17b all display signs of vortex avalanches known as flux jumps in figures 4.8
and 4.9 in their large peaks and rapid decline with increasing applied magnetic field.

The thin films Nb07, Nb08, Nb09, Nb17a, and Nb17b are the highest quality
samples with the optimal combination of superconducting properties like $T_c$, $\Delta T$, $B_{c2}$,
and $J_c$. While the information is somewhat obscure, it can be concluded that longer
preparation time will improve the quality (fig. 4.6 c)) because of greater deoxygenation
and general decontamination of the deposition chamber. A deposition temperature of
approximately 350$^\circ$C can produce high quality samples reasonably reliably, if other
conditions are adhered to, because it provides a good balance between cleanliness and
remaining below the bulk recrystalising temperature, which produces a thin film
microstructure with strong pinning properties\textsuperscript{10}. 
4.2 Flux jumps

Vortex avalanches describe the sudden and rapid penetration of large clusters of vortices in high and low-$T_c$ superconductors, and are currently explained by two main theories, flux jumps and self-organised criticality (SOC). Thin films are more prone to vortex avalanches than bulk superconductors because of nonlocal electrodynamics.\textsuperscript{7} Flux jumps are thermally triggered events that destroy the critical state of the superconductor. If the applied magnetic field is ramped up too quickly, vortices rush in, dissipating heat with their motion. If the thermal capacity and conductivity of the sample are small then this can produce local heating, which will further detach other vortices from their pinning sites leading to new motion and further heating. A positive feedback loop of vortex motion. This may destroy the critical state and quickly lower global supercurrents/magnetisation\textsuperscript{2}. SOC is a way of describing vortex distribution such that short range interactions lead to macroscopic distribution and behaviour, and the analogy is often made to a sandpile, where the magnetic field is represented by gravity and vortex pinning is represented by intergrain friction. In a sandpile, if one grain begins to move, it initiates the movement of others, so it never moves alone, and such a description is readily applied to vortex avalanches in superconductors. A critical point exists that is dependent on sample microstruture and measurement parameters that regulate vortex motion.

It is clear the vortex avalanches observed in figure 4.8 are flux jumps, since they are overwhelmingly reported\textsuperscript{2} in low $T_c$ superconductors at temperatures around 4 K.
4.3 Vibration effect on magnetization and critical current density of superconductors

This section will be a brief summary of the contents of an article I contributed to . . .


It is being included because parts of this work were the direct result of the Nb optimisation work described above.

This work made use of YBCO thin films, Nb thin films, bulk Nb, and MgB$_2$ to measure the effect of frequency and amplitude in vibrating sample magnetometry (VSM) measurements on superconducting properties. Unlike Nb, thermal activations influence vortex behaviour in YBCO near $B_{irr}^{36}$, so $B_{irr}$ and $B_{c2}$ are not close enough for the terms to be used interchangeably. The main results of this work can be summarised as follows.

In Nb thin films, higher vibration frequency and amplitude lead to degradation of the $J_c(B_a)$ curves above the low field region where flux jumps occur, but marginal effects are still visible below this point. There is also a lowering of $B_{irr}$, and formation of kinks in the $J_c(B_a)$ curve. Bulk Nb displayed similarly dependency on vibration amplitude and frequency but the effects are pushed back to relatively high magnetic fields ($B_a > 0.55$ T), and the $J_c(B_a)$ curves only show one kink.

The effects of vibration frequency and amplitude are mimicked by other superconductors like YBCO thin films, and bulk MgB$_2$. In MgB$_2$, $J_c(B_a)$ degradation is visible
4.3. Vibration effect on magnetization and critical current density of superconductors

but only at high fields (> 3.5 T) with only a single weak kink, pointing to differences in vortex dynamics between thin films and bulk samples. YBCO thin films show additional effects like an asymmetry in the descending and ascending branches of the magnetisation curves. The asymmetry of the descending branch is most obvious for the highest vibration frequency, and a tentative explanation is the vibrations have a stronger effect on the flux line lattice in decreasing fields rather than increasing fields, because an increasing field means more flux is still penetrating and can introduce some disorder increasing the pinning.
Chapter 5

Conclusion

The main goal of this thesis is to investigate the novel effects of uniform and non-uniform pinning arrays of large antidots in YBCO and Nb thin films.

The aim of this investigation into the effect of uniform and non-uniform arrays of large antidots in YBCO thin films where $\xi$ and $\lambda$ are much much smaller than the length of the antidot, was to build on the previous work surrounding uniform and non uniform arrays of pinning centres approximately the size of $2\xi(T)$. Uniform arrays of antidots on the order of this small length scale demonstrate $J_c(B_a)$ enhancement and peaks in $J_c(B_a)$ at matching fields when the vortex lattice period matches the pinning array period. These uniform arrays are also susceptible to interstitial vortex channels that form easily between the rows of antidots, which breaks down superconductivity. Later investigations into different arrays with antidots of a similar size revealed some broadening of the $J_c(B_a)$ peaks and suppression of the channelling.

It was demonstrated in this work that uniform arrays of large antidots reduce $J_c(B_a)$ across the whole applied magnetic field range for low and high temperatures, which is at odds with the success of uniform arrays in earlier works indicating that commensurability with the vortex lattice may not be as simple as matching the pin-
ning lattice parameter. Almost all non-uniform arrays of large antidots demonstrated significant enhancement of $J_c(B_a)$ across the whole field range for low and high temperatures. The graded and inverted array both demonstrate successful $J_c(B_a)$ enhancement regardless of the orientation of the triangular antidots. The evenly spaced ringed arrays show mixed results, where the narrowly spaced rings (20 $\mu$m and 40 $\mu$m) demonstrate a reduction of $J_c(B_a)$ and the wider spaced rings of antidots (80 $\mu$m and 140 $\mu$m) generally show an enhancement of $J_c(B_a)$ albeit smaller than that afforded by the graded and inverted arrays.

The success of these arrays has a number of possible explanations. Early investigations in films with little to no intrinsic pinning and relatively small pinning centres (saturation number $n_s \approx 1$) point to the suppression of interstitial vortex channels and restriction of vortex hopping between pinning centres. Given the high intrinsic pinning of YBCO and the large size of the triangular antidots ($n_s \geq 200$) it is not clear whether either of the effects demonstrated previously are taking place in these films. The high intrinsic pinning means that there may not be any tangible suppression of interstitial flux channelling as a result of the non-uniform array, and vortex hopping between antidots is not likely to be taking place in the case of collective vortex pinning. A novel situation is now proposed where a kind of vortex 'vacuum' is produced behind each row of antidots restricting vortex motion. An effect that can be tuned with the size and distribution of antidots. This vortex vacuum effect may also just be another way of interpreting the suppression of flux channelling.

The opportunity was also taken to investigate possible ratchet designs as part of these large antidot arrays that have previously been shown to initiate some ratchet effect because of collective vortex effects. It was proposed that some ratchet effect might be detectable between the graded and inverted arrays but none was discovered. More hope for a possible ratchet design was placed in variation of triangular antidot
orientation where vortices will be forced against the apex or base of the triangle, as applied field increases, producing more resistance in one direction than the other, but again no ratchet effect was reliably observed.

The optimisation of Nb thin film deposition was challenging because of the interdependence of chamber and plasma parameters, and one-to-one correlations were difficult to detect. It has been shown previously\textsuperscript{53} in ZnO thin films that only combinations of parameters were successful in producing high quality films and that same fact likely applies here. It was conclusively found that cleanliness prior to and during deposition are critical for Nb thin films so the deposition chamber needs to be thoroughly decontaminated of oxygen and other contaminants, which will adhere to the walls, and can be removed with a lengthy baking time prior to deposition. Producing ideal thin films also requires balancing the microstructure so it provides the necessary amount of intrinsic pinning and current transparency. This is tuned by controlling the microstructure, which is in turn manipulated by factors like substrate choice, cleanliness, target energy, deposition temperature etc. It was found that depositing these films at 350\textdegree C, was ideal, and consistently produced the best films when other parameters were kept within acceptable limits. The other parameters were not fully optimised, because of how fragile the Mantis Deposition System used in this work is. Working ranges for deposition parameters were established prior to these optimisation efforts but improvement was impeded by the continuous state of disrepair of multiple components of the system.

\section{Applications}

Superconductors are currently used worldwide, in MRI machines, particle colliders, and fusion reactors to name some. Research into antidots can be to develop tech-
niques in arresting vortex motion within current applications, increasing the maximum critical currents and hence the maximal magnetic field that can be produced. It has also been used in the suppression of low frequency flux noise in superconducting quantum interference devices. It can be used for not just improving current technology but also developing completely new technology, like nanoscale superconducting diodes, which use arrays of triangular dots and antidots to produce field tunable ratchet effects.43,51

5.2 Future Work

Future investigations on this topic could focus on simulation of uniform and non-uniform pinning arrays of large antidots and/or magneto-optical imaging of patterned films at low and high temperatures, to observe in real time how the magnetic flux front is affected by the presence of the antidots. It is intended to replicate these arrays in Nb thin films to compare the effect of arrays like these between low and high temperature superconductors. It would also be edifying to compare the effect of uniform and non-uniform arrays in Nb thin films with highly ordered and less ordered microstructures to more precisely determine the influence of intrinsic pinning.
Publications


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