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**Vortex pinning
in superconducting thin films
using ordered nanoporous templates**

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par

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List of publications

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List of symbols and abbreviations

Roman

$\vec{A}$: vector potential
 a_v : vortex interdistance
AAO : anodized aluminum oxide
 b : extrapolation length where $\psi = 0$ in a ferromagnet
 B : magnetic induction
BCS : Bardeen - Cooper - Schrieffer
CMP : chemical and mechanical polishing
 d : interdistance of the pinning centers
 D : diameter of a pinning center
 e : electron electric charge
 $\vec{E}$: electric field
 F : Landau free energy
 $\vec{F}_i^{vv}$: vortex-vortex interaction force
 $\vec{F}_i^p$: vortex-pinning center interaction force
 $\vec{F}_i^d$: external force
 $\vec{F}_i^T$: random force from thermal fluctuations
 f_L : Lorentz force per unit length of the vortex
 $\vec{F}_L$: Lorentz force
 $\vec{F}_{tot}$: total force
 $\vec{F}_P$: pinning force
 $\vec{F}_M$: Magnus force
 f_c : critical depinning force
 f_p : pinning force
 $g(\epsilon_F)$: electron density of states at the Fermi level
GL : Ginzburg-Landau
 H : magnetic field
 H_1 : first matching field

H_c : critical magnetic field (for type I) and thermodynamical critical magnetic field (for type II)

H_{c1} : first critical magnetic field

H_{c2} : second critical magnetic field

H_{c3} : third critical magnetic field

I_c : critical current

j : supercurrent density

J_c : critical current density

k_B : Boltzmann constant

k_F : wave vector at Fermi level

K_0 : zero-th order Bessel function

K_1 : modified Bessel function

l : jump width

l_{e-} : electron mean free path

M : magnetization

MBE : molecular beam epitaxy

m^* : Cooper pair mass

n_s : saturation number

n_{sc} : Cooper pair density

n_v : vortex density

q : Cooper pair electric charge

R : radius of a pinning center

R_p : radius of the parabolic trap

S_H : total surface of the holes

S_T : total surface of the sample

t : superconducting film thickness

T_c : critical temperature

T_{c0} : critical temperature at zero field

TAFF : thermally activated flux flow

U : height of the barrier potential

V : attractive potential between the 2 electrons of a Cooper pair

v_s : superfluid velocity

W : energy gained (or lost) by a vortex by jumping to a neighboring site

w : dimension of the superconductor perpendicular to the field

Greek

α : temperature dependent parameter

β : temperature dependent parameter

Δ : superconducting energy gap

η : viscous damping factor
 θ : phase of the superconducting order parameter
 Θ : Heaviside step function
 κ : Ginzburg-Landau parameter
 ξ : coherence length
 ξ_0 : intrinsic coherence length
 μ_0 : vacuum permeability
 λ : magnetic field penetration length
 Λ : modified field penetration length
 λ_L : London penetration length
 λ_{eff} : effective magnetic field penetration length
 λ_{BCS} : BCS magnetic penetration depth
 ν_0 : attempt frequency
 ϕ_0 : quantum of magnetic flux
 ψ : superconducting order parameter
 ρ_f : flux flow resistivity
 ρ_n : normal state resistivity
 ω_D : Debye's angular frequency

Miscellaneous

h : Planck constant
 $\hbar$: reduced Planck constant
 $\hat{H}$: hamiltonian

General introduction

Superconductivity is one of the most remarkable collective state in solid state physics. Indeed, the superconducting state, which is a macroscopic quantum phenomenon, shows two unique properties : infinite conductivity and perfect diamagnetism. However, both properties are limited by three important parameters : the critical magnetic field H_c , the critical current I_c and the critical temperature T_c above which the normal state is recovered. Therefore, a huge deal of effort is done in order to increase these critical parameters in order to make them more suitable for practical applications. The comprehension of the mechanism that defines the values of H_c , I_c and T_c is thus of major importance in order to be able to model the superconducting properties.

For type-II superconductors, the critical current is limited by the motion of vortices. Therefore, the introduction of efficient artificial pinning centers for the vortices in superconductors is a long-standing issue. Ion bombardment has proven to introduce efficient pinning center at the disordered track produced by the highly energetic ion [1]. However, optimum pinning of the vortex lattice is reached if the lattice of vortices can match the pinning center lattice. Using lithographic techniques many different ordered arrays of pinning centers have been studied and have proven to be efficient. Holes and blind holes in thin films were particularly investigated [2–7]. The interaction of the vortex lattice with the periodic arrays of pinning centers gives rise to interesting commensurability effects, or so-called matching effects, when the period of the vortex lattice matches the period of the pinning center arrays.

Recently, the use of ferromagnets to tailor the superconducting properties of thin films have been profusely reported [8–11]. In particular, the field compensation of the external magnetic field by the stray field of ferromagnets have been used to create field-induced superconductivity [9]. Another advantage of ferromagnets is the possibility to tune their pinning strength by modifying their magnetic state.

However, lithographic techniques are expensive and time-consuming. Therefore, so-called 'templates' and 'self-assembled' approaches have

been recently developed to achieve very dense array of pinning centers on very large surfaces [12–14]. These techniques have shown to maintain efficient vortex pinning at much higher field than conventional lithographic techniques. In this framework, anodized aluminum oxide membranes and templates have been used and have shown encouraging results.

The aim of this thesis is to **investigate the superconducting properties of NbN thin films containing a very dense array of defects of various types. We will present new approaches to imprint a lattice of defects in superconducting thin film based on the use of the porous alumina templates and membranes. On the one hand, we will take advantage of both surfaces of the alumina template to imprint antidot and blind hole array in NbN thin films. On the other hand, we will present a method to tailor the superconducting properties of thin films using electrodeposited Ni nanowires array in alumina membranes.** This issue will be addressed in five chapters.

Chapter 1 gives a short theoretical introduction to superconductivity, ferromagnetism and their mutual interactions. First, the main concepts of superconductivity are exposed and in particular the vortex pinning and motion. The second part is devoted to the ferromagnetism and particularly to the magnetic anisotropy and domain formation. Finally, the interactions between ferromagnets and superconductors are briefly discussed.

Chapter 2 describes the fabrication process and characterization of the different samples. The experimental setup is also described.

Chapter 3 mainly focuses on the introduction of core pinning (holes and blind holes) in NbN thin film taking advantage of the nanostructured surfaces of the AAO templates. First, we focus on the electrical properties of NbN thin films containing a triangular array of antidot with diameter 35nm and interdistance 50nm. Two different regimes are identified as the temperature is varied. Second, we discuss the vortex pinning properties in NbN thin films containing an hexagonal array of defects. The different stable vortices configurations (integer and fractional matching) are presented and the evolution of the pinning properties with the pinning strength is investigated. Finally, the particular case of the Nb deposition by MBE on the barrier layer is discussed. The electrical properties are investigated for different angles of deposition and thicknesses of the deposit. The resulting pinning center lattices are discussed in term of symmetry and efficiency.

Chapter 4 deals with the superconducting properties of a NbN thin films in close proximity with an array of ferromagnetic nanowires grown in the pores of an alumina membrane. First, the peculiar magnetic

properties of the single-domain Ni nanowire array are addressed. Second, the impact of the alumina surface roughness on the magnetic interaction between the superconductor and the ferromagnet is discussed. Finally, the vortex pinning properties of thin NbN film non-perforated by the nanowire array are analyzed. In this framework, the high field and the low field behavior are treated separately.

Chapter 5 is devoted to the electrical properties of NbN thin films perforated by a Ni nanowire array. We discuss the position and intensity of the first matching effect in terms of the magnetic state of the array. The influence of the magnetic history as well as the nanowire diameter is highlighted. Then, the origin of the misleading field-induced superconductivity is discussed in terms of the modification of the magnetic state of the nanowire array during the measurement.

Chapter 1

Theory

1.1 Superconductivity

1.1.1 Historical overview

Kamerlingh Onnes discovered superconductivity in 1911, three years after the first liquefaction of helium. While cooling mercury below 4.15K, he observed the abrupt drop of its resistivity to zero [15] (see figure 1.1(a)). The next years, many other metals showed the same abrupt transition and the highest critical temperature for the appearance of superconductivity in a pure metal was found to be 9.25K for Nb. During the following decades, many compounds were studied and the critical temperature of superconductivity slowly increased, reaching a plateau at 23K with the discovery of superconductivity in Nb_3Ge by Gavalier [16]. Moreover, the superconducting state can be suppressed by applying a certain (critical) current and magnetic field. In addition to the zero resistivity, Meissner and Ochsenfeld discovered in 1933 another unique property of the superconducting state : the perfect diamagnetism [17]. This so-called Meissner effect corresponds to the expulsion of the magnetic field out of the superconductor as represented in figure 1.1(b).

In 1935, the first theory of superconductivity was set by the London brothers [20]. Their theory was based on the Maxwell equation with an additionnal constraint necessary to explain the electric and magnetic field behavior inside a superconductor. This theory could not explain the microscopic origin of superconductivity but accounts for the Meissner effect. It was proved later that this oversimplified theory can be used very close to T_c and for high κ (see further) superconductors. The major limitation of this theory is that the superconducting electron density n_{sc} is suppose to be constant in space.

In 1950, Ginzburg and Landau [21] developed an adaption of the

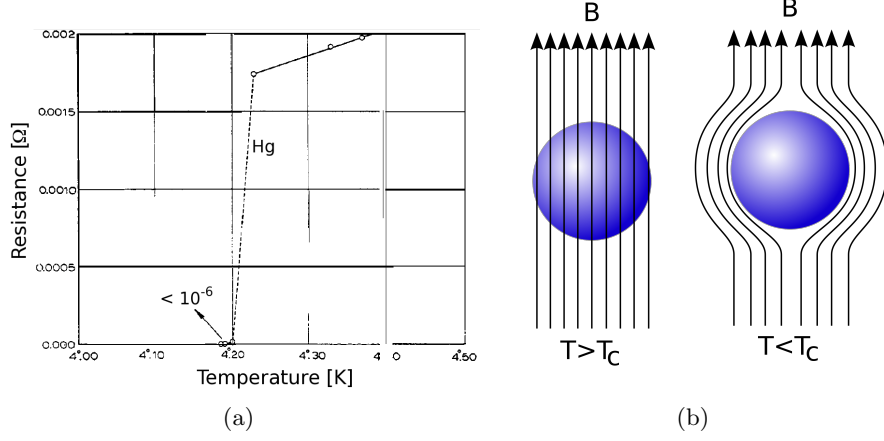


Figure 1.1: Superconductivity properties : (a) the perfect electrical conductivity (zero resistivity) and (b) the perfect diamagnetism. (a) Resistance as a function of temperature for a Hg sample at the superconducting transition. Measurement performed by H.K. Onnes et al. [18] when discovering superconductivity. (b) Schematic representation of the Meissner effect. Above T_c the magnetic field penetrates the sphere (normal state). Below T_c , the superconductor totally expels the magnetic field (perfect diamagnetism). Taken from reference [19]

general Landau theory of second order phase transitions for superconductor. The result was a generalization of the London theory to deal with situations in which n_{sc} varied in space, and also to deal with the nonlinear response to fields that are strong enough to change n_{sc} . Although, quite successful in explaining the behavior of spatially inhomogeneous superconductors, it was not accepted in the beginning because of its phenomenological foundation. In 1957, Bardeen, Cooper and Schrieffer [22] published a microscopic theory, which is now named the BCS theory. This Nobel prize awarded theory is widely accepted as the theory for superconductivity. In 1959, Gorkov demonstrated, using the BCS theory, the validity of the Ginzburg-Landau theory close to T_c and for 'not too abrupt' variations of n_{sc} and $\vec{A}$ (the potential vector). This allowed the acceptance of this theory by the scientific community and its extensive use.

In 1986, the path to radically higher T_c superconductors was opened by the discovery of superconductivity at $\sim 35K$ in LBCO (a mixed oxide of lanthanum, barium and copper) by Bednorz and Müller [23] for which they were awarded the Nobel prize the next year. Several months later, T_c reached 92K in "YBaCuO" [24], breaking the liquid nitrogen limit and therefore increasing tenfold the potential applications. This discovery

was surprising and exciting, not simply because of the large increase of T_c but also because it revealed that the oxides formed an unsuspected new class of superconducting materials with great potential. For such materials, the traditional BCS theory does not hold. At the moment, no consensus has been reached yet by the scientific community about the origin of the high temperature superconductivity.

It is worth mentioning that, up to know, 5 nobel prizes were awarded for ground-breaking researches in superconductivity which shows the scientific importance of such property.

1.1.2 The BCS theory

The BCS theory is the microscopic theory of superconductivity. Unfortunately, it's complexity limits its use and the Ginzburg-Landau theory is generally preferred for theoretical works. The basic phenomenon underneath the superconductivity is the electron-phonon coupling. Indeed, after the experimental demonstration that the critical temperature was dependent on the atomic mass (the isotope effect) by Fröhlich [25], Leon Cooper [26] demonstrated that two electrons in the Fermi sea can form a bound state with an attractive interaction even infinitely small. This attractive interaction between two electrons takes generally place through the exchange of phonons (which explains the isotopic effect). A schematic representation of this phenomenon is shown on figure 1.2 : a first electron attracts the surrounding positively charged nucleus and this increased concentration of positive charges, in turn attracts another electron. Despite this apparent simplicity, this phenomenon is in fact a purely quantum effect.

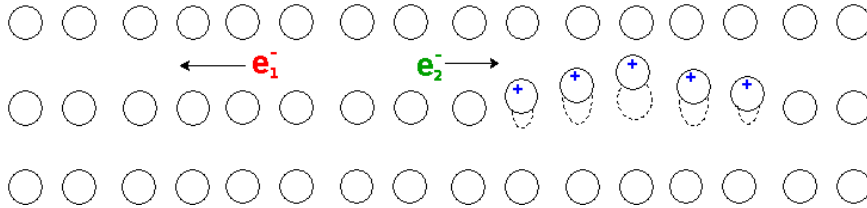


Figure 1.2: Schematic representation of a Cooper pair formation. A first electron locally deforms the positively charged lattice (left electron), a second electron with opposite direction (right electron) is attracted by the local concentration of positive charges. The two electrons form a Cooper pair through the exchange of a phonon.

Indeed, the BCS theory states that due to this attractive phenomenon, the electrons group together into pairs. The energy of a pair being lower than the energy of the two isolated electrons, some bound

and unbound states are formed (like in molecules) separated by a gap 2Δ . Therefore, this gap represents the difference between the superconducting and normal states. The bound states are restricted to certain conditions : the electrons must have opposite impulse and spin. Two electrons of a single pair actually move in opposite direction. The energy gap (the energy of the Cooper pair) is given by :

$$\Delta(0) = 2\hbar\omega_D \cdot \exp\left(-\frac{1}{g(\epsilon_F)V}\right) \quad (1.1)$$

where ω_D is Debye's angular frequency, $g(\epsilon_F)$ is the electron density of states at the Fermi level and V is the attractive potential. In order to increase the critical temperature, one can either increase the attractive potential V or the density of states at the Fermi level or the Debye's angular frequency. The BCS theory generally does not account for the high temperature superconductivity in its native form, because high T_c cuprates properties do not fit with the measured gap and T_c . Therefore, other electron pairing mechanism have been proposed but this is beyond the scope of this brief introduction. We would also like to emphasize that the three main critical properties of superconductivity are linked to the BCS gap by the following formulas :

$$T_{c0} = \frac{0.56\Delta(0)}{k_B}, \quad J_c(T) = \frac{n_{sc}q\Delta(T)}{\hbar k_F}, \quad H_c(T) = \sqrt{\frac{g(\epsilon_F)}{\mu_0}} \Delta(T) \quad (1.2)$$

It is also worth mentioning that due to the very large Cooper pair extension (much bigger than the mean electron-electron interdistance) many Cooper pairs are entangled which strongly complicates the theoretical treatment of the BCS theory.

1.1.3 The Ginzburg-Landau theory

The basic idea of Landau [27] was that the equilibrium value of a given order parameter (the magnetization, the polarization,...) at any temperature and pressure, can be determined by minimizing with respect to the order parameter, a variational thermodynamic potential $F(T,P,\psi)$, generally called free-energy. This potential is given by the external conditions imposed to the system. This theory is strongly based on the Group theory. The general formula for the expansion of the Landau free-energy is :

$$F(T, P, \psi) = F_0(T, P) + a(T, P)\psi^2 + b(T, P)\psi^4 + \hat{H}\psi \quad (1.3)$$

where $a(T, P)$ and $b(T, P)$ are parameters only dependent on the temperature and pressure, ψ is the order parameter and $\hat{H}$ is the hamiltonian of the system.

The Ginzburg-Landau theory is the adaptation of the Landau theory for superconductors. The superconducting electrons density ($|\psi|^2 = n_{sc}$) was chosen as the order parameter of the system. The free energy of the superconductor is :

$$F_s = F_n + \alpha|\psi|^2 + \frac{\beta}{2}|\psi|^4 + \frac{1}{2m^*}|(-i\hbar\vec{\nabla} - 2e\vec{A})\psi|^2 + \frac{B^2}{2\mu_0} \quad (1.4)$$

1.1.3.1 Ginzburg-Landau equations

Minimizing equation 1.4 with respect to ψ and $\vec{A}$ lead respectively to :

$$\frac{1}{2m^*}(-i\hbar\vec{\nabla} - 2e\vec{A})^2\psi + \beta|\psi|^2\psi = -\alpha\psi \quad (1.5)$$

and

$$\vec{j} = \frac{e}{m^*}[\psi^*(-i\hbar\vec{\nabla} - 2e\vec{A})\psi + \psi(i\hbar\vec{\nabla} - 2e\vec{A})\psi^*] \quad (1.6)$$

with $\vec{j}$ the supercurrent density. These two equations are called the Ginzburg-Landau (GL) equations. The Maxwell equation $\vec{j} = \vec{\nabla} \times \frac{\vec{B}}{\mu_0}$ and the vector potential definition $\vec{B} = \vec{\nabla} \times \vec{A}$ are used to solve self-consistently the 2 coupled GL equations and determine the spatial distribution of ψ and $\vec{A}$.

This phenomenological theory is still now extensively used because it allows fairly accurate prediction of the superconducting properties close to T_c despite its relative simplicity.

Using the usual notation of the order parameter with its modulus and phase : $\psi = |\psi|e^{i\theta}$, the second GL equation (equation 1.6) can be rewritten in a more intuitive form :

$$\vec{j} = \frac{2e}{m^*}|\psi|^2(\hbar\vec{\nabla}\theta - 2e\vec{A}) = 2e|\psi|^2\vec{v}_s \quad (1.7)$$

where $\vec{v}_s = (\hbar\vec{\nabla}\theta - 2e\vec{A})/m^*$ is the superfluid velocity.

In finite samples, boundary conditions are needed to describe the behavior of ψ and $\vec{A}$ at the interfaces. For superconducting/insulator interfaces the boundary condition is a zero perpendicular (to the interface) current :

$$(-i\hbar\vec{\nabla} - 2e\vec{A})\psi|_{\perp} = 0 \quad (1.8)$$

Which is very similar to the impulsion operator applied to a wave function.

1.1.3.2 Characteristic length scales

The 2 GL equations (eq. 1.5 and 1.6), the Maxwell equation and the boundary conditions are the starting point of the description of any superconducting system. While solving these equations, one gets the spatial distribution of ψ and $\vec{A}$. Whatever the problem to be solved, there are 2 characteristic lengths : the coherence length ξ and the magnetic penetration depth λ . The coherence length sets the length scale over which the order parameter ψ can spatially vary, while the magnetic penetration depth is the length over which the magnetic field can vary. This last length was already introduced in the London theory. These two lengths are fundamental values for a superconductor because many of its properties are directly related to the value of λ and ξ .

Both ξ and λ depend on the electronic mean free path l_{e-} , on the temperature T and on the intrinsic coherence length ξ_0 , which represents the smallest size of a wave packet that the superconducting charge carriers can form. ξ and λ both diverge when approaching T_{c0} . In the dirty limit, i.e., for $l_{e-} \ll \xi_0$:

$$\xi(T, l_{e-}) = 0.855 \sqrt{\frac{\xi_0 l_{e-}}{1 - \frac{T}{T_{c0}}}} \quad (1.9)$$

and

$$\lambda(T, l_{e-}) = 0.64 \lambda_L(0) \sqrt{\frac{\xi_0}{l_{e-} (1 - \frac{T}{T_{c0}})}} \quad (1.10)$$

where $\lambda_L(0) = \sqrt{\frac{m^*}{4\mu_0 e^2 n_s}}$ is the London penetration depth at $T=0$. n_{sc} is the Cooper pair density and $m^* = 2m_{e-}$ is the mass of the Cooper pair.

For the clean limit, i.e., $l_{e-} \gg \xi_0$:

$$\xi(T, l_{e-}) = 0.74 \frac{\xi_0}{\sqrt{1 - \frac{T}{T_{c0}}}} \quad (1.11)$$

and

$$\lambda(T, l_{e-}) = \frac{\lambda_L(0)}{\sqrt{2}\sqrt{1 - \frac{T}{T_{c0}}}} \quad (1.12)$$

When the dimension (t) of the superconductor perpendicular to the external field becomes smaller than the penetration depth, its extend in the superconductor is modified. Indeed, Pearl has demonstrated [28] that in thin superconducting film with perpendicular magnetic field with $t \ll \lambda$, the effective penetration depth should be renormalized as :

$$\lambda_{eff} = \frac{\lambda^2}{t} \quad (1.13)$$

For porous films, the easier penetration of the magnetic field into the film also leads to a modification of the effective penetration depth [29] :

$$\Lambda = \frac{\lambda}{\sqrt{1 - 2S_H/S_T}} \quad (1.14)$$

where S_H is the surface of the holes and S_T is the total surface. The ratio S_H/S_T corresponds to the porosity of the film. For porous thin film, both modifications should be made which can lead to very large penetration depth compared to the bulk material. These size effects on λ are a good example of how the superconducting properties are modified by the nanostructuration.

1.1.3.3 Type I and type II superconductors

The ratio between λ and ξ is called the Ginzburg-Landau parameter (κ) :

$$\kappa \equiv \frac{\lambda}{\xi} \quad (1.15)$$

This parameter determines the type of bulk superconductor. Type I ($\kappa < \frac{1}{\sqrt{2}}$) and type II ($\kappa > \frac{1}{\sqrt{2}}$) superconductors show very different behavior when exposed to magnetic fields.

The superconducting state is bounded by both the temperature and the magnetic field. Above a certain critical temperature (T_c), the material goes into the normal state. The H-T phase diagram for both type-I and type-II superconductors are shown in figure 1.3(a) and 1.3(b) respectively. A zero temperature, the critical magnetic field ($H_c(0)$) is maximum. The magnetization curve for both type-I and type-II superconductors are shown in figure 1.3(c) and 1.3(d) respectively. For type I superconductor, the zero resistivity and the Meissner state is present for

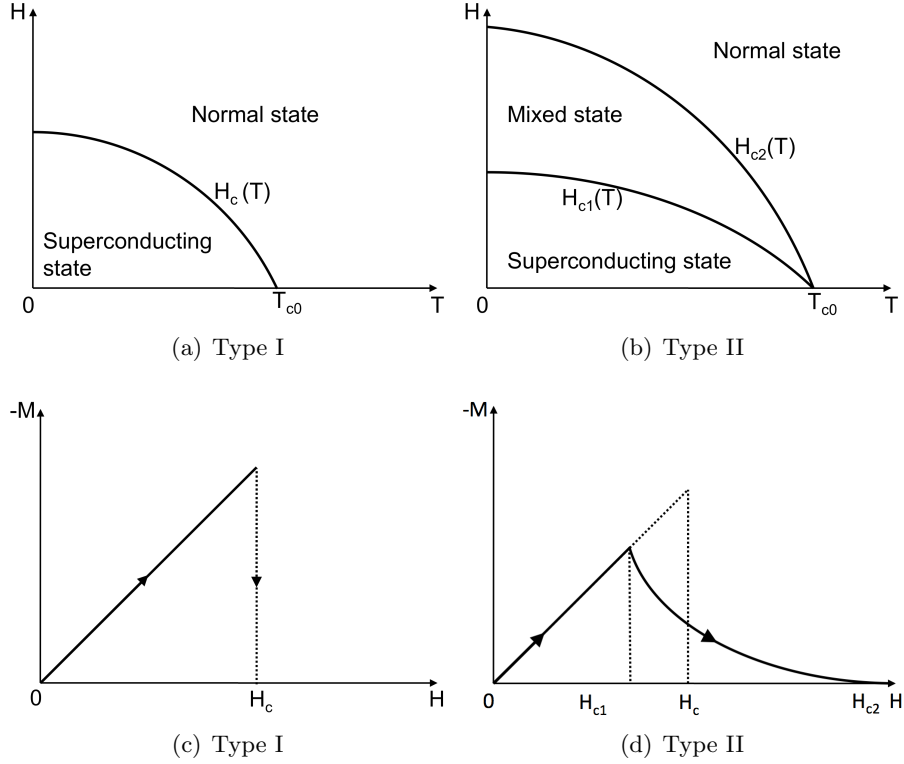


Figure 1.3: H-T phase diagram for (a) type I and (b) type II superconductors and magnetization curves for (c) type I and (d) type II superconductors. The Meissner effect is maintained below $H_c(T)$ for type I and below $H_{c1}(T)$ for type II superconductors : $M = -H$. The appearance of vortices above H_{c1} leads to the decrease of the diamagnetism (decrease of $|M|$). $\mu_0 H_c$ for type II superconductors corresponds to the thermodynamical critical magnetic field. The normal state is recovered above $H_c(T)$ for type I and above $H_{c2}(T)$ for type II superconductors.

any temperature and field below $H_c(T)$ curve. For type II superconductor, the same behavior as type I superconductors is observed at low field. However, above a certain critical magnetic field H_{c1} , the perfect diamagnetism is lost but the normal state is not recovered. The superconductor is in the mixed state, i.e. magnetic flux bundles called vortices penetrate the superconductor. The normal state is reached at a second critical magnetic field $H_{c2}(T)$ generally much higher than $H_{c1}(T)$ and typical values of $H_c(T)$ of type-I superconductors. Therefore, the magnetization curves for type-I and type-II superconductors are very different. The Meissner effect leads to a linear behavior of the magnetization below $H_c(T)$ for type-I and below $H_{c1}(T)$ for type-II superconductors. Above $H_{c1}(T)$, vortices appear in type-II superconductors which leads to a decrease of the diamagnetism. The normal state is recovered above $H_c(T)$ for type I and above $H_{c2}(T)$ for type II superconductors.

1.1.3.4 Vortices in type II superconductors

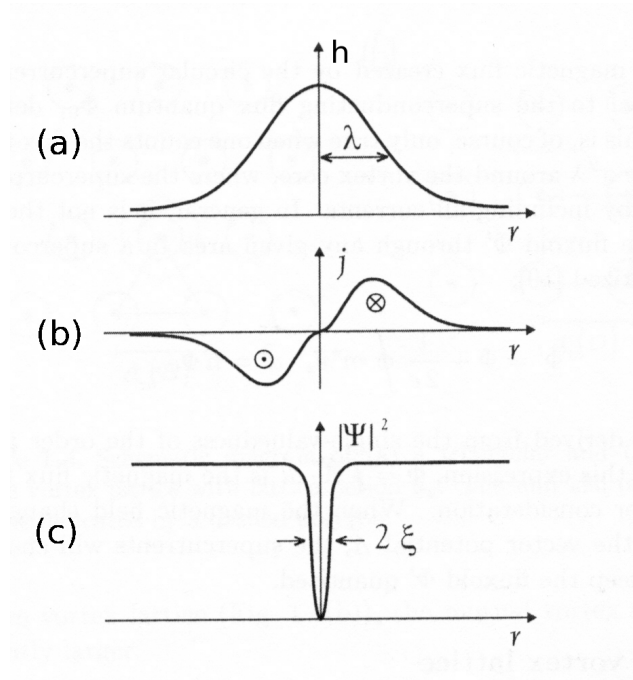


Figure 1.4: Schematic diagram of an Abrikosov vortex. Radial distribution of (a) the local field $h(r)$, (b) the supercurrent density $j(r)$ and (c) the Cooper pair density $n_{sc} = |\psi|^2$.

The apparition of the mixed state in type II superconductors is due

to a different superconductor/normal state interface energy which is related to κ . For $\kappa < \frac{1}{\sqrt{2}}$, this interface energy is positive whatever the field so this surface is minimized. These interfaces are limited to the physical boundary of the material only. When $\kappa > \frac{1}{\sqrt{2}}$, i.e. type II superconductor, it becomes energetically favorable to produce superconducting/normal state interface into the material above H_{c1} . Therefore, a certain amount of the external magnetic flux can penetrate the sample in tubes called vortices. These tubes are named vortices due to the surrounding supercurrent vortices that generate them. The magnetic field is maximum at the center of the vortex and drops to zero on an extend λ (see figure 1.4a). The radial distribution of the superconducting current density is represented in figure 1.4b. In the framework of the London model ($|\psi|^2(r) = n_{sc}(r) = cst$), which is valid for strong type-II superconductor ($\kappa \gg 1$), the field distribution can be calculated analytically [30] :

$$B(r) = \frac{\phi_0}{2\pi\lambda^2} K_0\left(\frac{r}{\lambda}\right) \quad (1.16)$$

with K_0 the zero-th order Bessel function and $\phi_0 = \frac{h}{2e}$ the quantum of magnetic flux.

Just at the center of the vortex, the Cooper pair density ($n_{sc}(r)$) drops to zero. The variation of the order parameter in the core of the vortex is spread on a length ξ (see figure 1.4c). Therefore, the vortex is a compromise between the loss of condensation energy on an extend ξ and a gain of magnetic energy on an extend λ . The vortex self-energy per unit length is given by [30] :

$$E_l = \frac{\phi_0^2}{4\pi\mu_0\lambda^2} (\ln\kappa + \epsilon) \quad (1.17)$$

where ϵ can be physically interpreted as the loss of condensation energy due to the normal core.

1.1.3.5 Vortex lattice

The interaction potential energy per unit length between two vortices separated by a distance r_{ij} can be calculated from the increase of energy associated to the overlapping magnetic fields of the vortices [31] :

$$U_{ij} = \frac{\phi_0^2}{4\pi\mu_0\lambda^2} K_0\left(\frac{r_{ij}}{\lambda}\right) \quad (1.18)$$

Thus vortices with the same polarity have a repulsive interaction. Therefore, the vortices will tend to maximize their interdistance to lo-

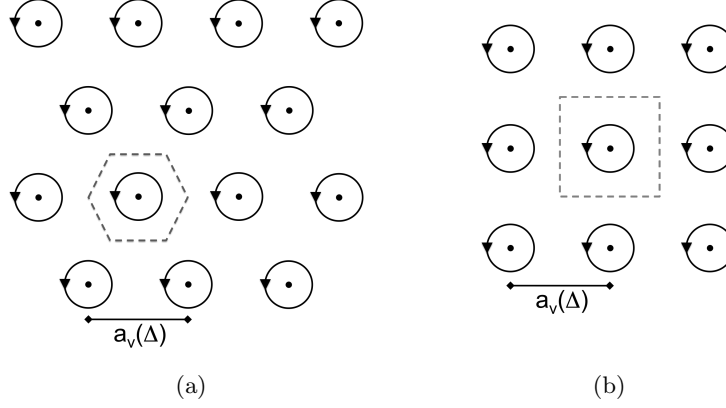


Figure 1.5: Examples of vortex lattices. (a) Triangular symmetry and (b) square symmetry. $a_v(\Delta)$ and $a_v(\square)$ are the vortex lattice interdistance in the triangular and square array respectively. The unit cell of the lattice is represented in dashed.

wer their interaction energy leading to the formation of a vortex lattice. Abrikosov found that the triangular lattice corresponds to the minimum energy (see figure 1.5(a)) [32] because it maximizes the interdistance between the vortices. Therefore, triangular lattices of vortices are often called Abrikosov lattices. Later, Kleiner et al. [33] extended the calculation of Abrikosov for a square lattice of vortices and found that its energy was a little bit higher than the triangular lattice. Accordingly, the triangular lattice of vortices is mostly found in experiments [34, 35]. However, in some materials, the symmetry of the underlying crystal structure appears to dominate over the small energy difference between triangular and square array of vortices (see figure 1.5(b)). This lead to a distorted triangle or square vortex lattice [36].

1.1.4 Vortex pinning

When a current flows in a type-II superconductor, a Lorentz force is exerted on the vortices. This force (per unit length of the vortex) is the vector product of the current density and the magnetic flux vector of the vortex :

$$\vec{f}_L = \vec{j} \times \vec{\phi}_0 \quad (1.19)$$

Therefore, the vortices are set in motion, generating dissipation. This dissipation leads to the breakdown of the superconducting state well below the depairing current.

1.1.4.1 Pinning mechanisms

Vortex in type-II superconductors can be pinned by local defects like grain boundaries, dislocations, precipitates, etc. Pinning centers can also be deliberately introduced in the sample to improve the vortex pinning. Several artificial pinning centers are columnar tracks introduced by ions [1, 37, 38] bombardment as well as holes [39, 40] or blind holes [3] introduced by lithographic techniques. Generally one can distinguish between two different interaction between the vortex and the pinning center [30] : core and electromagnetic interaction.

The **core interaction** takes its origin in the local decrease of the T_c at the defect. Therefore, condensation energy is saved when the core of the vortex is placed on the defect. Effective pinning of vortices is obtained for defects radius equal or larger than ξ . Indeed, Takezawa and Fukushima [41, 42] demonstrated that the pinning potential is very shallow for $R < \xi$ and that the depth of the pinning potential increases for increasing R in the range $\xi < R < \lambda$ while it saturates for $R > \lambda$.

The **electromagnetic interaction** is due to the perturbation of the supercurrents distribution of the vortices when it is close to a defect [43]. Therefore, this interaction takes place when the vortex is closer than λ to the defects. Such pinning is found for example in superconducting films with blind holes.

For sufficiently large defects, multi-quanta vortices also called 'giant' vortices can be pinned [4]. The maximum number of quanta that can be pinned by a defect (or the maximum vorticity of the giant vortex) strongly depends on the defect size, shape, type (insulating, superconducting, magnetic,...) and on the presence of surrounding vortices [39, 44, 45]. Two simple conditions deal with this problem and will be regularly used in this thesis. The saturation number n_s derived by Mkrtchyan and Schmidt gives the maximum number of vortices that can be pinned in a unique columnar defect situated in the center of an infinite sample [46]. The saturation number is given by :

$$n_s = \frac{D}{4\xi}, \quad (1.20)$$

with D the diameter of the columnar defect. This model can be used with good reliability for sample with low concentrations of defects. The condition of Buzdin [44] was derived from the London theory, which is basically valid close to T_c . It gives the minimum diameter D for columnar defects in a triangular lattice with interdistance d that can pin double-quantum vortices :

$$D > (8d\xi^2)^{\frac{1}{3}}. \quad (1.21)$$

1.1.4.2 Matching effect

The presence of pinning centers in a superconductor distorts the Abrikosov vortex lattice because the vortices are attracted by the pinning centers. Therefore, elastic energy is stored in the vortex lattice. For periodic pinning arrays, this elastic deformation energy is strongly reduced when all the pinning centers are occupied by one vortex. The most stable case is obtained for a triangular array of pinning centers because the deformation energy is zero. This leads to enhanced properties (critical current) at the magnetic fields at which the vortex lattice is commensurate with the pinning center lattice, this is called the matching effect. For large pinning forces, the vortex lattice can also commensurate with different symmetries than the triangular one because the gain of vortex pinning clearly overcomes the loss in deformation energy.

Therefore, the first matching effect is obtained when the vortex density and the pinning center density are equal. This condition is fulfilled at a magnetic field H_1 given by :

$$H_1^\Delta = \frac{2\phi_0}{\sqrt{3}d^2} \quad (1.22)$$

and

$$H_1^\circ = \frac{4\phi_0}{3\sqrt{3}d^2} \quad (1.23)$$

for respectively a triangular lattice and hexagonal lattice of pinning centers with interdistance d . The 'matching field' is the general term that designates the field at which a matching effect occurs.

Interstitial and giant vortices

At H_1 , all the pinning sites are occupied by a vortex. Therefore, additional vortices are either placed at interstitial positions of the pinning center lattice or on an occupied pinning site and form a doubly-quantized vortex (giant vortex of vorticity two). Basically, this is ruled by the saturation number n_s (or by the condition of Buzdin, equation 1.21). For $n_s > 2$, giant vortices are favored while for $n_s < 2$, interstitial vortices are favored. The superconducting properties strongly depend on the presence of interstitial vortices because they are much more mobile. Therefore, one usually tries to delay as much as possible the appearance

of interstitial vortices. If giant vortices are favored, the same fractional matching effects appear between H_1 and H_2 as between $H = 0$ and H_1 . Otherwise, new vortices configurations appears. Figure 1.6(a) and 1.6(b) shows respectively the vortices configurations at H_2 in a triangular lattice of pinning centers when giant vortices are favored and when interstitial vortices are favored respectively. Black dots represent the vortices and open circles represent the pinning centers. The giant vortices form a triangular lattice ($n_s > 2$) while the single-quantum vortices form an hexagonal array ($n_s < 2$).

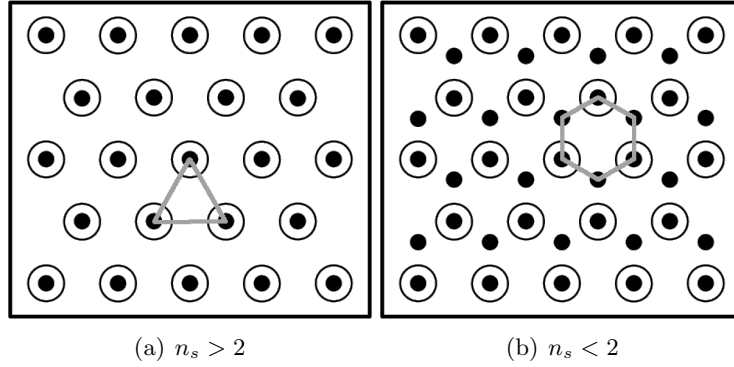


Figure 1.6: Vortex configurations in a triangular lattice of pinning centers at H_2 for (a) $n_s > 2$ and (b) $n_s < 2$. n_s is the saturation number, i.e. the maximum number of vortex that can be pinned in each pinning center. The open circles represent the pinning centers and the black dots represent the vortices. An hexagonal lattice of pinning centers is formed in presence of interstitial vortices.

1.1.4.3 The Bean critical state

The magnetization curve of ideal homogeneous singly-connected type-II superconductors is reversible and has been presented in figure 1.7. In the presence of pinning centers, the magnetization has an hysteretic behavior. This behavior can be qualitatively understood using the Bean model [47] which attributes the irreversible magnetization to the presence of field gradient (vortex density gradient) inside the sample. When a vortex enters the superconductor from its side, its movement to the center of the sample is hindered by pinning centers. When additional vortices enter the superconductor, the vortex-vortex repulsive interaction allows the initially pinned vortex to move inside the superconductor until the repulsive force with the neighboring vortices drops below the pinning force. Therefore, the gradient of vortices is directly related to the pinning force of the sample (i.e. the critical current density). The

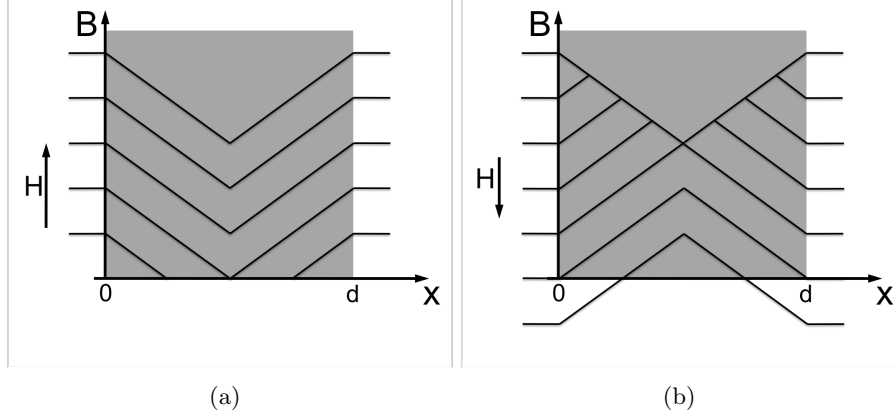


Figure 1.7: Magnetic field profile (vortex density profile) inside a type-II superconductor in the framework of the Bean model for different external fields. (a) In increasing field and (b) in decreasing field. The slope of the field profile is related to the critical current density.

field profile, i.e. the vortex density profile, is represented in figure 1.7 in the framework of the Bean model.

1.1.4.4 Vortex motion

When the current density becomes higher than the critical depinning current, vortices start to depin. Depending on the relative intensity of the pinning force and on the current density, one can have two different regimes called flux creep and flux flow. Flux creep corresponds to the vortices hopping from one pinning site to each other when the current density is of the order of the critical depinning current density. In some cases, this process will occur at a measurable rate. If the vortex pinning is weak compared to the driving force, the vortices will move in a rather steady motion, at a speed limited by viscous drag. This regime is referred as "flux flow".

Flux flow

When a current is applied in a type-II superconductor in the mixed state, the total force per unit length acting on a vortex is given by :

$$\vec{F}_{tot} = \vec{F}_L - \vec{F}_P - \eta \vec{v} + \vec{F}_M \quad (1.24)$$

where $\vec{F}_L = \vec{J} \times \vec{\phi}_0$ is the Lorentz force, $\vec{F}_P \approx \vec{J}_c \times \vec{\phi}_0$ is the pinning force, $\eta \vec{v}$ is the viscous damping force proportional to the vortex velocity

$\vec{v}$ and $\vec{F}_M$ is the Magnus force, which tends to move the vortices in a direction perpendicular to the Lorentz force and is usually negligible.

The phenomenological flux flow theory is based on the idea that moving vortices have no inertia. Due to the viscous damping, the vortices reach a constant velocity :

$$\vec{v} = \frac{(\vec{J} - \vec{J}_c) \times \vec{\phi}_0}{\eta} \quad (1.25)$$

The dissipation caused by the vortex motion is due to the generated electric field $\vec{E} = \vec{B} \times \vec{v} = n_v(\vec{\phi}_0 \times \vec{v})$ where n_v is the vortex density. Therefore, in the case of pinning free superconductor ($F_p = 0, J_c = 0$), the electric field is given by :

$$E = \frac{BJ\phi_0}{\eta} \quad (1.26)$$

In the case of steady flux-flow the E(J) or V(I) curves are therefore expected to be linear. The slope of the V(I) curves are proportional to the magnetic field, provided that the viscosity coefficient is independent of the magnetic field. Bardeen and Stephen have calculated the flux flow resistivity ρ_f that results from a steady vortex flow [48] :

$$\frac{\rho_f}{\rho_n} \approx \frac{2\pi\xi^2(T)B}{\phi_0} = \frac{B}{\mu_0 H_{c2}} = 2\pi n_v \xi^2(T) \quad (1.27)$$

where ρ_n is the normal state resistivity. In practice, the pinning force can generally not be neglected. A good agreement with this formula is then found when the pinning forces are completely overcome by the transport current ($J > J_c$), by replacing J by $J - J_c$.

Flux creep

When the applied current density J does not exceed the critical current J_c , the force supplied by the Lorentz force is insufficient to depin the vortices ($F_L < F_P$). However, at finite temperatures, the thermal energy may assist vortices to jump from one pinning site to another, generating vortex motion in the form of flux creep. The result will be a longitudinal voltage, proportional to the average creep velocity. This velocity is much lower than in the case of flux flow.

In the theory developed by Kim and Anderson, the electric field E , generated by the thermal hopping of vortex bundles between pinning sites, has a general expression given by [49] :

$$E = vB = \nu_0 l B \exp\left(-\frac{U}{k_B T}\right) \sinh\left(\frac{W}{k_B T}\right) \quad (1.28)$$

where ν_0 is the attempt frequency, l is the jump width, $U(H, T, J)$ is the height of the potential barrier between two adjacent pinning sites (activation energy), k_B is the Boltzmann constant, and v is the average vortex velocity. W is the energy that the vortex loses (or gains) by jumping to a neighboring pinning site. When $J \ll J_c \frac{k_B T}{U}$, the general formula reduces to

$$E \propto J \exp\left(-\frac{U(T, H, J)}{k_B T}\right) \quad (1.29)$$

This regime is called the Thermally Activated Flux Flow (TAFF) regime. For current densities much higher than the critical current density ($J \gg J_c$), i.e. in the flux flow regime, a linear $E(J)$ relation is found, in agreement with the equation 1.26 :

$$E \propto \sqrt{J^2 - J_c^2} \approx J \quad (1.30)$$

When the current density is of the order of the critical current density ($J \approx J_c$), expression 1.28 can be rewritten as :

$$E \propto \exp\left(-\frac{U(T, H, J)}{k_B T}\right) \quad (1.31)$$

The temperature, field and current dependence of the activation energy $U(H, T, J)$ can differ from system to system. For conventional type-II superconductors, one often uses the Anderson expression :

$$U(T, H, J) = U_0 \left(1 - \frac{J}{J_c}\right) \quad (1.32)$$

which expresses the tilt of the potential, proportional to the current density, due to the Lorentz force. U_0 is the potential depth at zero current. Depending on the pinning potential and the elastic response of the vortex lattice, other expressions for $U(T, H, J)$ [50] or even completely different $E(J)$ functions [51] may be more suitable to describe the experimental data.

1.1.4.5 Molecular dynamics simulations

In general, the vortex motion in the presence of periodic array of pinning centers is quite complex and the theory of Kim et Anderson is not valid. In principle, the time-dependent Ginzburg-Landau theory should be used to determine the different vortex motion regime but this is a

complicated task and time-demanding method for large systems. Therefore, molecular dynamics have been extensively used to model the vortices dynamics. Reichardt et al. have been very active in this field. They notably deeply investigated the vortex motion and stable configurations in triangular [52,53], honeycomb [54], kagome [54], partly disordered [55] and disordered [56] arrays of pinning centers. In this subsection, we briefly explain their methodology and some of their major results that are directly related to our experimental works.

The dynamics of a vortex i located at position R_i is determined by the following overdamped equation of motion :

$$\eta \frac{d\vec{R}_i}{dt} = \vec{F}_i^{vv} + \vec{F}_i^p + \vec{F}_i^d + \vec{F}_i^T. \quad (1.33)$$

Where $\vec{F}_i^{vv}$ is the repulsive vortex-vortex interaction force, $\vec{F}_i^p$ is the force from the pinning sites, $\vec{F}_i^d$ is the force from an external drive, and $\vec{F}_i^T$ is the random force from thermal fluctuations. The damping constant $\eta = \phi_0^2 d / 2\pi \xi^2 \rho_n$, where d is the thickness of the superconducting sample and ρ_n is the normal state resistivity of the material. The vortex-vortex interaction force is given by :

$$\vec{F}_i^{vv} = \sum_{j \neq i}^{N_v} f_0 K_1 \left(\frac{R_{ij}}{\lambda} \right) \hat{\mathbf{R}}_{ij}, \quad (1.34)$$

where K_1 is the modified Bessel function, $f_0 = \phi_0^2 / (2\pi \mu_0 \lambda^3)$, $R_{ij} = |\mathbf{R}_i - \mathbf{R}_j|$, and $\hat{\mathbf{R}}_{ij} = (\mathbf{R}_i - \mathbf{R}_j) / R_{ij}$.

The pinning sites are modeled as N_p attractive parabolic traps with radius $R_p = 0.3\lambda$ and strength f_p :

$$\vec{F}_i^p = - \sum_{k=1}^{N_p} \frac{f_p}{R_p} R_{ik}^{(p)} \Theta \left(\frac{R_p - R_{ik}^{(p)}}{\lambda} \right) \hat{\mathbf{R}}_{ik}^{(p)}. \quad (1.35)$$

Here, $\mathbf{R}_k^{(p)}$ is the location of the pinning site k , $R_{ik}^{(p)} = |\mathbf{R}_i - \mathbf{R}_k^{(p)}|$, $\hat{\mathbf{R}}_{ik}^{(p)} = (\mathbf{R}_i - \mathbf{R}_k^{(p)}) / R_{ik}^{(p)}$ and Θ is the Heaviside step function.

Finally, the thermal force $\mathbf{F}_i^T$ is modeled as random Langevin kicks with the properties $\langle \mathbf{F}_i^T \rangle = 0$ and $\langle \mathbf{F}_i^T(t) \mathbf{F}_j^T(t') \rangle = 2\eta k_B T \delta(t - t') \delta_{ij}$.

In this work, we mainly considered triangular and honeycomb lattices of pinning centers. In the following paragraphs, we present the theoretical predictions of Reichardt et al. [52–55] that are linked to our experimental findings.

Triangular lattice of pinning centers

Fractional matching effects strongly depend on the relative pinning strength compared to the vortex interaction force. For strong pinning centers, matching effects are observed for $H_n = n \cdot H_1$ with $n = 1, 6/7, 2/3, 1/3, 1/4$ and $1/7$. No ordered state is predicted at $n = 1/2$. The vortices positions for $n = 1/2$ in the case of strong pinning centers are represented in figure 1.8(a) which was taken from Reichhardt et al. [53]. In contrary, for weaker pinning strength a matching effect is possible at $H_{1/2} = 1/2 \cdot H_1$. In this case, half of the vortices are located at interstitial positions of the pinning array. The overall vortex lattice has a square symmetry as shown in figure 1.8(b). This configuration is possible due to the strong vortex-vortex repulsive interaction compared to the pinning force.

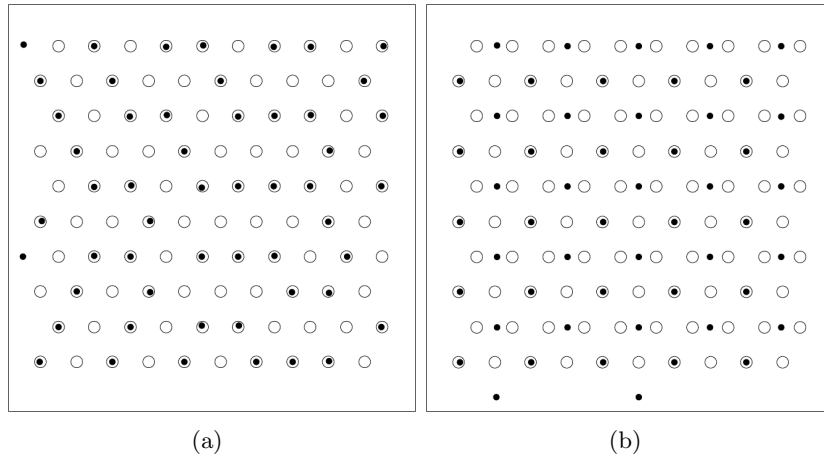


Figure 1.8: Vortices configurations at $H = \frac{1}{2}H_1$ for a triangular lattice of pinning centers. (a) With strong pinning strength, a disordered vortex configuration is observed. (b) With weak pinning strength, a square vortex configuration is observed with half of the vortices placed at interstitial positions. Taken from Reichhardt et al. [53].

Hexagonal lattice of pinning centers

For strong pinning strength, the vortices positions at different integer and fractional matching field calculated by Reichhardt et al. [54] are shown in figure 1.9 and 1.10. The vortices are represented by black dots and the pinning centers by open circles. For each field, the vortex position is shown with and without the pinning centers. Figure 1.9(a) and 1.9(b) shows the $H_{1/2}$ case. A triangular arrangement of the vortices

is observed which corresponds to a highly stable state. At H_1 (figure 1.9(c) and 1.9(d)), each pinning center is occupied by a vortex and therefore an honeycomb array of vortex is formed. At $H_{3/2}$ (figure 1.9(e) and 1.9(f)), an interstitial vortex is placed at the center of each hexagon. The overall vortex lattice forms a triangular lattice which explain the particular stability at this matching field. At H_2 (figure 3.10(b) and 1.10(b)), two vortices are present at the center of the hexagons. Therefore, the caging potential is reduced due to the interstitial vortex-vortex interactions. The matching peak is weaker. At low temperature, the interstitial vortices of different hexagons tends to align themselves. At higher temperature, due to thermal energy, the arrangement is lost and the two vortices continuously transit to the three degenerated states. At $H_{5/2}$ (figure 1.10(c) and 1.10(d)), three vortices are placed in the hexagon forming a triangle. The overall lattice shows a set of pentagons. The stability of this lattice is almost as high as the one at H_2 even though the number of vortices is higher. Finally at H_3 (figure 3.10(c) and 1.10(f)), the four interstitial vortices are placed in diamond-shape. The lattice of vortex is a set of irregular pentagons and the stability of this configuration is quite weak.

Using molecular dynamics, Reichhardt et al. [54] calculated the critical depinning force of the vortices and the vortices positions in an honeycomb pinning array with different pinning strengths. Their results for three different pinning strengths are represented in figure 1.11(a). The pinning center diameter was fixed as 0.6λ and the pinning center interdistance to 1.57λ which are very different values than those of our experiments where the pinning center diameter is about $\sim 0.01\lambda$ and the interdistance $\sim 0.02 - 0.03\lambda$ at 0K. Figure 1.11(a) shows that for strong pinning centers, the critical depinning force f_c is identical to the pinning force f_p at $H_{1/2}$ and H_1 . On the contrary, for weak pinning strength, the critical depinning force at H_1 is significantly lower than the pinning force while $f_c = f_p$ at $H_{1/2}$. The matching effect at $H_{3/2}$ has a similar behavior to the one at $H_{1/2}$. Indeed, it is more robust on the decrease of the pinning force than the one at H_1 . This is due to the higher stability of the triangular lattice of vortex when the vortex-vortex interaction is more important than the pinning strength. Figure 1.11(b) shows the evolution of the critical depinning force (f_c) as a function of the pinning strength (f_p) for three different magnetic fields (squares = $H_{1/2}$, circles = H_1 and triangles = $H_{1.5}$). At $H_{1/2}$, the critical depinning force is equal to the pinning strength for all strengths due to the triangular arrangement of the vortices. On the contrary, at H_1 , the critical depinning force is lower than the pinning strength for low strengths.

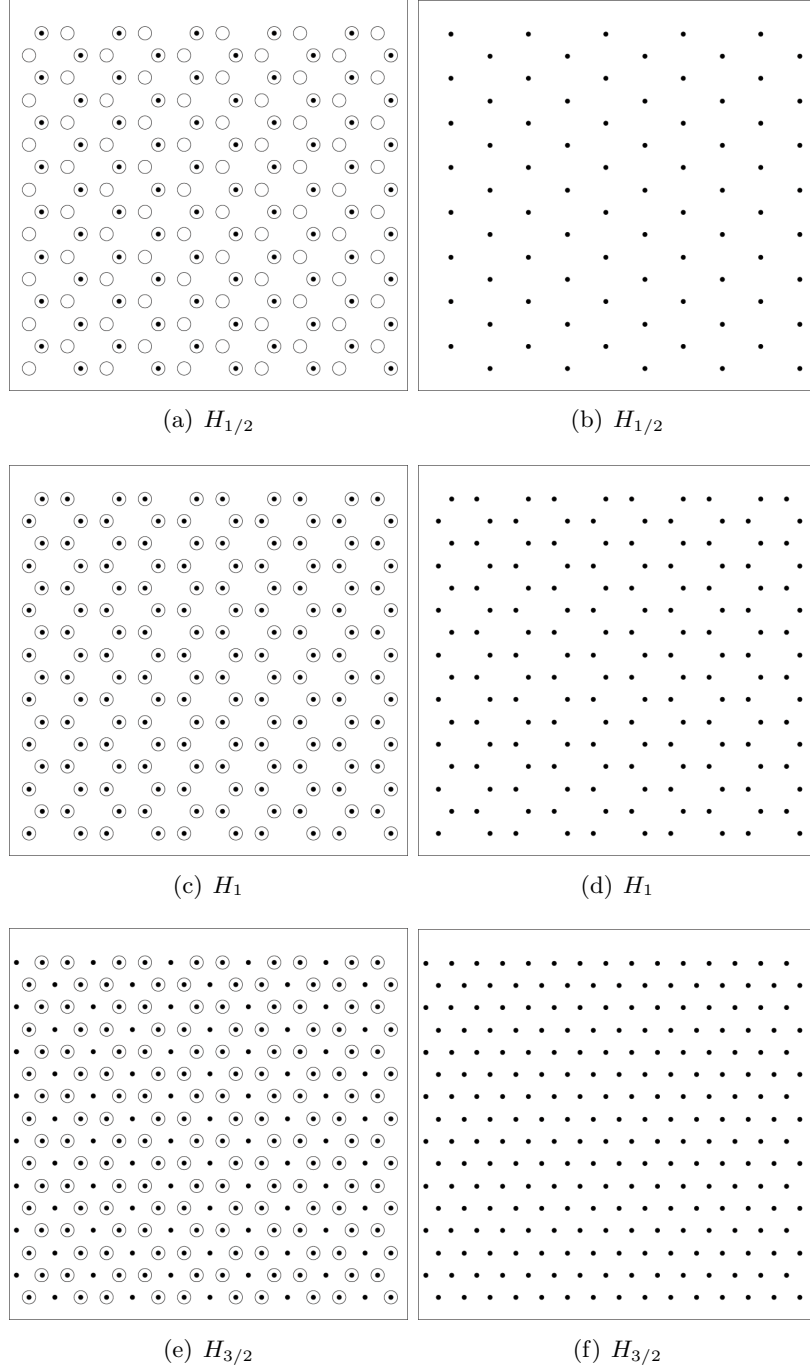


Figure 1.9: Vortices positions (black dots) for honeycomb lattice of pinning centers (open circles) at several matching fields, calculated using molecular dynamics. (a) and (b) $H_{1/2}$. (c) and (d) H_1 . (e) and (f) $H_{3/2}$. Vortex positions only : (b), (d) and (f). Taken from Reichhardt et al. [54].

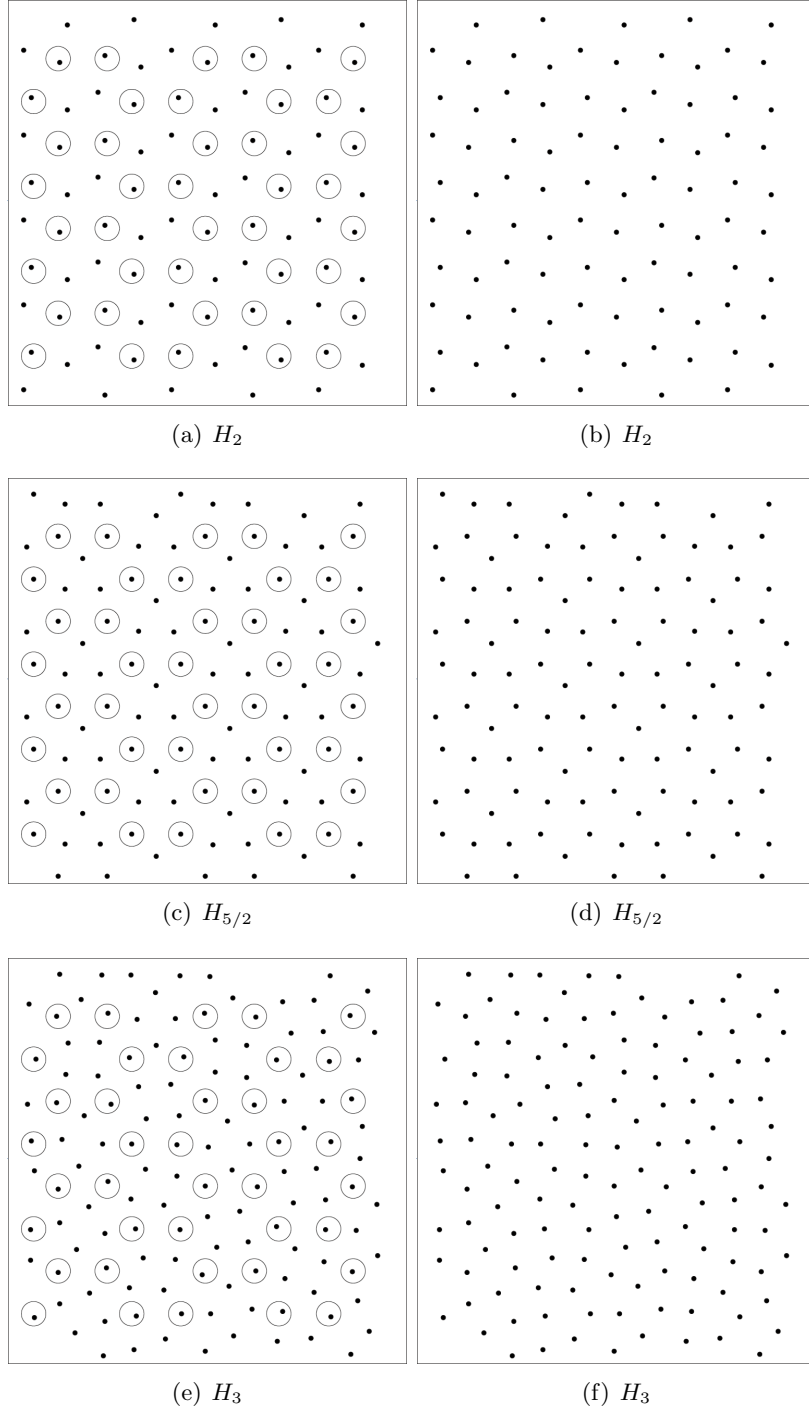


Figure 1.10: Vortices positions (black dots) for honeycomb lattice of pinning centers (open circles) at several matching fields, calculated using molecular dynamics. (a) and (b) H_2 . (c) and (d) $H_{5/2}$. (e) and (f) H_3 . Vortex positions only : (b), (d) and (f). Taken from Reichhardt et al. [54].

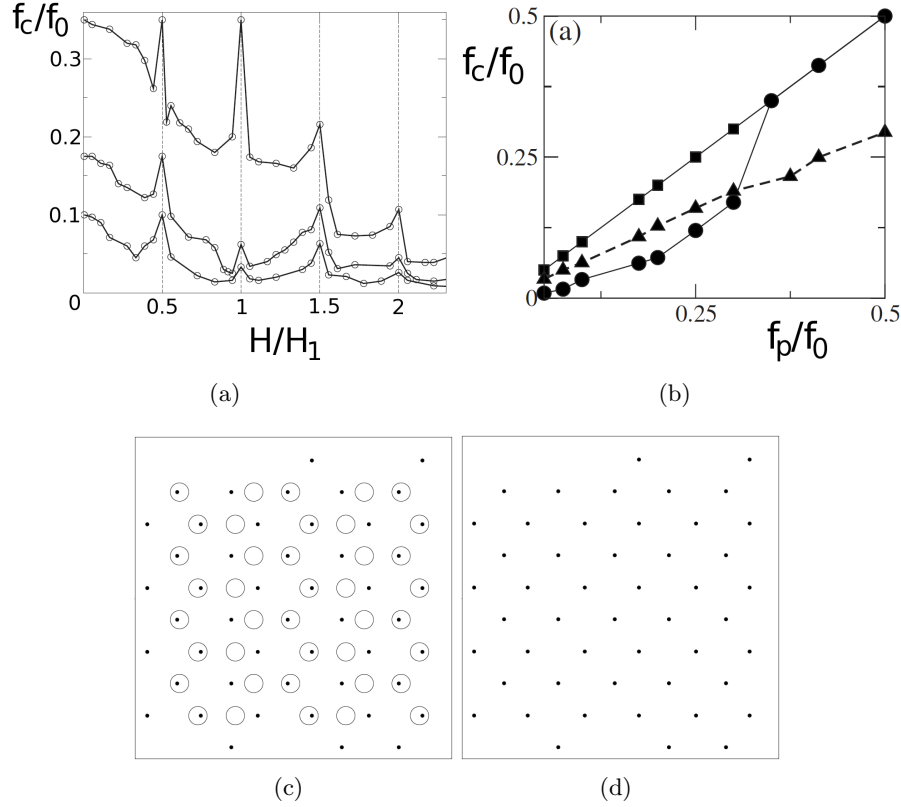


Figure 1.11: Theoretical results obtained from molecular dynamics for vortices in hexagonal array of pinning centers (taken from Reichhardt et al [54]). (a) Critical depinning force (f_c) as a function of magnetic field for three different pinning strengths. Upper curve : $f_p = 0.35f_0$, middle curve : $f_p = 0.175f_0$ and lower curve : $f_p = 0.1f_0$ (b) Critical depinning force (f_c) as a function of the pinning strength (f_p) for three different magnetic fields (squares = $H_{1/2}$, circles = H_1 and triangles = $H_{1.5}$). $f_0 = \frac{\phi_0^2}{2\pi\mu_0\lambda^3}$. (c) Vortices positions (black dots) and pinning site location (open circles) at $H = H_1$ for $f_p = 0.175f_0$. (d) Same as figure 1.11(c) with the vortex positions only.

1.1.5 Nucleation of superconductivity and critical fields

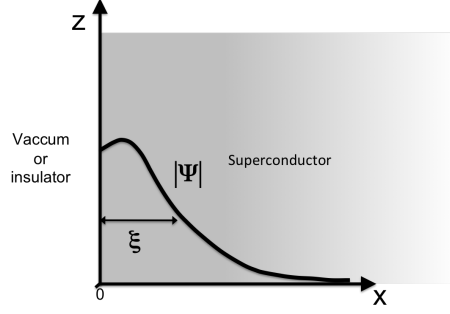


Figure 1.12: Schematic representation of the nucleation of superconductivity at the interface between vacuum or insulator and a semi-infinite superconductor. $|\psi|$ is the modulus of the order parameter and ξ is the coherence length.

For infinite samples, superconductivity nucleates below the second critical field H_{c2} . However, real samples have boundaries which modifies the superconductivity nucleation. For a magnetic field perpendicular to the surface, the nucleation field is equal to H_{c2} . For fields parallel to the surface, Saint-James and de Gennes have shown that superconductivity nucleates at a higher field [57] :

$$H_{c3} \equiv 1.69H_{c2} = 2.4\kappa H_c \quad (1.36)$$

Between H_{c3} and H_{c2} , the superconducting state is present on the boundary of the sample on a sheath of thickness ξ (see figure 1.12). The nucleation field is between H_{c3} and H_{c2} when the magnetic field is tilted at a certain angle from the surface.

For bulk superconductors, the second critical field is given by [30] :

$$B_{c2}(T) = \frac{\phi_0}{2\pi\xi^2(T)} = \frac{\phi_0}{2\pi\xi^2(0)} \cdot \left(1 - \frac{T}{T_{c0}}\right) \quad (1.37)$$

Therefore, the critical field (resp. the critical temperature) depends linearly on the temperature (resp. on the magnetic field) with the slope give by the sample characteristics ($\xi(0) = f(\xi_0, l)$). For mesoscopic samples, i.e. when $w \sim \xi$ (w one dimension of the superconductor), the order parameter is almost constant along this direction. The superconducting properties dramatically changes and it results notably in a modification of the field dependence of the critical temperature. Indeed, the critical field becomes [30] :

$$B_{c2}(T) = \frac{\sqrt{12}\phi_0}{2\pi w\xi(T)} \quad (1.38)$$

This leads to a quadratic critical temperature dependence on the magnetic field :

$$T_c(H) = T_{c0} \cdot \left(1 - \frac{\pi^2 w^2 \xi^2(0)}{3\phi_0^2} H^2\right) \quad (1.39)$$

with the quadratic coefficient depending on the superconducting thickness w and on the intrinsic properties of the sample ($\xi(0)$).

Determination of coherence length and penetration depth of NbN

The coherence length of bulk NbN can be extracted from the temperature dependence of the critical magnetic field (equation 1.37). However, it was found experimentally that $B_{c2}(0)$ of NbN is better approximated by the relation [58] :

$$B_{c2}(0) = 0.69T_c \left| \frac{dB_{c2}}{dT} \right|_{T_c}. \quad (1.40)$$

This relation state that the critical field has not a linear dependence on the temperature as expressed by equation 1.37 but the effective critical field is somewhat smaller (a factor 0.69). Then, the coherence length is obtained by using the relation $\xi(0) = \frac{\phi_0}{2\pi B_{c2}(0)}$. Using these relations gives for example $\xi(0) \sim 3nm$ for 20nm NbN films deposited on flat sapphire substrates. For NbN films deposited on nanostructured surfaces (AAO template), the coherence length can drop down to $\xi(0) \sim 1.6nm$.

The magnetic penetration depth at zero temperature is expressed in the dirty limit by the Bardeen-Cooper-Schrieffer (BCS) expression :

$$\lambda_{BCS}(0) = \sqrt{\frac{\hbar\rho_n}{\pi\mu_0\Delta(0)}}, \quad (1.41)$$

where $\hbar$ is the reduced Planck constant, μ_0 is the vacuum permeability, $\Delta(0)$ is the superconducting energy gap at zero temperature and ρ_n is the normal state resistivity. Using the relation $2\Delta_{NbN}(0) = 4.15k_B T_c$ [30], allows us to express λ only as a function of the normal state resistivity and the critical temperature. Therefore, the penetration depth increases with increasing resistivity and with decreasing critical temperature. Using typical value of resistivity ($\sim 4000\mu\Omega cm$) and critical temperature ($\sim 9K$) of the nanostructured sample leads to $\lambda(0) \sim 1 - 2\mu m$.

However, in this case the penetration depth should be corrected by taking into account the film thickness (see section 1.1.3.2, page 10) and the effective penetration depth becomes : $\lambda_{eff}(0) \sim 50 - 200\mu m$, i.e. several tenths of microns! Therefore, our samples are all in the extreme type-II limit since $\kappa > 10^4$.

1.1.6 Fluxoid quantization

A remarkable consequence of the quantum nature of the superconducting state is the fluxoid quantization effect. It is a direct consequence of the single valuedness of the order parameter. When taking the contour integral of the superconducting current i.e. the second GL equation (equation 1.7), around any closed path in the superconductor, one get :

$$\oint m^* \vec{v}_s \cdot d\vec{l} = \hbar \oint \vec{\nabla} \theta \cdot d\vec{l} - 2e \oint \vec{A} \cdot d\vec{l} \quad (1.42)$$

Taking into account that $\oint \vec{A} \cdot d\vec{l} = \phi$ (Stokes theorem; ϕ is the magnetic flux through the contour) and that ψ is a single valued function ($\oint \vec{\nabla} \theta \cdot d\vec{l} = 2\pi$), it gives :

$$\phi + \oint m^* \vec{v}_s \cdot d\vec{l} = n \frac{h}{2e} \equiv n \phi_0 \quad (1.43)$$

The left-hand part is called the fluxoid and is quantized because n is an integer.

This fluxoid quantization rule leads in some particular cases to the magnetic flux quantization. For example, imagine a type-I superconductor with a hole submitted to an external magnetic field above its critical temperature. If the sample is cooled below T_c , due to the Meissner effect, the field will be expelled. Therefore, there is some magnetic flux trapped in the hole. If we apply equation 1.43 to a contour around the hole but sufficiently far from it such that the superconducting screening currents are zero ($\vec{v}_s = 0$). Then, we get :

$$\phi = n \phi_0 \quad (1.44)$$

where ϕ is the magnetic flux into the contour of integration. This relation defines ϕ_0 , the quantum of flux. This remarkable result states that the magnetic flux into a superconducting region is quantized. This is also valid for the vortices if they are far from each other, i.e. if there is a closed path around them free of current.

But it can also lead to spectacular effects even when the superconductor is so thin that no contour free of current can be found. Such

experiment was performed by Little and Parks [59]. They measured the superconducting properties of a thin walled hollow cylinder submitted to a magnetic field parallel to the cylinder axis. Close to T_c , the coherence length diverges which means that the Cooper pair density ($|\psi|^2$) is constant in the superconductor and the screening currents are negligible. If the radius (R) of the cylinder is much bigger than the wall thickness, equation 1.43 can be rewritten :

$$v_s = \frac{\hbar}{m^* R} \left(n - \frac{\phi}{\phi_0} \right) \quad (1.45)$$

Therefore, the supercurrents will be directly proportional to the external magnetic flux through the cylinder. At $\phi = 1/2\phi_0$, it is energetically favorable to increase the phase number n from 0 to 1. Indeed, the kinetic energy of the Cooper pair is proportional to v_s^2 . As a result, the current changes its sign, i.e. it flows in the opposite direction. Periodic oscillations of T_c are thus observed with a minimum for $n - \frac{\phi}{\phi_0} = \frac{1}{2}$.

1.1.7 The collective regime or one-dimensional superconducting wire network limit

Superconducting one-dimensional wire networks (1DWN) consist of superconducting strands with width w smaller than the coherence length and with length L larger than the coherence length that are linked together thus forming a network. The square wire network in figure 1.13(a) can be considered as 1DWN if $w < 1.8\xi < L$ [60]. In this case, vortices can not be placed at interstitial positions and the superconducting order parameter is constant along the width of the strand and vary only along the length of the strand. Each superconducting strand is coupled to its neighbors leading to a collective behavior. Finite networks were first considered theoretically by de Gennes [61,62] in order to explain the behavior of granular superconductors. Then, Alexander extended the works of de Gennes for large networks and infinite networks [63]¹. The experimental realization of the 1DWN was first demonstrated by Pannetier et al. [64] and then by van der Zant et al. [65]. Superconducting 1DWN are very interesting because they allow to study well-known frustration effects and its is notably the experimental proof of the magnetic spectrum of the tight-binding electron model predicted by Hofstadter [66]. Indeed, ordered 1DWN are similar to Little-Parks loops [59] connected to each other. In this case, the fluxoid quantization must be fulfilled for each closed path of the network. In addition, the Kirchhoff law of current

1. In annexe A.3, the de Gennes-Alexander model is to our superconducting-ferromagnets hybrids.

should be fulfilled at each node of the network. The current loop in one cell is not always compatible with the current in the neighboring cells which lead to the so-called frustration effect. This frustration manifests itself as periodic decrease of the critical temperature of the network.

For this reason, many theoretical works have been performed on superconducting 1DWN [67–69]. An important feature of superconducting 1DWN is that the conventional concept of vortex disappear. Indeed, the current in one strand of the network is determined collectively by the external magnetic field and the symmetry of the lattice. For example, the current distribution at $f = 1/2$ (f is the frustration parameter defined by $f = H/H_1$) in an hexagonal 1DWN is shown in figure 1.13(b). Clearly, no conventional vortex is present in this case.

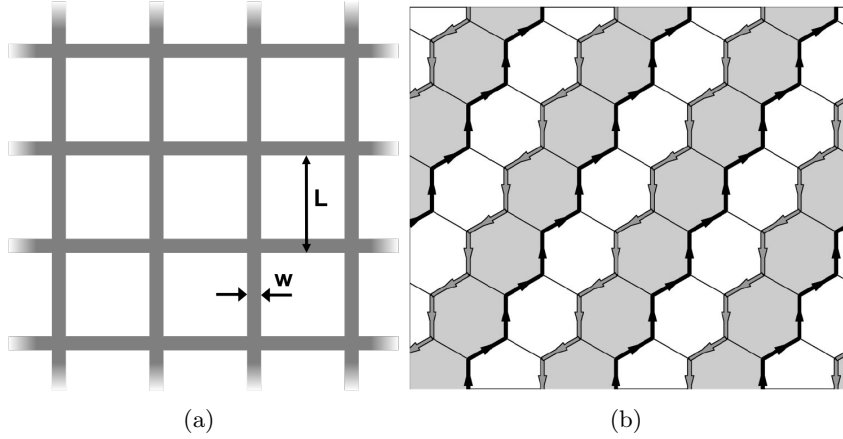


Figure 1.13: (a) Schematic representation of a square wire network. The lattice periodicity is L and the wire width is w . The wires are considered one-dimensional if $w < 1.8\xi$ and $L > 1.8\xi$ [60]. (b) Current distribution in a one-dimensional honeycomb wire network at $f = H/H_1 = 1/2$ (taken from [69]). No vortex in the usual sense is present.

As the coherence length diverges at T_c , thin superconducting films with ordered lattice of pinning centers reach the one-dimensional wire network for temperatures sufficiently close to T_c because the coherence length becomes larger than the pinning center interspace. Therefore, enhanced $T_c(H)$ oscillations are usually observed close to T_{c0} [39, 60, 70–74].

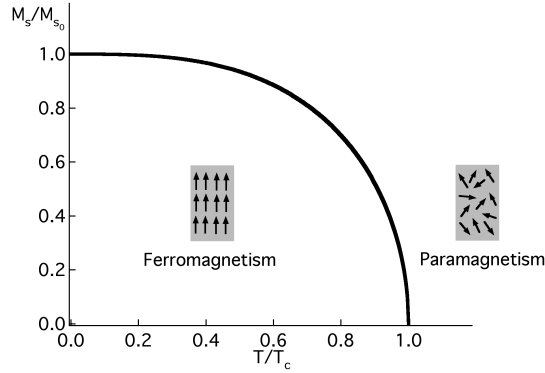


Figure 1.14: Magnetization at saturation as a function of temperature of a ferromagnetic sample. Below the Curie temperature T_c , the ferromagnetic order is present. Above T_c , the thermal energy is too high for the ordering of the spins, thus paramagnetism is observed. M_s is the magnetization at saturation and M_{s_0} is the zero temperature magnetization at saturation.

1.2 Ferromagnetism

Ferromagnetism is the property of certain materials to be able to form permanent magnets. In ferromagnetic materials, the spins align with each other on a macroscopic scale and form a magnet. However, this property is suppressed at high temperature by the thermal agitation energy which tends to disorder the spin alignment. When all the spins are aligned, the saturated state of the magnet is reached. The saturation magnetization decreases with increasing temperature. Figure 1.14 shows the typical dependence of the saturation magnetization as a function of temperature. The magnetization is maximum at 0K and decreases rapidly when getting close to the Curie temperature T_c where the ferromagnetic order disappears. Above T_c , the material exhibits a paramagnetic behavior. The Curie temperature of Ni is 631K, therefore, at the cryogenic temperature used in this thesis ($T < 10K$), the saturation magnetization can be considered independent on the temperature.

In this section, we briefly discuss the basic concepts of ferromagnetism that are relevant to understand the magnetic behavior of the Ni nanowires arrays used in this thesis.

1.2.1 Origin of magnetism

The ferromagnetism takes its origin in the exchange energy, whose origin was identified by Heisenberg [75] as a quantum mechanical exchange effect due to the overlapping of wave functions of neighboring

atoms with magnetic moments $\vec{m}_i$. The exchange energy is given by :

$$E_{ex} = -2\sigma \sum \sum \vec{m}_i \cdot \vec{m}_j \quad (1.46)$$

where σ is a term that characterizes the strength of the interaction between the magnetic moments. For ferromagnetic materials, σ is positive, resulting in a minimum exchange energy when all the moments are aligned.

1.2.2 Demagnetizing field

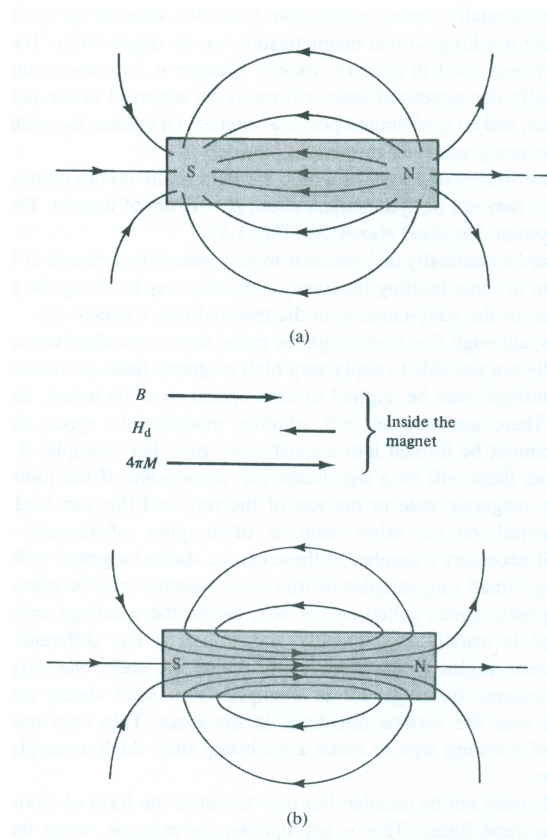


Figure 1.15: Schematic representation of the different fields inside and outside the ferromagnetic bar. (a) Demagnetizing field $\vec{H}_d = -N\vec{M}$. (b) Magnetic field $\vec{B} = \mu_0(\vec{H}_d + \vec{M})$. Inside the ferromagnet : $\vec{B} = \mu_0(\vec{H}_d + \vec{M}) = \mu_0(1 - N)\vec{M}$. Outside the ferromagnet : $\vec{B} = \mu_0\vec{H}_d$.

A magnetic field can be produced either by electric currents or by magnetic poles. If due to currents, the lines of $\vec{H}$ are continuous and form closed loops. If due to poles, the $\vec{H}$ lines begin on north poles and

end on south poles. Outside the sample, this field is generally called the stray field. Inside the sample, it is called the demagnetization field because it is opposed to the magnetization (see figure 1.15). The demagnetizing field is related to the magnetization by a tensor called the demagnetization factor ($\underline{N}$) :

$$\vec{H}_{dem} = -\underline{N} \cdot \vec{M} \quad (1.47)$$

Therefore, the magnetic field $\vec{B}(r)$ inside a sample with magnetization $\vec{M}(r)$ is given by :

$$\vec{B} = \mu_0(-\vec{H}_{dem} + \vec{H}_{ext} + \vec{M}) = \mu_0(1 - \underline{N}) \cdot \vec{M} + \mu_0\vec{H}_{ext}. \quad (1.48)$$

with $\vec{H}_{ext}$ the external field.

For infinitely long rods with magnetization along its axis (e.g. along z), the demagnetization factors are $N_z = 0$, $N_x = 1/2$, $N_y = 1/2$. Therefore, with the external field align with the nanowires axis, the magnetic field in the nanowire is : $B_z = \mu_0(H_{ext,z} + M_z)$.

On the opposite, for infinite films in the xy plane, the demagnetization factors are $N_z = 1$, $N_x = 0$, $N_y = 0$ and therefore $B_z = \mu_0 H_{ext,z}$ ($B_x = \mu_0(H_{ext,x} + M_x)$ and $B_y = \mu_0(H_{ext,y} + M_y)$). Therefore, for thin superconducting films, the vortex density n_v is directly related to the applied external field : $n_v = \frac{\mu_0 H_{ext}}{\phi_0}$.

1.2.3 Domain formation

Ferromagnets are not only ruled by the exchange energy, several other energies play a role in the final magnetic state of the ferromagnet. Here we briefly discuss these different energies.

1.2.3.1 Zeeman energy

The Zeeman energy corresponds to the energy of a magnetic moment placed in a magnetic field. The energy is maximum when the magnetization is anti-parallel to the external magnetic field and minimum when parallel. For a magnetization distribution $\vec{M}$, the Zeeman energy is :

$$E_H = -\mu_0 \int \vec{H} \cdot \vec{M} dV \quad (1.49)$$

This term favors the alignment of the magnetic moments with the external magnetic field.

1.2.3.2 Anisotropy energy

Magnetic anisotropy means that the magnetic properties depend on the direction in which they are measured. In the present work, we should only consider two contributions to the magnetic anisotropy :

- Crystal anisotropy or magnetocrystalline anisotropy :

The spin alignment in some directions is generally favored in certain cristalline directions. This terms thus strongly depends on the crystal lattice. However, in this thesis, we used Ni nanowires for which the magnetocrystalline anisotropy is relatively weak. One can associate an energy cost for the alignment of the magnetization at a certain angle from the easy axis, this is generally represented by the anisotropy energy K .

- Shape anisotropy :

Depending on the shape of the sample, some directions of magnetization will lead to a lower energy. For example, in the case of long nanowires (the present case), the wires axis constitutes an easy axis of magnetization.

1.2.3.3 Stray field energy

The stray field energy represent the energy associated to the presence of the stray magnetic field and is given by :

$$E_{stray} = \frac{\mu_0}{2} \int |\vec{H}_{stray}|^2 dV. \quad (1.50)$$

The stray field energy can never be negative. In a perfectly demagnetized samples, it is zero. This term favors the multi-domain state. Indeed, as represented in figure 1.16, the stray field energy associated to the homogeneously magnetized sample is large while the stray field of a demagnetized sample is reduced to zero.

1.2.3.4 Domain wall

Domain walls are interfaces between regions in which the spontaneous magnetization has different orientations. The first theoretical examination of the structure of a domain-wall was made by F. Bloch in 1932 [76], and domain walls are accordingly often called Bloch walls. Domain walls cannot correspond to abrupt changes in magnetization from one easy axis to another one. Indeed, in this case, the exchange energy, which tends to align the spin would be extremely large in the domain wall. Therefore, the exchange energy will tend to enlarge as much as possible the domain wall thickness in oder to minimize the different

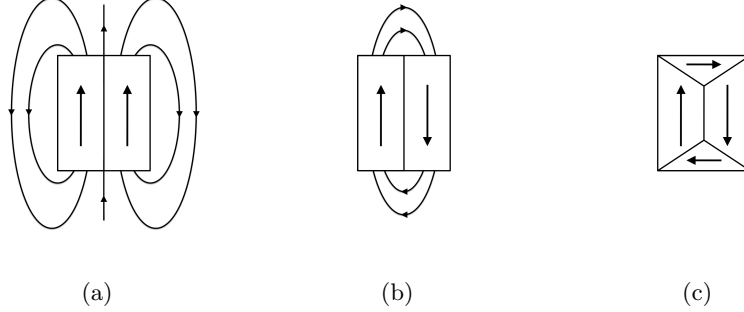


Figure 1.16: Schematic representations of different domain states and their corresponding stray fields. (a) Monodomain. (b) Bi-domain. (c) Demagnetized state. The stray field energy is clearly reduced by creating domains.

angular orientation between adjacent spins. Indeed, the exchange energy between two spins is given by :

$$E_{ex} = -2JS^2 \cos(\phi_{ij}) \quad (1.51)$$

with $\cos\phi = 1 - \frac{\phi^2}{2}$ for small ϕ . It is clear that distribute the rotation on several spin is favorable. However, the spin within the walls are pointing away from easy axis so that the crystal anisotropy energy is higher within the wall. Therefore, the crystal anisotropy energy will tend to make the wall as thin as possible. The compromise between these two terms leads to an equilibrium domain wall thickness which is given by :

$$\delta = \pi \sqrt{\frac{A}{K}} \quad (1.52)$$

in the case of the uniaxial anisotropy. A is the exchange stiffness or exchange constant and K the anisotropy constant.

When some dimensions of the sample become comparable to the domain wall thickness, the energy associated with the free poles that arise where a Bloch wall meets the surface becomes significant. This can lead to change in the wall structure, with the magnetization rotating in the plane of the sample rather than in the plane of the wall. Such a magnetic domain wall is called a Néel wall. For sufficiently small particles, the creation of a domain wall is not energetically favorable. These particles are single-domain, i.e. the magnetization of the particle is homogeneously distributed along one direction. Such behavior has been observed also in thin ferromagnetic nanowires with diameter below the domain wall thickness [77, 78]. The strong shape anisotropy of

these structures only allows the two magnetic states corresponding to the homogeneous magnetization along the nanowires axis in either directions. The magnetic properties of Ni nanowires arrays will be discussed thoroughly in section 4.2 (page 103).

1.3 Superconductivity-ferromagnetism interaction

Superconductivity and ferromagnetism are in a sense opposite properties. While the internal magnetic field of ferromagnets is very high, the superconductors tends to expel the magnetic field from its interior. Moreover, the microscopic origin of these two fundamental properties of solid matter are opposite. In this section we briefly discuss the type of interactions that results when placing ferromagnetic and superconducting material in contact or in close proximity.

1.3.1 The exchange interaction

When superconductors and ferromagnets are placed in electrical contact, electrons can be transferred from one material to the other. This changes the boundary conditions (equation 1.8) for a superconductor-insulator interface to [31] :

$$\vec{n} \cdot (-i\hbar\vec{\nabla} - 2e\vec{A})\psi = \frac{i\hbar}{b}\psi \quad (1.53)$$

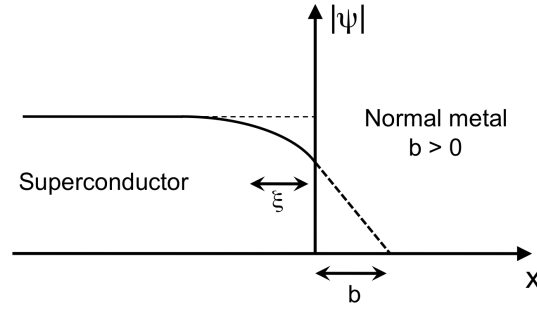


Figure 1.17: Spatial dependence of the superconducting order parameter modulus $|\psi|$ at the interface between a superconductor and a normal metal ($b > 0$). ξ is the coherence length and b a positive constant corresponding to the linear extrapolation of the distance in the normal metal at which the order parameter is zero.

where b is a real constant and $\vec{A}$ is the potential vector. As shown in figure 1.17, if $\vec{n} \cdot \vec{A} = 0$, b is the extrapolation length to the point

where $\psi(r)$ becomes zero in the contacting metal. This representation is valid for normal metal for which b can range from a few nm to several μm . In the case of ferromagnets, b is limited to $5 - 10 nm$ due to the strong Cooper pair breaking caused by the exchange interaction in the ferromagnet which tends to align the spins. Moreover, the wave-function has an additional oscillatory dependence. This peculiar property has led, for example, to the experimental realization of so-called π -junctions in $S/F/S$ trilayers [79] or superconducting spin switches which appear due to the spin polarization in the ferromagnetic layers [80]. In this case, the critical temperature of an $F/S/F$ trilayer depends on the relative orientation of the magnetization of the two F-layers.

1.3.2 Electromagnetic interaction

In this thesis, we are only interested in the electromagnetic interaction between a superconductor and a ferromagnet. Therefore, an insulating layer is placed between the superconductor and the ferromagnets so that no proximity effect is involved.

1.3.2.1 Influence of the ferromagnet on the superconductor

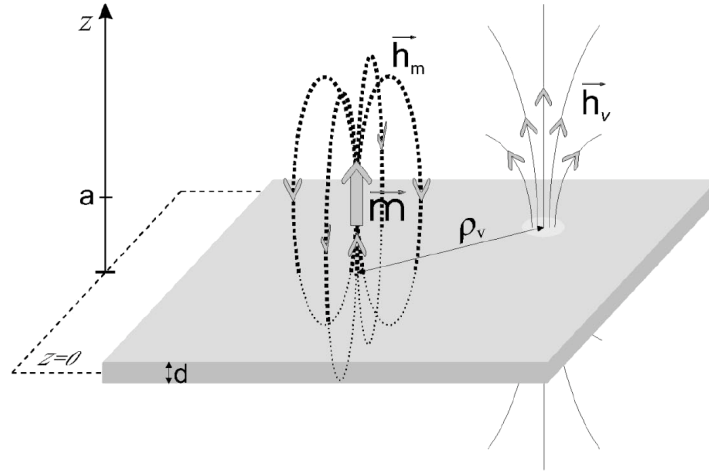


Figure 1.18: Schematic representation of a superconducting film of thickness d containing a vortex and interacting magnetically with a magnetic moment $\vec{m}$ placed at a distance a above the film and pointing upward. $\vec{h}_m$ is the magnetic field of the magnetic moment $\vec{m}$, $\vec{h}_v$ is the magnetic field of the vortex and ρ_v is the distance between the moment and the vortex in the superconducting plane.

The influence of the stray field of the ferromagnet on the superconductor is very complex and should be generally calculated from the

Ginzburg-Landau equations for each particular case.

The interaction energy between a vortex and a magnetic moment is determined by three different interaction terms : (i) a magnetic energy term U_{magn} describing the direct interaction between the magnetic field of the vortices $\vec{B}_v$ and the magnetic moments $\vec{m}$ in the ferromagnet, (ii) a kinetic energy term U_{kin} that takes into account the supercurrents which are generated in the superconductor by the stray field $\vec{B}_{stray}$ of the ferromagnet and (iii) if the stray fields are strong enough to generate a vortex-antivortex pair in the superconductor, one has to take into account the interaction of vortices created by the external field with such vortex-antivortex pairs. As will be discussed in chapter 5, in our case, the last term should not be taken into account because no signature of the presence of V-AV pairs was observed experimentally.

The first interaction energy term U_{magn} can be written as a Zeeman energy ($E = -\vec{m} \cdot \vec{B}$) but integrated on the volume of the ferromagnet :

$$U_{magn} = - \int_V \vec{M}(r) \cdot \vec{B}_v(r) dV \quad (1.54)$$

where $\vec{M}(r)$ is the magnetization of the ferromagnet and $\vec{B}_v(r)$ the local stray field of the vortex [81,82]. Therefore, this term is negative for parallel alignment of $\vec{M}$ and $\vec{H}$ and positive for anti-parallel alignment. The screening current terms also favors the parallel alignment but can not be calculated analytically.

Quantitative calculations of the interaction between a single magnetic dipole with out of plane magnetization above an infinite superconducting plane and a vortex (see figure 1.18) have been carried out within the London approximation [83], taking into account all these interaction terms. The total energy $U = U_{magn} + U_{kin}$ for the case of a parallel m_z and H was found to be :

$$U = \frac{m_z \phi_0}{2\pi\lambda^2} U_{\perp}(r/\lambda) \quad (1.55)$$

where r is the lateral distance between the dipole and the vortex and

$$U_{\perp}(r/\lambda) = - \int_0^{\infty} \frac{q}{k(k + q \coth(kd/2))} J_0(qr/\lambda) \exp(-qg) dq \quad (1.56)$$

with $k = \sqrt{1 + q^2}$, g the distance between the dipole and the top sheet of the superconductor, J_0 the Bessel function and d the thickness of the superconductor. Figure 1.19 illustrates the behavior of U as a function of r for different heights a of the dipole relative to the center of the superconducting film.

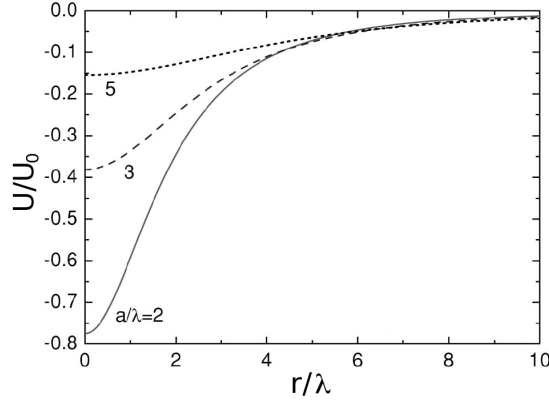


Figure 1.19: The interaction energy U of a vortex with a magnetic dipole m_z in units of $U_0 = \phi_0^2/(\pi\lambda)$ as a function of the lateral distance between vortex and dipole for different heights from the center of the superconducting film $a = d/2 + g$ (see figure 1.18). The following parameters were chosen for the calculation : $d = 1.5\lambda$ and $m_z = 10\phi_0\lambda$.

1.3.2.2 Influence of the superconductor on the ferromagnet

The stray field of the ferromagnet in close proximity with a superconductor induce screening current in the superconductor, i.e. a magnetization, which in turn interact with the stray fields of the ferromagnet. Therefore, the magnetic state of a ferromagnet-superconductor hybrid should be calculated self-consistently [84]. Recently, Tamegai et al. [85] reported a shrinkage of the magnetic domains in a superconductor-ferromagnet bilayer. However, in the case of strong ferromagnets, which is the case considering 3d transition metals such as Ni, the impact of the superconductor on the magnetic state of the ferromagnet can be neglected [81]. Indeed, the ratio between the energy associated to both phenomenon is proportional to the ratio between the Curie temperature (for ferromagnets) and the critical temperature (for superconductors), which is generally about one hundred. Therefore, ferromagnetism is generally a much stronger phenomenon than superconductivity.

Chapter 2

Materials and methods

2.1 Alumina membrane fabrication and characterization

2.1.1 Anodization of aluminum

Although the anodization of aluminum is used in the industry since 1923 as a protection from corrosion [86], it is only recently that the scientific community is aware of the potential of the electrochemically anodized aluminum to create ordered arrays of nanostructures [87]. The AAO templates consists of a triangular array of cylindrical pores as shown in figure 2.2(a). A careful tuning of the oxidation process in a suitable chemical environment allows one to obtain long range ordered triangular array of pores in Al_2O_3 (see figure 2.1(a)). The anodized aluminum is separated from the non-anodized Al by a barrier layer formed of bumps. The anodized template can be freed from the Al substrate by selective etching resulting in a template whose one face consists in holes and the other face, in bumps (see figure 2.1(b)).

The AAO templates with pore interdistance of 100nm have been fabricated by a two step anodization process in oxalic acid (0.3 M) at $2^\circ C$ under a constant voltage of 40V. A 500 μm thick ultra-pure (99.999%) Al foil is first electro-polished then partially anodized to a depth of about 50 μm to promote the ordering. This first anodized layer is then selectively removed by chemical etching with a mixture of H_3PO_4 (6 wt%) and chromic acid (1.8 wt%) at $60^\circ C$. A second anodization (20 μm) under the same conditions is then performed in order to obtain a highly ordered structure.

During the growth, due to internal stresses, the pores self-organize into a triangular lattice [90]. At the anodization front, a rounded barrier

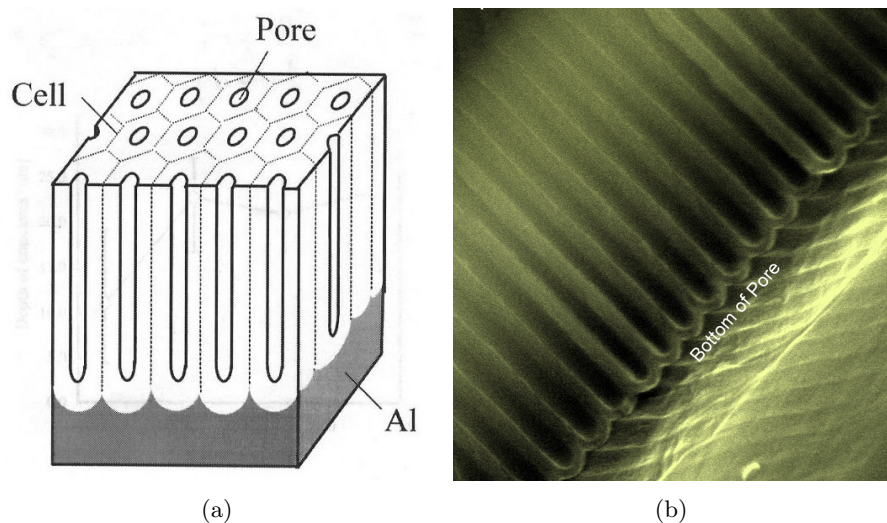


Figure 2.1: (a) Schematic representation of an anodized aluminum oxide template showing the triangular array of cylindrical channels closed at the end by the barrier layer. Taken from reference [88]. (b) SEM side-view image of an alumina template revealing the bumpy surface of the barrier layer. Taken from reference [89].

layer closes the pores. This barrier layer has a slightly different composition than the alumina template. A transversal view of an alumina template is shown in figure 2.1(b) (taken from reference [89]) after the selective removal of aluminum by chemical etching. On the upper left corner, the porous surface of the template is visible while the end of the anodization is clearly observed in the central part of the frame as the rounded barrier layer closing the pores.

2.1.2 Pores enlargement

The surface of an alumina template after the anodization at 40V is shown in figure 2.2(a). The interdistance between the pores is 100nm and the pore diameter is about 30-35nm. A pore enlargement procedure have been developed to control the porosity of the templates. The templates are dipped in sulfuric acid 0.5M at 40°C. After enlargement, the templates are rinsed in pure water. Figure 2.2(b) shows a SEM image of a template after a 80 minutes enlargement time. The resulting pore diameter is about 60nm.

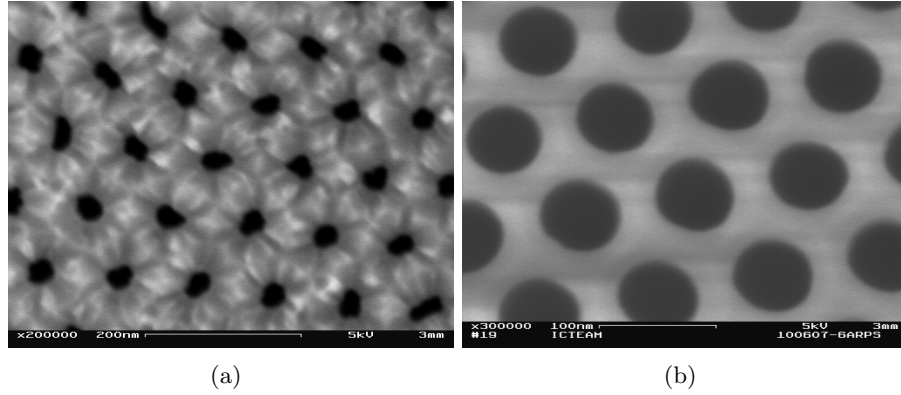


Figure 2.2: SEM image of a AAO template after anodisation at 40V, i.e. with pore interdistance of 100nm. (a) After the anodization, the pore diameter is 35nm. (b) After a 90min enlargement process, the diameter of the pores is about 60nm.

The pore diameter as a function of time in sulfuric acid is shown in figure 2.3 for the templates with initial pore of diameter about 35nm. The enlargement rate was calibrated using SEM.

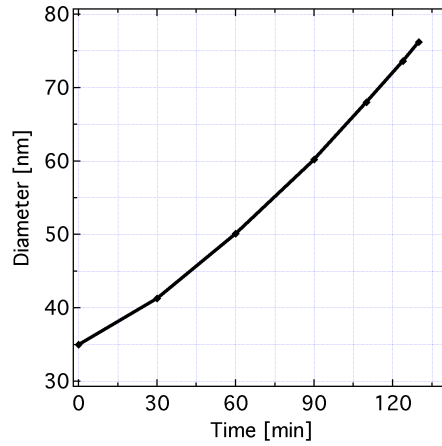


Figure 2.3: Diameter of the pores as a function of time during the enlargement procedure of a Synkera template with initial pore diameter of 35nm and pore interdistance of 100nm.

2.1.3 Alumina templates characterization

Two types of AAO templates were used in this study : commercial membranes and home-made templates. The commercial membranes are produced by Synkera Technologies, inc, they are self-suported and ty-

pically $50\mu m$ thick. The home-made templates can be of two types : thin-film supported templates (a few microns thick) or thick-film self-supported membranes. However, the long-range order only slightly varies from one type to the other because a two step anodization process has been performed in all cases.

The SEM image for sample NbN_h20(30,50) is shown in figure 2.4(a) prior to the deposition of the superconducting film and in figure 2.4(b) after the NbN deposit. A precise analysis of the pore diameter distribution was not possible because the typical dimensions of the sample are close to the spatial resolution of our SEM. However, figure 2.4(b) shows that the triangular lattice of pores transferred to the NbN film has pores slightly smaller compared to figure 2.4(a) due to the partial covering of the pores by the NbN deposit. The hole diameter in the NbN film was estimated around $D \sim 30nm$.

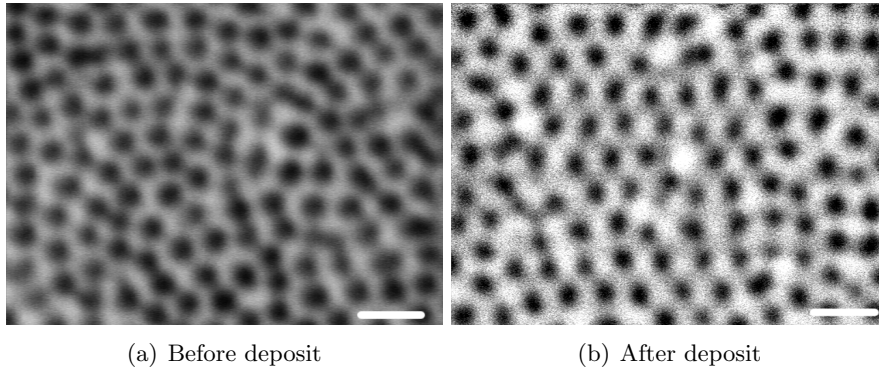


Figure 2.4: SEM image of the hole surface of an alumina template with pore interdistance $\sim 50nm$ and pore diameter $\sim 35nm$ (a) before and (b) after the deposition of a 20nm thick NbN film. The white bar is 100nm long. After the deposition (b) the hole diameter is reduced to $\sim 30nm$.

Figure 2.5(a) shows a SEM image of the barrier layer face of a AAO template with pore interdistance of 100nm. The barrier layer of the AAO template gives rise to an ordered array of bumps. As the pore array has a triangular arrangement, both holes and bumps arrays form the same triangular lattice. However, the bumps shows a hexagonal shape resulting from compression due to the presence of the neighboring bumps. The hexagonal arrangement of the interstitial sites is clearly visible. These sites correspond to narrow dips. We expect that the deposition of a thin film onto this surface will induce small regions with weakened superconductivity at the dips location.

Figure 2.5(b) shows a SEM image of the barrier layer over a large

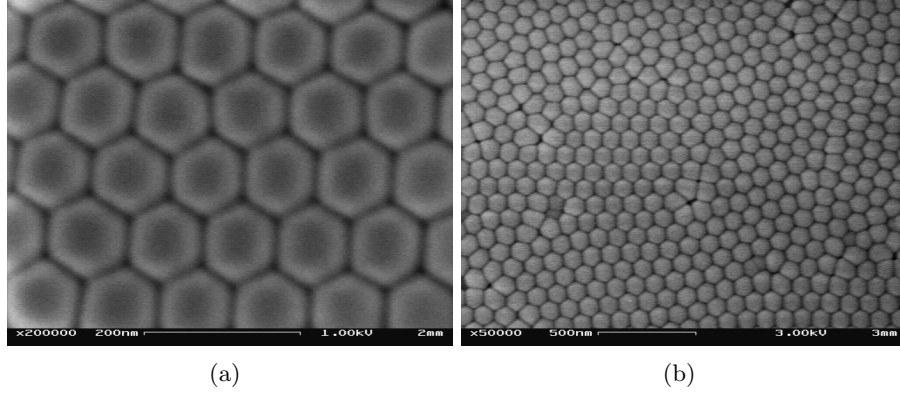


Figure 2.5: SEM images of the surface of the barrier layer of an alumina template with a triangular array of pores (interdistance 100nm). (a) Zoom on the bumps lattice revealing the hexagonal arrangement of the interstitial sites. (b) Enlarged scale showing the different defects present in the lattice.

area. Several types of defects in the triangular array of bumps are observed. First, the bumps array shows a multi-domain arrangement with a typical domain size of $1\mu m^2$ [91]. This local disorder at the domain boundary is observed resulting in the presence of bumps with a deformed shape (pentagon or heptagon instead of hexagon). Note that this barrier layer can be easily eliminated by chemical etching to produce AAO membrane with arrays of pores on both face of the AAO film.

2.2 Superconductor-ferromagnet hybrid fabrication and characterization

In this section, the different steps of the fabrication of the superconductor - ferromagnet hybrid are explained. The end of this section is devoted to the characterization of this hybrid.

Commercial nanoporous alumina templates (Synkera Technologies, Inc.) were used to fabricate our superconductor-ferromagnet hybrids. These self-supported templates are $50\mu m$ thick, have a triangular lattice of straight pores with interdistance of 100nm and a typical ordered domain size of $1\mu m^2$. After the growth of the Ni nanowires by electrodeposition, the gold cathode is removed using a [KI 100g - I_2 27g - 1000g H_2O] solution for 5-10min at room temperature, then the Cr layer is removed by CMP. Finally, the NbN thin film is deposited on the surface by sputtering.

2.2.1 Arrays of electrodeposited Ni nanowires

A thin Cr or Ti layer ($\sim 5\text{nm}$) is deposited on one face of the AAO membrane as an adhesion layer. Then, a thick Au layer of about 3-5 times the pores diameter (typically 300nm) is deposited to cover the pores. This Au layer is then used as the cathode for the electrodeposition process. The other face of the template is exposed to an electrolyte containing the Ni ions. The solution penetrates into the pores and the nanowires grow from the cathode. The electrodeposition is achieved in a conventional three electrodes cell (see figure 2.6). In addition to the cathode, Ag/AgCl reference electrode is used to control the deposition potential and the current that flows between the graphite or Pt anode and the Au cathode. The solution used for the Ni deposition is composed of 0.5M or 1M NiSO_4 and 0.4M H_3BO_3 .

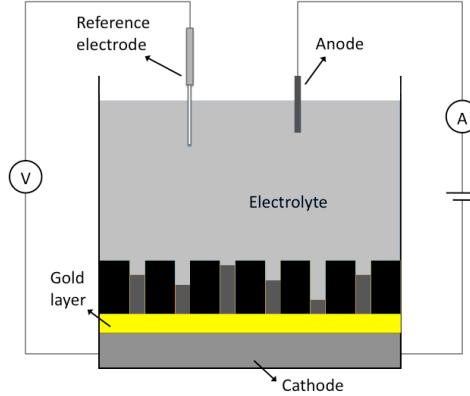
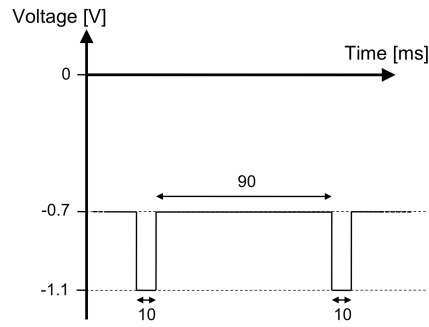


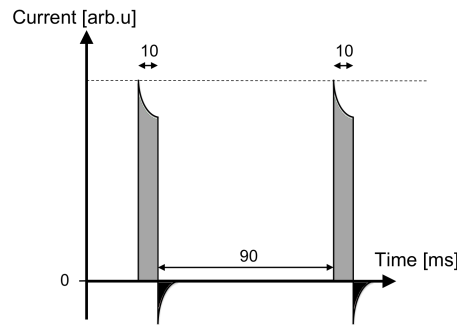
Figure 2.6: Scheme of the 3-electrode electrodeposition cell. The alumina membrane is represented in green and the Ni nanowires in orange.

In order to maximize the filling of the pores, we use the pulsed electrodeposition technique [77]. Indeed, with constant potentiostatic or galvanostatic deposition, the deposition rate can vary significantly from one pore to the other. Instead, pulsed electrodeposition allows to homogenize the deposition rate in each pores. The time dependence of the voltage and the current during our pulsed electrodeposition are represented respectively in figure 2.7(a) and 2.7(b). A voltage pulse of -1.1V is applied during 10ms to force the nucleation in each pore (figure 2.7(a)). This potential is quite high and leads to a high deposition current (figure 2.7(b)). The grey area corresponds to the deposited charge during the pulse. During this pulse, the deposition current drops rapidly due to the decrease of the Ni ions concentration near the growing nanowires. Then, the equilibrium potential (-0.7V) is applied during 90ms (figure

2.7(a)) for which the deposition current is zero (figure 2.7(b)). This potential corresponds to the equilibrium potential of the system. This period allows the ions to migrate into the pores and to homogenize the ions concentration. The gradient of concentration present during and just after the deposition leads to a small dissolution current when the equilibrium potential is applied (black area of figure 2.7(b)). This is due to the discharge of the double-layer capacity at the nanowire tips and to the higher equilibrium potential of the solution due to the lower concentration of Ni close to the nanowire tip. However, the dissolution current rapidly drops to zero as the equilibrium concentration of the solution is recovered. This potential sequence is applied thousands of times (for a total duration of Ni electrodeposition of about 16h) to grow nanowires of several microns long. The net charge deposited per cycle corresponds to the grey area minus the black area.



(a)



(b)

Figure 2.7: Schematic representation of a pulsed electrodeposition of Ni. (a) Voltage versus time (b) Current versus time. The grey area corresponds to the deposited charge and the black region to the dissolved charge.

2.2.2 Polishing

In order to reveal the nanowire tip, the cathode is removed using a chemical and mechanical polishing (CMP) procedure using the *Syton* commercial solution (half diluted in pure water) as slurry. This slurry is composed of colloidal silica particles with mean size of 20nm. The setup is schematized in figure 2.8(a). The polishing setup is a MECAPOL P230 from PRESI. The polishing discs are MECAPREX - SUPRA5 discs from PRESI. The rotation frequency is generally fixed to 5Hz and the slurry flow to about 6 ml/min.

The final step consist in a polishing with water for several minutes in order to clean the sample surface. This step is performed in order to remove the silica particles that cover the sample surface at the end of the CMP procedure as shown in figure2.8(b) (white dots).

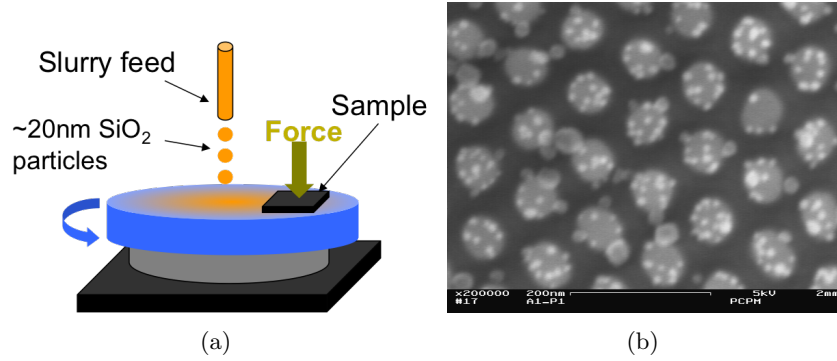


Figure 2.8: (a) Schematic representation of the CMP setup. (b) Typical sample surface without cleaning step after the CMP with *Syton*. The white dots are the Syton particles.

Also, the different polishing rates (either mechanical or chemical) of the nanowires (metallic) and of the alumina membrane lead to a pronounced surface corrugation. In fact, we found experimentally that the alumina is etched faster than the nanowires. However, the corrugation of the surface, i.e. the height difference between the nanowires tip and the substrate surface, can strongly vary from one CMP procedure to the other. This leads to reproducibility problem since the superconducting properties are strongly dependent on the surface corrugation. Therefore, the cleaning step with water is crucial because it considerably flattens the surface (corrugation is reduced to about 2nm), leading to much more reproducible thin film deposit quality.

It is also worth mentioning that the Syton particles can also be removed by sonication in pure water. Nevertheless, in this case, the

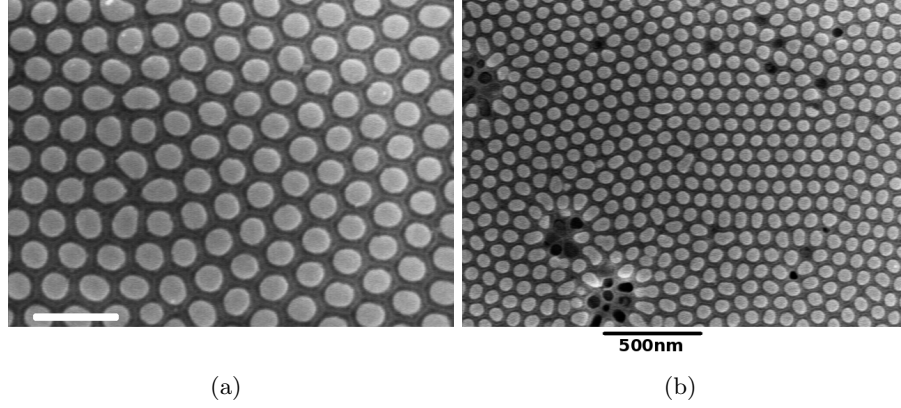


Figure 2.9: SEM image of the surface of an alumina membrane filled with Ni by electrodeposition. (a) Ordered region. The white bar is 200nm and the mean nanowires diameter is about 75nm. (b) Region showing boundary defects. Vacancies in the Ni nanowire array and deformation of the triangular arrangement are observed as well as shape and size modifications of the nanowires.

corrugation of the surface persists.

2.2.3 SEM characterization of the filled AAO surface

A SEM image of the surface of the template obtained after the cleaning step is shown on figure 2.9(a) for a sample with pore diameter of 75nm. This image is representative of all samples with wide pores. Indeed, an almost complete ($> 99\%$) pore filling is obtained for enlarged pore diameter larger than 50nm. The triangular array of nanowires is ordered over area of about $1\mu m^2$. These domains are separated by thin regions (similar to grain boundaries in polycrystals) in which the triangular arrangement is not maintained. Also, at these boundaries, the shape of the nanowires can be significantly deformed.

A larger region of the filled membrane with an enhanced concentration of defects in the triangular array of Ni nanowires is shown in figure 2.9(b). Two types of defects are present in the nanowire array :

1. Periodicity defects

The periodicity defects have two origins. First, at the boundary of two ordered domains with different spatial orientations, the triangular lattice of pores is strongly deformed in order to match both orientations. Second, there is a small fraction of empty pores or vacancies in the nanowire array. These vacancies (less than 1% of the pores) are mainly concentrated at the boundaries of the ordered domain. These vacancies are shown in figure 2.9(b).

2. Shape defects

The shape defects refer to the non uniform nanowire shape and size within the sample. Indeed, figure 2.9(a) and 2.9(b) clearly show that the nanowires are not perfectly circular and that their diameter may differ. The main shape deformations are mostly concentrated at the grain boundaries.

Therefore, our sample can be considered as a polycrystalline triangular array of Ni nanowires with defects concentrated at the grain boundaries. However, due to their low concentration, these defects do not significantly impact on the vortex pinning properties as will be discussed in chapter 4 and 5.

2.2.4 AFM characterization of the filled AAO surface

Atomic force microscopy was developed by Gerd Binnig and Heinrich Rohrer in 1986 [92]. A schematic representation of an AFM setup is shown in figure 2.10. A tip fixed on a cantilever interacts with the surface (Van der Waals forces,...) and the cantilever is bent. The curvature of the cantilever is detected by a laser which is reflected on it and sent to a photodetector. The interaction force can be directly related to the deflection of the cantilever.

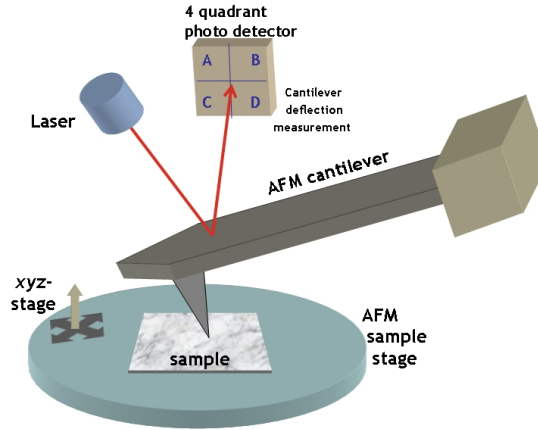


Figure 2.10: Scheme of a AFM setup. Taken from reference [93].

Topography images can also be obtained in dynamic mode (which is the case for our samples). In this case, the tip is placed at a certain distance from the surface where it interacts with it. The cantilever vibrates at its resonant frequency and the height of the sample or of the cantilever is adjusted while scanning the surface in order to maintain the same resonant frequency.

AFM measurements¹ have been performed in order to characterize the topography of the surface of our samples prior to NbN film deposition. Figure 2.11(a) shows a topographic image of the surface of sample NbNNW25(75,100) after the evaporation of 10nm of Al_2O_3 as an insulating layer. In this case, only a CMP was performed, followed by a sonication in pure water for the Syton removal. The topography along the white line of figure 2.11(a) is shown in figure 2.11(b) (black curve). An important corrugation is observed with peak to peak height differences of about 15nm. However, the real surface corrugation is probably more important because the AFM tip cannot perfectly reproduce the interwire region. As will be thoroughly discussed in chapters 4 and 5, the surface corrugation plays a key role in the magnetic interaction between the superconducting film and the Ni nanowire array. However, the corrugation strongly vary from one CMP to the other, which makes difficult the comparison between the samples. Therefore, an alternative process was developed to control the surface corrugation of our samples. Indeed, figure 2.11(b) (red curve) shows that a relatively flat surface (corrugation $\sim 2 - 3nm$) can be obtained when the CMP is followed by a polishing step with water (for 5 to 10 minutes). This additional polishing step flattens the surface. The resulting flat surface is much more reproducible than the one obtained after the CMP and the ultrasonic cleaning. In order to study the influence of the surface topography on the magnetic interaction between the superconductor and the Ni nanowires array, the surface topography was artificially modified in a controlled manner. After the flattening of the surface (i.e. polishing with water), a selective chemical etch (using 0.2M NaOH solution) of the alumina was performed in order to increase the corrugation. In these conditions, the corrugation was controlled by adjusting the etching time from 0 to 5 minutes.

A SEM image of a sample with Ni nanowires (50nm of diameter and 100nm of interdistance) with a strong relief is shown in figure 2.11(c). This image was obtained after polishing with *Syton* and cleaning in ultrasounds. The SEM image reveals an additional relief on the alumina template. Indeed, the interwire region of the alumina template shows brightness modulation. SEM contrast is known to originate mainly from the topography of the surface [94]. Therefore, the darker regions (around the nanowires) are dips in the alumina while the bright lines forming a honeycomb array (in the central part of the interwire region) are maxima. This is due to the fact that the anodization process of alu-

1. Performed by B. Nysten of the Institute of Condensed Matter and Nanosciences of UCL Belgium

minum leads to inhomogeneous chemical and structural composition of the alumina template. The template is known to be less chemically and mechanically resistant in the region surrounding the pores [87]. Therefore, not only the nanowire tips are above the alumina surface but also the latter is not flat in-between the nanowires. A schematic representation of a transversal view of the alumina template is shown in figure 2.11(d) prior to NbN deposition and in figure 2.11(e) after NbN deposition. It is clear that the combination of a pronounced surface relief and thin film deposition leads to the perforation of the NbN deposit by the Ni nanowires. In this limit, the superconducting region of interest is represented in figure 2.11(f) as the green - white hatched region forming an hexagonal array of NbN strands. The hexagonal unit cell of the array is represented in red. In this case, the superconducting behavior is expected to be different from the one of a continuous film close to the Ni nanowires array for two reasons : first, the superconducting film contains antidots array and second, the magnetic field modulation for perforated films is much more pronounced than if the superconducting film lies on top of the nanowire array (as will be quantitatively evaluated in section 4.3).

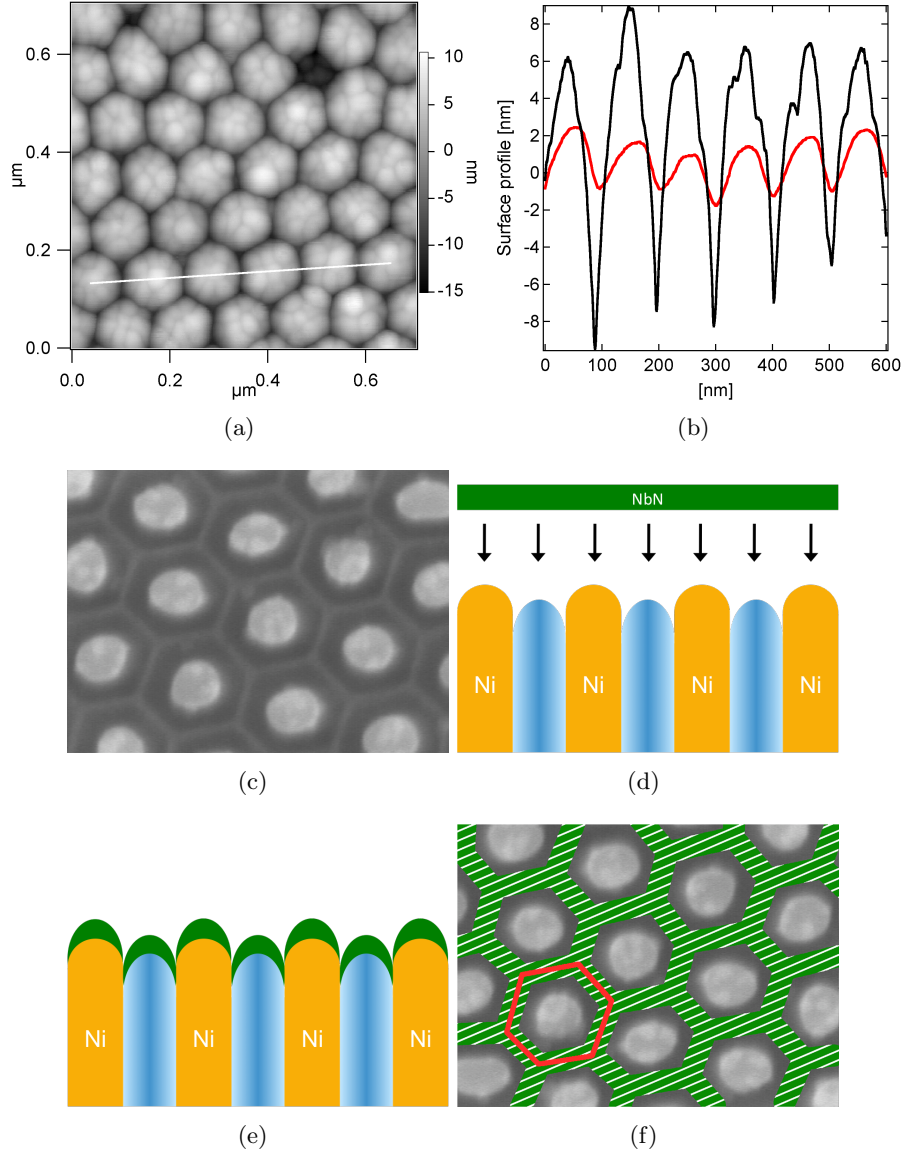


Figure 2.11: (a) Topographic AFM image of the surface of a sample with Ni nanowire with diameter 75nm and interdistance 100nm (see figure 2.9(a)) after the e-beam deposition of 10nm of Al_2O_3 as insulating layer. (b) Surface profile along the white line of figure 2.11(a) revealing a peak-to-peak corrugation of at least 15nm. The black and red curves correspond respectively to the surface corrugation before and after the polishing with water. (c) SEM image of the surface of a AAO membrane filled with Ni nanowires (50nm in diameter) just after the polishing with *Syton* and cleaning. (d) Schematic representation of the surface profile of figure 2.11(c) (transversal view). (e) Schematic representation of the deposit on the rough surface of figure 2.11(d) (transversal view). For sufficiently large corrugation, the deposit on the Ni nanowires is not in contact with the one in-between the nanowires, i.e. at the surface of the AAO. (f) Schematic representation of the conductive region of the NbN deposit on the rough surface (green - white hatched region) of figure 2.11(c) and 2.11(d). The red hexagon represents the unit cell of the array. AFM measurements were performed by B. Nysten of the Institute of Condensed Matter and Nanosciences of UCL Belgium

2.3 Thin films growth methods

We used reactive sputtering to grow the NbN thin films. The deposition of the Nb films were performed by molecular beam epitaxy at the KULeuven (Victor Moshchalkov team). The Al_2O_3 insulating layers were deposited either by direct evaporation or deposition of Al (evaporation or sputtering) and oxidation in air. In each case, the thin films are deposited through 4-point contact Ni mask. The measured bar is 3.2mm long and 0.2mm wide.

2.3.1 Cathodic sputtering

Cathodic sputtering have been performed with a commercial Plassys. The material is extracted from a target using a DC Ar plasma. This plasma is created by applying a high voltage between the sample and the target at room temperature. Nitrogen is also present in the plasma and reacts with the Nb atoms that are ejected from the target. NbN is formed and is deposited on the substrate. The partial pressures are 8 mTorr for Ar and 0.9mTorr for N_2 . A current of 1A is imposed leading to a voltage of 370V. The target is 4 inches in diameter and is 8 cm from the surface of the sample.

The NbN film deposition is performed by reactive DC-sputtering at room temperature. The structural properties of the NbN thin film strongly depends on the surface on which the NbN is grown (for details see reference [95] and references therein). Figure 2.12(a) shows a SEM image of a 25nm thick NbN deposited on a sapphire substrate and (b) on the surface of an AAO membrane filled with Ni nanowires with diameter 75nm and interdistance 100nm (shown in figure 2.9(a)). The strong granularity of both films is clearly observed. Moreover, as will be discussed in the next chapters, the structural composition of the deposit is affected by the difference of substrates between the reference sample and the nanostructured sample.

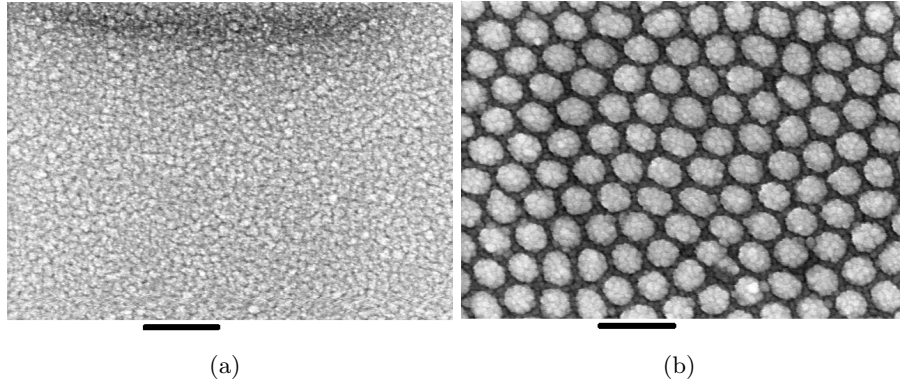


Figure 2.12: SEM image of a 25nm thick deposit of NbN on (a) a sapphire substrate and (b) on the surface of an AAO membrane filled with Ni nanowires with diameter 75nm and interdistance 100nm (see figures 2.9(a), 2.9(b) and 2.11(a)). The black bars are 200nm long.

2.3.2 Evaporation

Evaporation was performed with a commercial Pfeiffer500. Samples are stucked on a plate placed about 20cm above a crucible containing the material to deposit. The crucible is heated by an electron beam which intensity and energy can be easily controlled. The electron beam can be directed by a local magnetic field. A high vacuum (down to 10^{-7} mbar) is maintained in the chamber in order to avoid oxidation of the material and to limit the inclusion of impurities during the film growth. Due to the low pressure, the deposition is directional. The amount of material deposited on the sample is determined by a crystal vibrating at its resonant frequency. The resonant frequency is modified when the material is deposited on its surface. Knowing the acoustic impedance and the density of the material, the change of resonant frequency can be converted to a deposition rate.

2.3.3 Molecular beam epitaxy

Molecular beam epitaxy (MBE) is one method used to deposit single-crystals. The most important characteristic of MBE is the slow deposition rate (typically less than $1\mu\text{m}$ per hour) which allows the films to grow epitaxially. Therefore, the pressure in MBE chamber should be lower (typically 10^{-8} Pa) than other deposition techniques (evaporation,...) in order to introduce the same amount of defects in the deposit. Materials to be deposited are heated in separate quasi-Knudsen effusion cells, sublimates and condenses on the substrate. During operation,

reflection high energy electron diffraction (RHEED) is often used for monitoring the growth of the crystal layers. Thin films of Nb were deposited using molecular beam epitaxy (MBE) at room temperature, at a working pressure of 10^{-10} torr and evaporation rate of $1.5\text{\AA}s^{-1}$.

2.4 Measurement of the electrical and magnetic properties

After or during preparation, the samples have been characterized to confirm their structural quality and physical properties. Magnetic force microscopy was used to probe the local magnetic state of the Ni nanowires array while a Superconducting Quantum Interference Device (SQUID) magnetometer was used to determine the magnetic hysteresis loop of the array. The superconducting properties of the superconducting-ferromagnets hybrids were studied by electrical transport measurements.

2.4.1 Magnetic measurements

2.4.1.1 Magnetic force microscopy

The principle of the magnetic force microscopy is similar to the AFM technique excepted that the tip is either in ferromagnetic material or covered of ferromagnetic material (evaporation,...). Therefore, in addition to the Van der Waals interaction of the tip and the surface, magnetic interaction can also occur if the surface is magnetic. Therefore, a contrast can be obtained depending on the relative orientation of the magnetization of the tip and the local magnetization of the surface. The MFM measurement presented in this thesis have been performed by Werner Gillijns in K.U. Leuven (Victor Moshchalkov team).

2.4.1.2 SQUID magnetometry

Two different SQUID magnetometers were used to measure the hysteresis loops of the ferromagnetic nanowire arrays. Both are commercial *Magnetic Properties Measurement System (MPMS)* from Quantum Design. The SQUID contains an He cryostat and can operate from 1.9K to 400K with a stability of 0.01K at low temperatures. The sample is placed into a drinking straw with cotton-wool plugs. These materials are non-magnetic so their influence is negligible. The straw is attached to a 1m long rod which is brought into the cryostat. To determine the magnetic moment of the sample, it is displaced through a second-order gradiometer (see figure 2.13(a)). This gradiometer consists of three coils

separated by about 1.52cm. The central coil has two turns in counter-clockwise windings and the upper and lower coils have a single turn in clockwise winding. The gradiometer configuration is used to reduce the noise in the detection circuit caused by fluctuations of the large magnetic field created by a superconducting magnet. It also provides an easy way to center the sample in the center of the gradiometer. One SQUID was performing DC measurement while the other was performing AC measurements. In both cases, the sample is moved in the gradiometer inducing a voltage which varies as a function of the position of the sample. The typical shape of the output signal of the SQUID is shown in figure 2.13(b) in the case of a negative moment. Magnetic field uniformity on a 4cm scan is about 0.01% and the maximum sample size is 9mm. The maximum magnetic field is 5T for the DC SQUID and 5.5T for the AC SQUID. Further details can be found on the Quantum Design website [96].

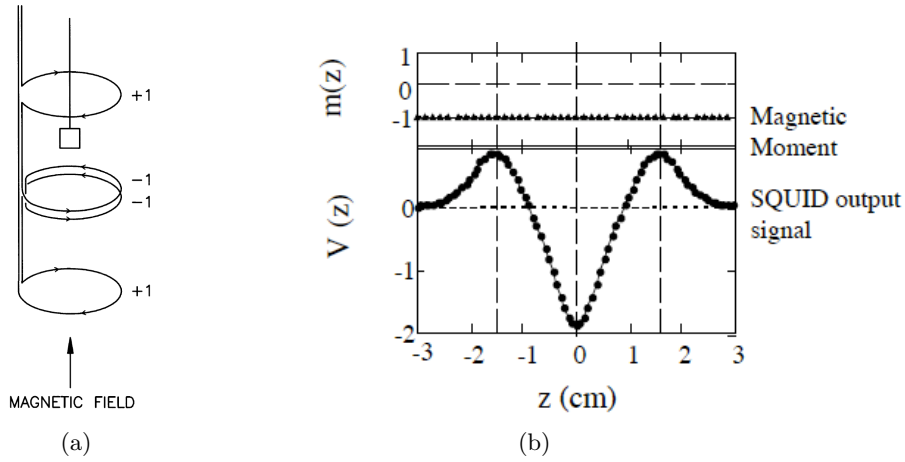


Figure 2.13: (a) Scheme of the second-order gradiometer used in our SQUID. (b) SQUID output signal for a point-like negative magnetic dipole as a function of its position in the gradiometer. Taken from reference [97].

2.4.2 Electric transport measurements

The electric transport measurements are carried out in an home-made ^4He cryostat. The sample is placed on the measuring stick and sealed in a pot with Wood's alloy low temperature welding. The pot is immersed in the liquid He and the temperature of the sample is regulated by a resistance placed nearby the sample and connected to a temperature controller (Lakeshore 340). A low He pressure is maintained in the

pot in order to reduce the temperature gradients. The pot is placed at the center of a 5T superconducting solenoid running in driving mode and controlled by an *intelligent superconducting magnet power supply* (Oxford IPS120-10). A 4-point contact measurement is performed. The current source is a Keithley 224 and the voltage is measured with a Keithley 2182A nanovoltmeter with a resolution of a few tenth of nanovolts.

2.5 Sample label

Throughout this manuscript, the different samples will be labeled using the expression shown in figure 2.14.

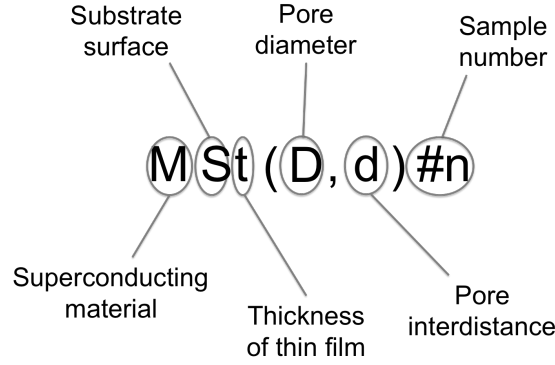


Figure 2.14: Signification of the sample label

M refers to the superconducting material which can be either Nb or NbN.

S is the surface of the substrate on which the superconducting film is deposited, i.e. h = hole surface, b = bump surface and NW = Ni nanowires embedded in AAO template.

t refers to the thickness of the superconducting film.

D refers to the diameter of the holes or nanowires.

d is the pore interdistance.

$\#n$ refers to the number of the sample if several samples have the same characteristics.

Moreover, for the Nb deposition by MBE, the angle is specified respectively by $\perp$ and 30° for perpendicular and angular deposition.

Naturally, in the case of sample prepared using bump array, the pore diameter is not relevant. For example, NbNh20(60,100) corresponds to a 20nm thick NbN film deposited on the porous surface of an alumina template with hole array of 60nm in diameter and 100nm interdistance.

Table 2.1 resumes the characteristics of the different samples studied in this thesis. Each sample has its reference sample simultaneously deposited on a sapphire substrate. These substrates were used because they have a similar composition to the AAO templates and because the sapphire displays a good surface for the growth of NbN [95]. These substrates have a very low surface roughness ($< 2\text{\AA}$).

Table 2.1: Sample label and characteristics.

Label	SC film	Substrate periodic surface	Film thickness [nm]	Pore/wire diameter [nm]	Pore interdistance [nm]	Sample number	T_c [T_c^{ref}] [K]
NbN _h 20(60,100)	NbN	holes	20	60	100	-	8.3 [-]
NbN _h 20(30,50)	NbN	holes	20	35	50	-	7.6 [9.1]
NbNb ₂₅ (-,100)#1	NbN	bumps	25	-	100	1	9 [-]
NbNb+i25(-,100)	NbN	bumps + insulator (25nm)	25	-	100	-	8.05 [10.8]
NbNb10(-,100)	NbN	bumps	10	-	100	-	7.4 [8.7]
NbNb20(-,100)	NbN	bumps	20	-	100	-	8.15 [9]
NbNb25(-,100)#2	NbN	bumps	25	-	100	2	9.37 [10]
NbNb30(-,100)	NbN	bumps	30	-	100	-	9.78 [10.1]

Table 2.2: Rest of table 2.1

Label	SC film	Substrate periodic surface	Film thickness [nm]	Pore/wire diameter [nm]	Pore interdistance [nm]	Sample number	T_c [T_c^{ref}] [K]
Nbb30($\perp$)(-,100)	Nb	bumps	30($\perp$)	-	100	-	5.4 [-]
Nbb30(30°)(-,100)	Nb	bumps	30(30°)	-	100	-	5.2 [-]
Nbb50($\perp$)(-,100)	Nb	bumps	50($\perp$)	-	100	-	6 [-]
Nbb50(30°)(-,100)	Nb	bumps	50(30°)	-	100	-	5.9 [-]

NbNNW25(75,100)	NbN	Ni nanowires	25	75	100	-	9.57 [11]
NbNNW25(60,100)	NbN	Ni nanowires	25	60	100	-	9.5 [10.5]
NbNNW20(60,100)#1	NbN	Ni nanowires	20	60	100	1	9.1[10.1]
NbNNW20(70,100)	NbN	Ni nanowires	20	70	100	-	9.5 [10]
NbNNW20(50,100)	NbN	Ni nanowires	20	50	100	-	9.25 [10]
NbNNW20(40,100)	NbN	Ni nanowires	20	40	100	-	8.7 [8.1]
NbNNW20(60,100)#2	NbN	Ni nanowires	20	60	100	2	9.25 [9.75]

Chapter 3

Core pinning of vortices in thin superconducting films using alumina templates

3.1 Introduction

3.1.1 Context

The nanostructuration of superconducting thin films has known a growing interest among the scientific community due to the possibility to tailor the superconducting properties of materials. The strong improvement of the lithographic techniques is a key factor for this booming. By locally modifying the superconductor, one can for example create vortex pinning centers. Indeed, a rich variety of vortices pinning centers arrays have been created this last decade in order to study the interaction of the vortex lattice with the pinning centers arrays. Particular attention have been given to the commensurate states or so-called matching effects of the vortex lattice with the pinning center lattice [3, 12, 98–100]. In this framework, visualization techniques like bitter decoration [3, 101–103], scanning SQUID microscopy [104], Lorentz force microscopy [105], neutron diffraction [106] and more recently MFM measurements [107] have revealed a rich diversity of commensurate states and have brought key informations about the vortex motion in periodic lattice of pinning centers. The influence of the size and the shape of the pinning centers as well as the interdistance and the symmetry of the pinning center lattice have been thoroughly studied. In particular, guided vortex motion (or the so-called vortex ratchet) was achieved by taking advantage of the shape of the pinning centers [14, 40, 108]. Also many different kinds of pinning

centers were investigated : antidot [2, 5, 6, 12], blind hole [3], structural defect [1, 38] and various magnetic pinning centers [9, 100, 109–111].

Nevertheless, lithography based pinning arrays have technological limitations on the size and the interdistance between the pinning centers and moreover, these techniques are time-consuming and expensive (the total surface of the lattice is limited). In addition, the majority of previous works used "clean" superconductors like Al or Pb despite their limited potential for applications. Therefore, new approaches based on self-assembly techniques, have been developed in order to create pinning centers arrays with very high densities and larger extend [12, 13, 112]. One of these approaches is based on polystyrene sphere array and allowed to create dense honeycomb pinning center arrays in Nb thin films. Another approach concerns the anodized aluminum oxide porous membranes and templates. Indeed, these membranes contain a triangular array of straight pores with an interdistance which can range from few tenths of nm to about $500nm$ and is determined by the processing conditions. Welp et al. [12, 113] have first investigated the possibility to use these templates to produce Nb thin films with a triangular array of antidot with $100nm$ of interdistance. Later, Vinckx et al. [114] investigated the limits of this approach using a template with pores with $50nm$ of interdistance. Recently, Villegas et al. used such alumina membrane as a mask for ion bombardment of high- T_c thin films [37].

3.1.2 Motivation

In this chapter, an extensive study of the different possibilities that offers the AAO templates to create very dense periodic pinning centers lattices is achieved. Indeed, in addition to the approaches of Welp et al. [12, 113] and Villegas et al. [14] that were limited to the triangular lattice of pores, we demonstrate the feasibility of two new approaches using the barrier layer of the alumina template to create periodic pinning arrays.

This chapter is divided in three major sections. In the first section, the vortex pinning properties of thin NbN films grown on the holes side of the membrane are studied. We demonstrate how the previous results can be significantly improved by correctly tuning the superconducting film thickness and the hole diameter. In particular, the case of the lattice of holes with interdistance of $50nm$ is thoroughly studied. We demonstrate that despite the short range order of the lattice, several strong matching effect can be obtained.

In the second section, the vortex pinning properties of thin NbN films deposited on the barrier layer of AAO templates are investigated.

We demonstrate that this new approach generates an hexagonal array of pinning centers in the NbN film and that the pinning centers density is twice the one of a triangular lattice of pores. The evolution of the pinning properties with the pinning strength is studied by varying the film thickness and by the prior deposition of an insulating film to flatten the surface.

Finally, in the third section, the case of the MBE deposition of Nb on the barrier layer is discussed. The pinning properties are investigated for different angles of deposition and thicknesses of the deposit. We argue that the angular deposition decomposes the hexagonal lattice into two triangular lattices of pinning centers.

3.2 Vortex pinning in NbN thin films by a triangular lattice of antidots

3.2.1 Motivation

In this section, we report on the superconducting properties of NbN thin films with thickness spatially modulated by the hole surface of a alumina membrane. Indeed, by depositing the NbN on the porous surface, the triangular array of holes is transferred to the superconducting film (figure 3.1). This technique has already been used by Welp et al. [12,113] and by Vinckx et al. [114,115] in order to modify the superconducting properties of Nb thin films. In this section, we demonstrate that with the appropriate tuning of both the superconductor thickness and the diameter of the holes, much stronger vortex pinning can be achieved, leading to high field matching effects.

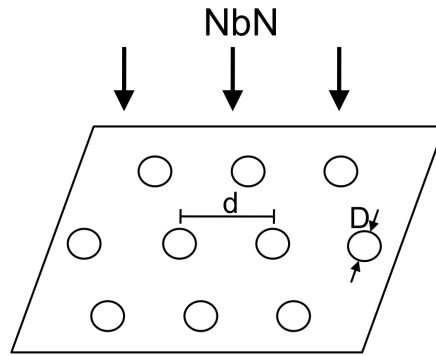


Figure 3.1: Schematic representation of the NbN deposition on the porous face of a alumina template with pore interdistance d and pore diameter D .

First, the case of the triangular array of hole with $d=100\text{nm}$ (hole

interdistance) is briefly discussed and will be considered as a reference for the next chapters. Then, the case of a triangular array of holes with $d=50\text{nm}$ (hole interdistance) is considered. The preliminary study of Vinckx et al. [115] is extended to the case of thin NbN films and enlarged pores. Using this approach, the vortex pinning properties are strongly improved.

3.2.2 Antidot lattice with 100nm interdistance

Figure 3.2 shows the superconducting transition (resistance versus temperature curve) for sample NbNh20(60,100) (solid curve) and for its reference sample (dashed curve). The presence of the antidot array strongly modifies the electrical properties of the film. A strong increase of the normal state resistance, a decrease of the critical temperature and an enlargement of the transition are observed. The strong increase of the normal state resistance is due to the structural modification of the deposit and to the reduced section for the current flow. On the one hand, the quality of thin NbN films is highly dependent on both crystallographic arrangement of the substrate and its roughness [95, 116, 117]. On the other hand, due to the presence of the holes, the current flows in $\sim 40\text{nm}$ wide interconnected channels (between the pores). Using simple geometrical considerations, we estimated that the increase of resistance due to the 32% porosity amounts to about 2.5.

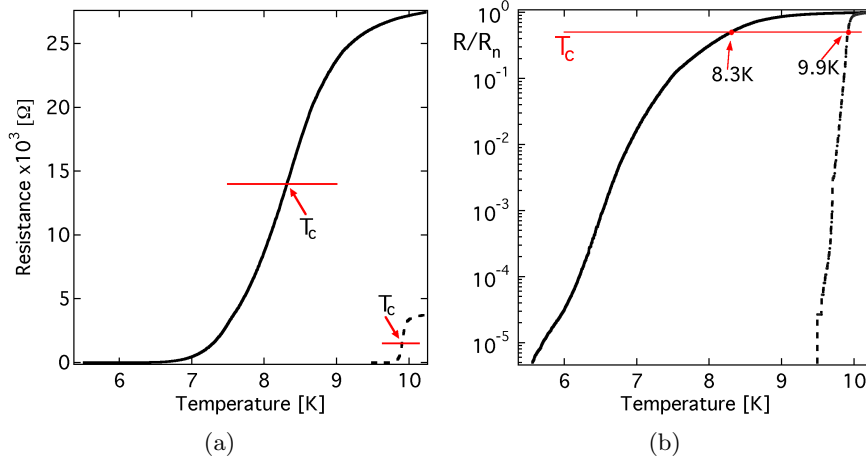


Figure 3.2: Resistance versus temperature at $H=0$ for sample NbNh20(60,100) (black solid curve) and for its reference sample (dashed curve). (a) Linear scale. (b) Normalized curves R/R_n (R_n is the normal state resistance) and logarithmic scale on the ordinate. The critical temperatures (T_c), chosen as the mid transition temperature are indicated by the arrows.

The reduction of the critical temperature is attributed to the lower structural quality of the NbN thin film due to the nanostructuring and to the larger roughness of the substrate [118, 119]. The large transition is associated to the percolative behavior of the nanostructured film [120] which is enhanced by the high granularity of the NbN films [121]. Indeed, the inter-hole channel width is varying as well as their structural quality which results in a distribution of their critical temperatures.

To maximize the number and magnitude of the matching effects, we used long-range ordered alumina membranes (ordered domain size of about $1\mu m^2$) and the pores were enlarged to increase the pinning force. The magnetoresistance curve for sample NbNh20(60,100) at $t = 0.84$ ($I_{DC} = 1\mu A$) is shown in figure 3.3(a). Deep resistance dips are observed at periodic magnetic fields, which is the signature of the matching between the triangular lattice of vortices and the triangular lattice of antidots. Indeed, the first matching effect appears at $H_1 = 0.236T$ in very good agreement with the theoretically expected value $H_1 = 2\phi_0/\sqrt{3}d^2 = 0.239T$ for a triangular array with $d = 100nm$ (see subsection 1.1.4.2, page 1.1.4.2). The number of peaks in the $R(H)$ curves as well as the matching intensities are much larger than what have been previously observed for similar measurements [12, 113, 115]. In the work of Vinckx et al. [115], the matching features were much less pronounced. A peak was only observed at the first matching field while the following matching effects appear as shoulder-like behavior (see the magnetization curve in figure 3.3(b)). The more pronounced matching effects in our case are due to the enlarged holes and the long-range order of our membranes. Indeed, bigger holes allow to pin multi-quanta vortices and therefore the appearance of interstitial vortices, which strongly weaken the superconducting properties, is postponed [4, 5]. The larger vortex-vortex interaction in NbN compared to Nb can also account for larger matching effects.

As such samples have already been intensively studied [12, 113, 115], we will not further discuss the results hereafter. They will rather be used as a reference for the other systems presented in the next chapters.

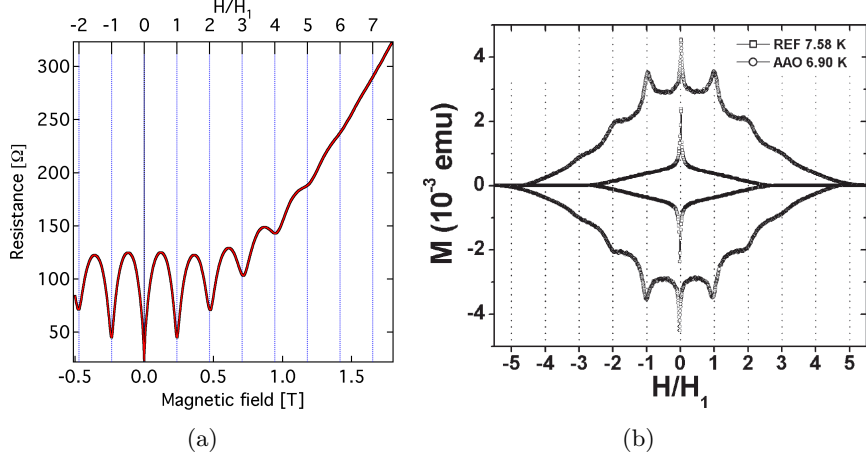


Figure 3.3: (a) Magnetoresistance curve for sample NbNh20(60,100) at $t = 0.84$ (slightly above the zero-field onset of resistance) with $I_{DC} = 1\mu A$. (b) Magnetization curve at $t = 0.85$ for a 50nm thick Nb film deposited on a AAO templates ($d=100\text{nm}$ and $D=40\text{nm}$) measured by Vinckx et al. [115].

3.2.3 Antidot lattice with 50nm interdistance

In this subsection, we discuss the superconducting properties of sample NbNh20(30,50), i.e. a NbN thin film with a triangular array of antidots with diameter of 30 nm and pore interdistance of 50 nm. This interdistance is, up to now, the smallest interdistance that can be achieved by the anodization of aluminum while maintaining a sufficient short-range order.

3.2.3.1 Phase diagram

The critical temperatures as a function of magnetic field, i.e. the phase diagram of superconductivity, for sample NbNh20(30,50) (solid curves) and its reference (dashed curves) are shown in figure 3.4(a) considering different resistive criteria. These curves have been extracted from a series of resistance versus temperature curves at different magnetic fields measured with a dc current of $10\mu A$, by taking the temperature at which the resistance of the sample is equal to the resistive criterion. The use of different criteria allows us to probe different superconducting phenomena such as nucleation of superconductivity (high resistance) or vortex motion (low resistance). The blue curves corresponds to the onset of resistance $R_c = 0.01R_n$ (R_n is the normal state resistance), the green curves corresponds to a mid-transition criterion and the black curves to the nucleation of superconductivity $R_c = 0.99R_n$.

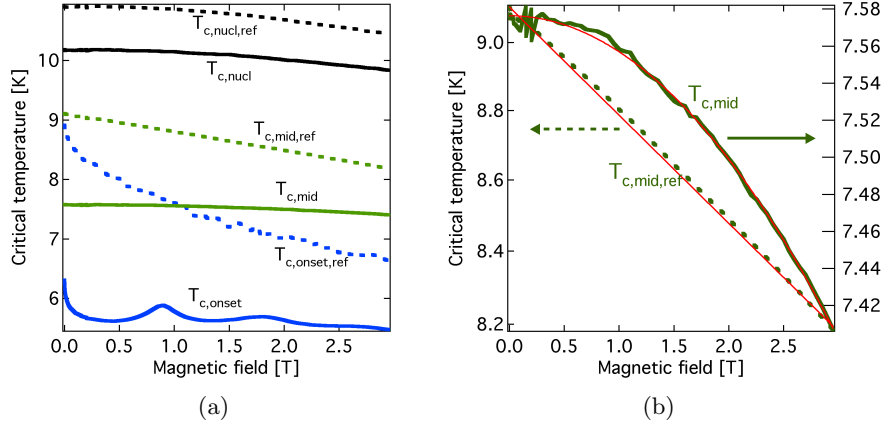


Figure 3.4: (a) Critical temperature versus magnetic field for sample NbNh20(30,50) (solid curves) and for its reference (dashed curve) for different resistive criteria. The blue curves ($T_{c,onset}$ and $T_{c,onset}^{ref}$) corresponds to the onset of resistance $R_c = 0.01R_n$ (R_n is the normal state resistance), the green curves ($T_{c,mid}$ and $T_{c,mid}^{ref}$) corresponds to a mid-transition criterion ($R_c = 0.5R_n$ - figure 3.4(b)) and the black curves ($T_{c,nuc}$ and $T_{c,nuc}^{ref}$) to the nucleation of superconductivity $R_c = 0.99R_n$. These curves have been extracted from a set of resistance vs temperature curves at different magnetic fields measured with a dc current of $10\mu A$. (b) Critical temperature versus magnetic field for sample NbNh20(30,50) (green curve - right axis) and for its reference (green dotted curve - left axis) extracted from a mid-transition criterion. The red curves represent fit of the experimental data with both the 1D (quadratic) and bulk (linear) model.

For the reference sample, different magnetic field dependence of the critical temperature are observed for the different criteria. Indeed, very different phenomena are probed when looking to the appearance of resistance or to the appearance of the superconducting state. When using a high resistive criterion such as $R_c = 0.99R_n$ (black curves), a quadratic behavior is observed. This is due to the localized nucleation of superconductivity within the NbN grains. Since the NbN grains are smaller than the coherence length in a certain temperature range ($\xi \rightarrow \infty$), the grains behave as 1D superconductors. A quadratic dependence of the critical temperature on the magnetic field is thus expected (equation 1.39 (page 29)). The mid-transition critical temperature (green curves) is much lower than the one of the nucleation. This reflects the high granularity of the NbN thin films deposited with our setup [95]. Indeed, due to the slight structural and/or chemical heterogeneities of the different grains, their critical temperature will be slightly different and the superconducting transition follows a percolative behavior [120]. The bulk behavior is observed for the mid transition criterion ($T_{c,mid}^{ref}$). Therefore, the super-

conducting regions are larger than the coherence length. However, the granular film did not yet reached the point of percolation. Only a slight decrease of the temperature is necessary to reach this point. Indeed, at zero field, the critical temperatures corresponding to the appearance of resistance and to the mid-transition are close to each other which denotes a sharp transition for low resistive states. This transition broadens quite fast when a magnetic field is applied due to the appearance of vortex motion.

In the case of sample NbNh20(30,50), the quadratic behavior (typical of 1D limit) is observed for the three criteria. Indeed, the superconducting channels are very thin, therefore the coherence length is larger than the strip width over a wide temperature range. The zero field superconducting transition ($T_{c,nucl} - T_{c,onset}$) is very large. This large transition is due to the distribution of the T_c of individual grains leading to a distribution of the T_c of the 1D channels. As a result, a percolative behavior is obtained and the zero resistance state is reached when the superconductivity has percolated in the network so that the superconducting current can flow across the sample. The $T_{c,onset}(H)$ curve (blue solid curve) shows periodic oscillations with peaks located at $H_n = n \cdot 0.9T$ (with n an integer). These peaks corresponds to Little-Parks like oscillations due to the fluxoid quantization effect in the one-dimensional wire network (1DWN) [65]. Indeed, the overall trend of the curve seems to show a quadratic behavior (but less marked than for higher resistance criteria). T_c oscillations rapidly vanish due to the disorder of the network. The critical temperature oscillations of 1DWN with positional disorder have been theoretically investigated by Benz et al. [122]. For perfectly ordered arrays, the oscillations shows a constant amplitude. The introduction of positional disorder of the nodes of the network produces a decrease of the amplitude of the oscillations with increasing magnetic field due to frustration. Also, the amplitude of these oscillations strongly decreases when increasing the resistance criterion. Indeed, at $T_{c,mid}$ no oscillations are visible. We argue that the increase of the resistance corresponds to the decrease of the number of superconducting channels in the network. Therefore, the disorder (and thus the frustration) is increased by the removal of superconducting channels in the network [123].

Figure 3.4(b) shows a zoom of the mid-transition critical temperatures for both samples. The shape of these curves allows us to get information about the superconducting properties of the samples and notably the coherence length (see subsection 1.1.5, page 28). For the reference sample, a linear dependence is found over the whole magnetic field range (a linear fit is shown on the same figure in red) which is the typical behavior of bulk superconductor. From the slope of the line, the zero tem-

perature coherence length can be extracted (see subsection 1.1.5, page 29) : $\xi(0) \approx 4nm$ ¹. On the contrary, $T_{c,mid}(H)$ (sample NbNh20(30,50)) shows a quadratic behavior which corresponds to the one-dimensional wire networks (1DWN) limit (see subsection 1.13, page 32). In this case, the critical temperature depends on both the coherence length and the width of the 1D stripe : $T_c(H) = T_{c0} \cdot (1 - \frac{\pi^2 w^2 \xi^2(0)}{3\phi_0^2} H^2)$, with T_{c0} the zero field critical temperature and w the 1D stripe width. Using a 1D stripe width of $w = 20nm$ (estimated from the SEM picture of figure 2.4(b)), the quadratic fit shown in figure 3.4(b) gives : $\xi(0) = 2.9nm$. A cross-over to the bulk behavior is expected when $\xi(T) < w/1.8$ (see subsection 1.13, page 32), which corresponds to $T \approx 7K$, i.e. at $H \approx 5.2T$ in the phase diagram, which is out of the range of our measurements. The evolution of the coherence length as a function of temperature is represented in figure 3.5. Therefore, close to T_c the collective regime is reached.

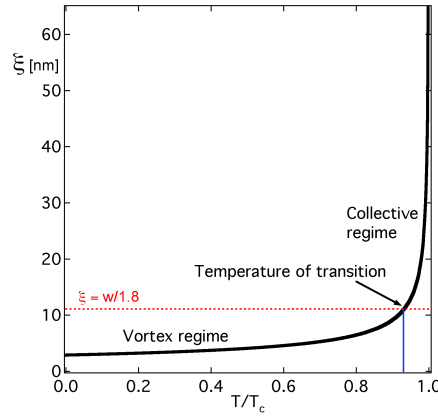


Figure 3.5: Evolution of the coherence length as a function of the reduced temperature (T/T_c). The transition from the vortex regime $\xi < w/1.8$ to the collective regime $\xi > w/1.8$ is represented by the crossing of the horizontal dashed line.

3.2.3.2 Transition from the collective regime to the vortex regime

The collective regime, i.e. the one dimensional wire network regime, is generally only observed in a small temperature range close to T_c . This is

1. Using this value of $\xi(0)$ and fitting $T_{c,nuc}^{ref}(H)$ with the 1DWN model (see subsection 1.1.5, page 28), one obtain a superconducting strip width of $\sim 20nm$, which corresponds roughly to the NbN grains size [95]. The use of the 1D model is thus consistent.

due to the rapid decrease of the coherence length when the temperature is lowered from the critical temperature. Indeed, below a certain temperature, the coherence length becomes lower than the network strand width (w) and a transition to the vortex regime is observed. The temperature of transition from one regime to the other thus depends on T_{c0} , on $\xi(0)$ and on w . However, the choice of the critical temperature of a granular sample is not straightforward. Indeed, the large superconducting transition suggests that the critical temperature strongly varies from one grain to the other.

Using the mid-transition criterion $T_{c,mid}(0)$, the cross-over between the 1D and the bulk behavior is expected around $T \approx 7K$. However, the behavior of $T_{c,onset}(H)$ (see figure 3.4(a)) seems to indicate that the collective regime is still present at lower temperature. Indeed, grains with lower T_c than $T_{c,mid}(0)$ tend to decrease the cross-over temperature. Therefore, it is not surprising that the transition from the collective regime to the vortex regime happens at a lower temperature than the one estimated from equation $w < 1.8\xi$ (see subsection 1.13, page 32) which is valid for homogeneous superconductors.

The magnetoresistance curve for sample NbNh20(30,50) at 5.5K (figure 3.6(a)) with $I_{DC} = 1\mu A$ corroborates the fact that the collective regime is maintained at $T < 7K$. Indeed, several dips are observed reflecting the critical temperature oscillations of the 1DWN observed in figure 3.4(a). The inset of figure 3.6(a) shows the derivative of the magnetoresistance curve. Modulations are observed up to the fourth matching effect, i.e. up to 3.6T. This is much higher than the results obtained by Vinckx et al. [114] for 50nm thick Nb films deposited on an AAO template with pores interdistance 50nm and pores diameter 25nm. Indeed, only the first matching effect was clearly visible and a small change of slope at the second matching field was also present. Therefore, more pronounced matching effects can be obtained using a more judicious choice of the geometrical parameters of the system, i.e. a thinner superconducting film and larger pores. This avoids to cover the pores as it was partially observed in the work of Vinckx et al. [114]. Also, the use of NbN, instead of Nb, increases the interaction between the vortices due to the larger penetration depth. The maximum number of vortices that can be placed in a hole is also higher in our system due to the reduced coherence length of NbN compared to Nb and to the larger pores. All of these are key factors for the observation of stronger matching effects.

At $T = 3K$ and $I_{DC} = 640\mu A$ (figure 3.6(b)) the magnetoresistance curve shows only one shallow dip in resistance at H_1 . Indeed, at $H = 1.28T$, the sample abruptly transit to the normal state. This lower critical magnetic field at 3K compared to 5.5K is due to the much larger

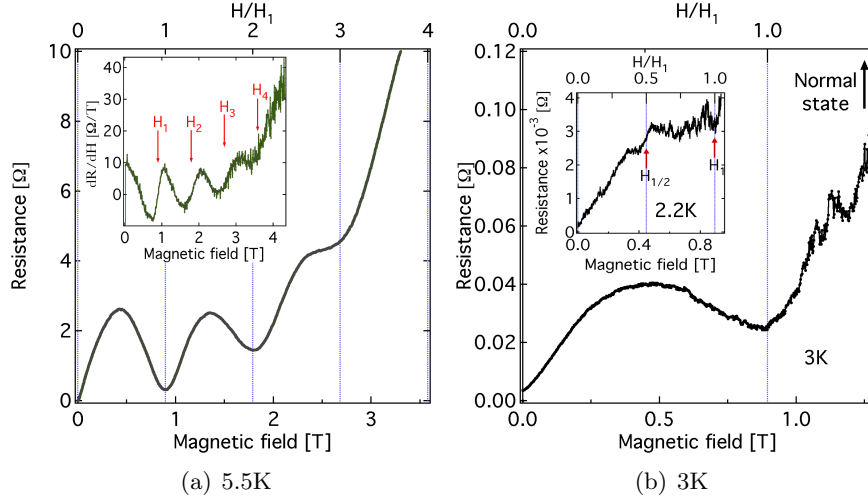


Figure 3.6: Magnetoresistance curve for sample NbNh20(30,50) (a) at $T = 5.5K$ with $I_{DC} = 1\mu A$ (Inset : derivative of the main curve) and (b) at $T = 3K$ with $I_{DC} = 640\mu A$ (Inset : $T = 2.2K$ with $I_{DC} = 660\mu A$). (a) Collective regime and (b) vortex regime. In the vortex regime, $H_{1/2}$ is observed as well as vortex avalanches (noise-like resistance fluctuations).

current of measurement. Also, large resistance fluctuations, reminiscent of vortex avalanches [12,124] are observed. In addition, a fractional matching effect appears at the lowest temperature (see figure 3.6(b) - inset) at $H_{1/2}$. Therefore, it is very likely that a transition from the collective regime to the vortex regime takes place at low temperature.

At the first matching field at $T = 3K$, each hole is occupied by one vortex. However, for $H > H_1$, the additional vortices can be placed either in a hole, forming a doubly-quantized vortex, or going in an interstitial position, depending mainly on the coherence length and on the hole diameter and spacing (see subsection 1.1.4.1, page 16). Using the model of Buzdin [44] with $T_{c0} = T_{c,mid}(0)$ one obtained a minimum hole diameter of $D = 18nm$ for doubly-quantized vortices pinning. Therefore, it is likely that double-quantized vortices appear at $H > H_1$. The second matching effect is not visible since the sample has passed through the normal state.

The lowering of the first matching field effect amplitude at $T = 3K$ compared to what is observed at $T = 5.5K$ can be ascribed to several factors. First, the transition from the collective regime to the vortex regime. Second, the intrinsic pinning which is generally important in such granular films at low temperatures [125,126]. This effect is mainly due to the reduction of the coherence length and to the decrease of

the thermal energy. Third, the appearance of the Bean critical state (see subsection 1.1.4.3, page 18) at low temperatures also weaken the matching features. Indeed, due to vortex concentration gradients, the matching condition is not fulfilled in the entire sample but rather in small regions where the vortex concentration is close to the one of the pinning centers [127,128]. This decreases the impact of the matching effect on the vortex lattice stability. Third, Reichhardt et al. [52] suggested that the observed weakening of the matching effects at low temperatures could result from the increase of the pinning strength of the array instead of the intrinsic pinning usually evoked. Indeed, they showed that when the pinning strength becomes much more important than the vortex-vortex interactions, the matching effects are less pronounced.

3.2.4 Conclusion of section 3.2

The superconducting properties of thin NbN films containing a dense triangular lattice of antidots have been studied. The lattice of antidots has been implanted into the superconducting films by depositing NbN on the porous face of alumina templates. Two different samples have been studied. First, a NbN film containing an antidot lattice with a period of 100nm and hole diameter of about 60nm have been investigated. The superconducting transition is much larger than the one of its reference sample and the T_c is decreased due to nanostructuration. The numerous matching effects present in the magnetoresistance curves reflect the strong vortex pinning of the antidot lattice. It was demonstrated that the number and the intensity of matching effects can be significantly enhanced compared to previous works by the appropriate modification of the geometrical parameters. Second, a triangular array of antidots with interdistance of 50nm and hole diameter of 30nm was studied. Due to the small superconducting strand width, the one-dimensional superconducting network limit is maintained over a relatively large temperature range, i.e. down to $t \sim 0.7$. The high temperature magnetoresistance curves show matching effects up to the fourth matching field. The number of matching effects is limited by the short-range order present in this array of antidots. At lower temperature ($t < 0.7$), the sample shows a transition from the collective regime (1DWN) to the vortex regime in which only the first matching effect is observed. This transition to the vortex regime is corroborated by the appearance of a matching feature at $H_{1/2}$ and vortex avalanches.

3.3 Vortex pinning in NbN thin films by an hexagonal lattice of defects

3.3.1 Motivation

In the previous section we have shown that a dense triangular array of antidots can be created in a thin superconducting film by the deposition of NbN on the porous face of an anodized aluminum oxide template. It is also possible to take advantage of the barrier layer of the alumina template to create another type of pinning center lattice in superconducting thin films. Indeed, the interstitial sites of the bump lattice are expected to create localized defects in the NbN deposit thus creating an hexagonal array of pinning centers. In this section, we investigate the case of NbN deposited by sputtering while the case of Nb deposited by MBE will be studied in the next section.

Figure 3.7 shows the advantage of the hexagonal array of pinning centers compared to the triangular array of holes. Indeed, if we consider a triangular unit cell (as represented in red on both figures), there is an half pinning center per unit cell in the case of the triangular array of holes (figure 3.7(b)) while there is one pinning center per unit cell in the case of the hexagonal array of defects (figure 3.7(c)). Therefore, two times more vortices can be pinned in the case of the hexagonal lattice.

Another advantage of the honeycomb lattice compared to the triangular lattice is the enhanced stability of the interstitial vortices. Indeed, the hexagonal arrangement of the pinned vortices produces a caging potential at the center of the hexagon which is much more pronounced compared to the triangular lattice.

It should be pointed out that a similar approach was used by Vinckx et al. [13] to produce hexagonal array of pinning centers. The interstitial voids of a self-assembled triangular array of polystyrene (PS) spheres were exploited to produce an hexagonal array of pinning centers in Nb thin films. Basically, our approach allows us to produce about 4-5 times denser pinning centers lattice.

This section is organized as follows : first, the impact of the nanostructuration on the phase diagram of superconductivity is discussed. Then, the evolution of the matching effects with the temperature is studied and compared to theoretical results. Finally, the impact of the pinning force on the matching effects is investigated.

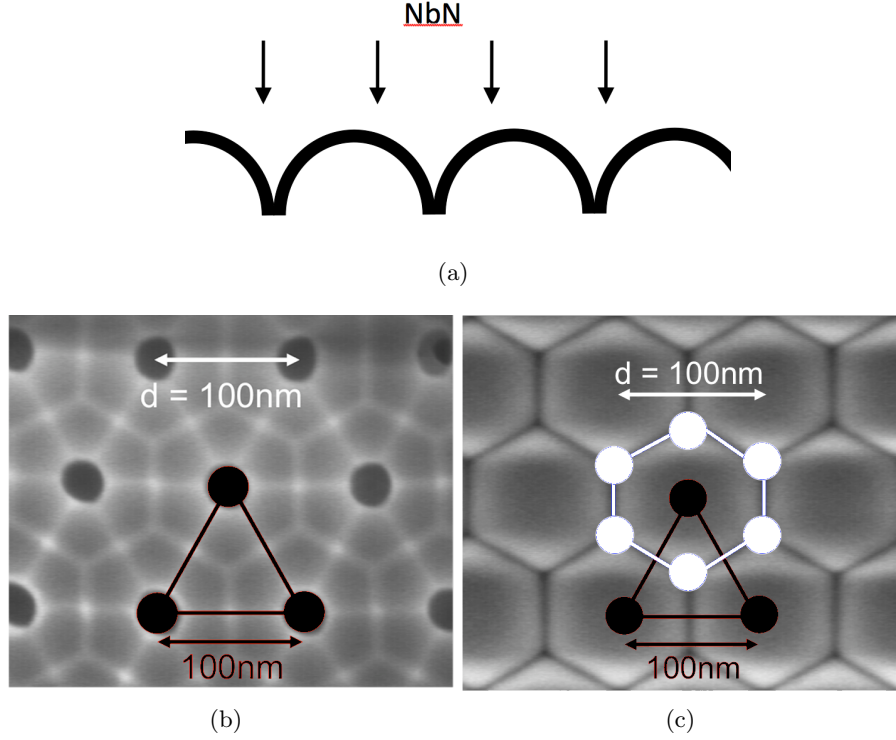


Figure 3.7: (a) Schematic representation of the deposition of NbN on the barrier layer side of the alumina templates. Pinning centers are created at the interstitial sites of the bumps. (b) and (c) Vortices configurations when all the pinning centers are occupied by one vortex, i.e. the first matching field (H_1). (a) Using the hole surface of an AAO template and (b) when using the barrier layer surface. The red triangle represents a unit cell of the pore lattice. There is an half hole per unit cell while there is one interstitial site per unit cell. There are thus twice more pinning centers for NbN deposited on the barrier layer surface than for NbN deposited on the hole surface.

3.3.2 Phase diagram

The phase diagram for sample NbNb25(-,100)#1 is represented on figure 3.8. This sample is a 25nm thick NbN layer deposited by dc-magnetron sputtering on the barrier layer of an alumina template achieved in our lab (see figure 2.5, page 47). The critical temperature was obtained by adjusting the temperature while varying the magnetic field in order to maintain a resistance of $R = 10^{-3}R_n$ (R_n is the normal state resistance) using a dc-current of $20\mu A$ ².

A pronounced critical temperature enhancement is observed at $H_1 =$

2. It should be mentioned that for this sample it was not possible to measure simultaneously a reference sample due to the large size of the sample

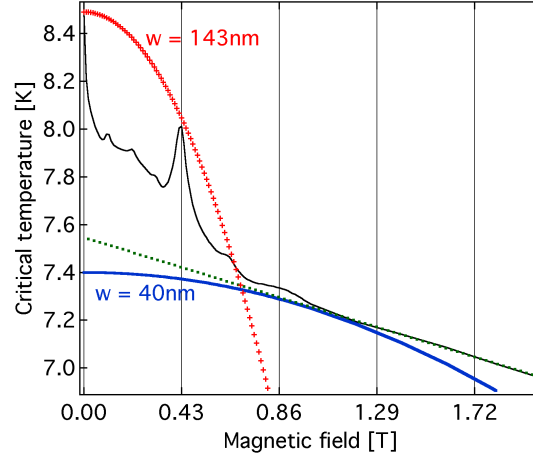


Figure 3.8: Critical temperature as a function of magnetic field (phase diagram) for sample *NbNb25(-, 100)#1*. The black curve was obtained by adjusting the temperature while varying the magnetic field in order to maintain a resistance of $R = 10^{-3}R_n$ (R_n the normal state resistance) using a dc-current of $20\mu A$. The dotted line corresponds to the linear fit of the high field critical temperature. This behavior gives a value of $\xi(0) = 4.2nm$. The relation $T_c = T_{c0} \cdot (1 - \frac{\pi^2 w^2 \xi^2(0)}{3\phi_0^2} H^2)$ for a 1D superconductor, is represented on the graph with $\xi(0) = 4.2nm$, $w = 143nm$, $T_{c0} = 8.49K$ (curve with a plus marker) and with $\xi(0) = 4.2nm$, $w = 40nm$, $T_{c0} = 7.4K$ (lower continuous parabolic curve). w is the strand width of the wire network.

0.43T which agrees well with the theoretical matching field of an hexagonal array of pinning centers with interdistance 58nm ($H_1 = \frac{4\phi_0}{3\sqrt{3}d^2}$). Fractional matching features are observed at field $H_n = n \cdot H_1$ for $n = 1/4, 1/2, 3/4$ and $3/2$. A shoulder is also observed at H_2 . In addition to the matching peaks, a change of trend is observed around 0.7T. The low field pronounced drop of resistance (without considering the matching peaks) becomes a linear decrease at $H > 0.7T$.

The high field linear dependence is associated to the bulk behavior (see subsection 1.1.5, page 28). It allows us to extract $\xi(T = 0)$ from the slope of the curve (see subsection 1.1.5, page 28). A value of 4.2 nm is obtained which was used to trace the theoretical behavior of a 1DWN array for different wire width. The envelop of the low field behavior is represented in figure 3.8 (curve with a plus marker) and corresponds to a superconducting strand width of 143 nm. This result is of course not physical. The more plausible case of superconducting channel of 40 nm wide is also shown in figure 3.8 (lower continuous parabolic curve). This last case also strongly deviates from the experimental data. Therefore, the phase diagram for sample *NbNb25(-, 100)#1* doesn't correspond to the collective regime (1DWN limit).

Reichhardt et al. [54] have calculated using molecular dynamics simulations, the stable vortex configurations as well as their corresponding critical current (critical depinning force) as a function of field (number of vortices in the system) of a superconductor containing a hexagonal lattice of pinning centers. Critical current enhancements were predicted at each integer value of n up to $n = 4$ and at each half integer value up to $n = 4.5$. Nevertheless, the low field region was not precisely investigated which doesn't allow us to know if matching features are present at $n = 1/4$ and $3/4$.

From the phase diagram shown in figure 3.8, we can thus conclude that an hexagonal array of pinning centers have been implanted in the NbN thin film. In the next subsection, we discuss the temperature dependence of the matching effects in comparison with previous experimental and theoretical works. The vortex configurations at the different matching fields are also presented.

3.3.3 Influence of the temperature on the matching effects

3.3.3.1 High field matching effects

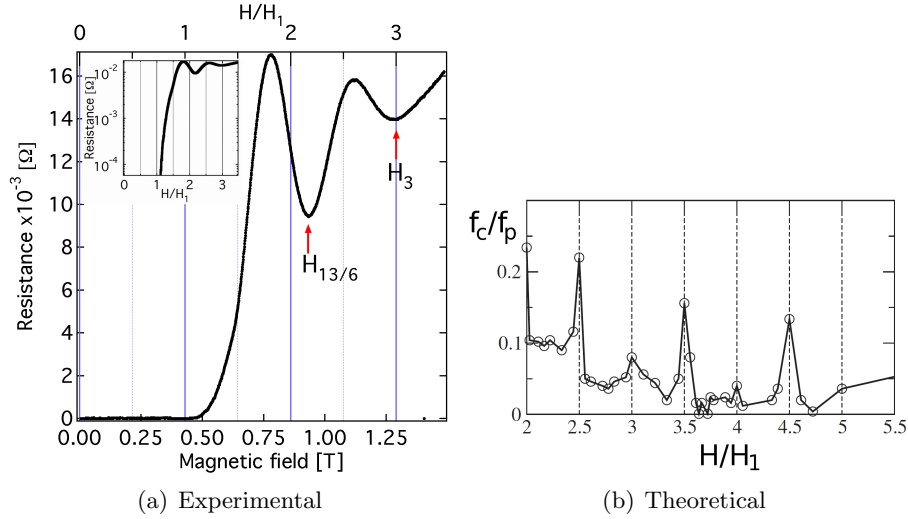


Figure 3.9: (a) Magnetoresistance curve for sample NbNb25(-,100)#1 at $T = 4.2K$ with $I_{DC} = 4mA$. Inset : Same curve with log-scale on the ordinate that emphasizes the abrupt increase of resistance after H_1 . (b) Theoretical critical depinning force (f_c) as a function of magnetic field taken from Reichhardt et al. [54]. Curve normalized by the pinning force f_p and the first matching field H_1 . $f_p = 0.5f_0$, where $f_0 = \phi_0^2/2\pi\mu_0\lambda^3$

The vortices arrangements strongly depend on the symmetry of the pinning lattice but also on the temperature. Indeed, the temperature modifies both the coherence length and the London penetration depth. The coherence length determines how many vortices can be pinned on a single pinning center. The penetration depth modifies the length scale on which the vortices interact with each other and with the pinning centers. Moreover, the thermal energy is also playing an important role in the stability of the vortices configurations. Therefore, it is very likely that the matching effects will be strongly modified by the temperature.

Figure 3.9(a) shows the magnetoresistance curve for sample NbNb25(-,100)#1 at $T = 4.2K$ and $I_{DC} = 4mA$. On this figure only the behavior above the first matching field ($H_1 = 0.43T$) is visible because, at lower fields, the resistance is below the resolution of our setup. Several important features can be observed. First, a steep increase of the resistance is observed just after the first matching field. Indeed, at $H < H_1$ the resistance is below the resolution, i.e. $R < 2 \cdot 10^{-5}\Omega$ and just after the first matching field ($H > H_1$), the resistance increases by at least 3 orders of magnitude (up to a maximum value of $1.7 \cdot 10^{-2}\Omega$ (see the inset of figure 3.9(a))). This steep resistance increase is a clear signature of the appearance of interstitial vortices [51]. Therefore, even at such low temperature, only one vortex can be pinned in each pinning center. This gives us information about the maximum size of the pinning center. Using the theoretical model of Mkrtchyan and Schmidt (see subsection 1.1.4.1, page 16), one obtains that the diameter of the defect should be below $D < 47nm$. Using the more appropriate model of Buzdin (see subsection 1.1.4.1, page 16) because it takes into account the vortex-vortex interaction, the diameter should be below $D < 25nm$. Since the pinning center interdistance is 57nm, it seems plausible to have pinning center diameter below 25nm. However, the model of Buzdin was developed for a triangular array of pinning centers. Applying the model of Buzdin to an honeycomb array would lead to an overestimate because the vortex-vortex interaction energy is larger which favors the formation of giant vortices. Therefore, it is very likely that the pinning center diameter is below 25nm.

Figure 3.9(a) shows that strong matching effects are still visible at low temperatures. This is a major difference with previous experimental works in which matching effects disappear at low temperatures [6,39,115]. We believe that this difference is due to the very large London penetration depth in our samples (see subsection 1.1.5, page 29) which leads to long range vortex-vortex interactions even at the lowest temperature. It should also be pointed out that contrary to what is generally found in the litterature, matching effects are observed at higher field at $T = 4.2K$

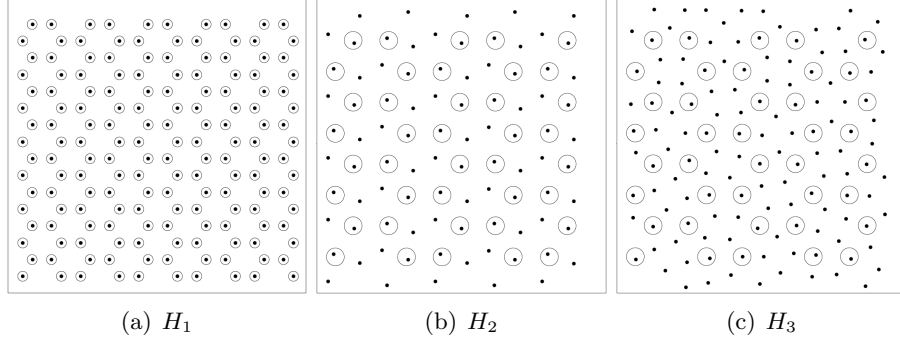


Figure 3.10: Vortices positions (black dots) for honeycomb lattice of pinning centers (open circles) at several matching fields calculated using molecular dynamics (taken from Reichhardt et al. [54]). (a) H_1 . (b) H_2 . (c) H_3 .

than in the phase diagram. Indeed, strong matching effects are observed at H_2 and H_3 . Moreover, the second matching dip is not at its theoretical value (H_2) but rather corresponds to $H_{\frac{13}{6}}$. We don't have any explanation for this particular value of $H_{\frac{13}{6}}$. Vortex imaging would be necessary to clarify this point but unfortunately, it is not possible at this time due to a too weak resolution of the imaging techniques.

Using molecular dynamics, Reichhardt et al. [54] calculated the critical depinning force of the vortex lattice at different magnetic fields and for different pinning strengths. Figure 3.9(b) shows the critical depinning force (f_c) normalized to the pinning force (f_p) as a function of magnetic field in the field range $[H_2; H_{5.5}]$. The vortices configurations at H_1 , H_2 and H_3 predicted by Reichhardt et al. are shown in figure 3.10. All the predicted matching configurations have been presented in subsection 1.1.4.5. The high field behavior of our sample does not agree very well with the theoretical results. The principal difference resides in the theoretical prediction of intense matching peaks at half integer matching fields (notably $H_{2.5}$ and $H_{3.5}$) due to the formation of a triangular lattice of vortex. In theory, the peak at $H_{3.5}$ is even much more pronounced than the one at H_3 . Also, the peak shift from H_2 to $H_{\frac{13}{6}}$ was not predicted. The disagreement from the experimental data and the theoretical predictions are likely due to different superconducting parameters. Indeed, the simulations of Reichhardt et al. were performed with a certain set of parameters (λ , diameter of pinning centers, interdistance of pinning centers,...) mainly corresponding to previous experimental studies using lithographic techniques and relatively low κ superconductors. In our case, our NbN film is an extreme type II superconductors ($\kappa \gg 1$).

Therefore, the vortex-vortex interaction is much stronger in our samples than in the one considered in Reichhardt's work. A second reason could be that the molecular dynamic simulations is not appropriate for such systems and that Ginzburg-Landau simulations are required. Also, the presence of intrinsic pinning and non-flatness of the film can strongly affect the stable vortices configurations.

3.3.3.2 Low field matching effects

A zoom on the low field part ($0 < H < H_1$) of the magnetoresistance curves for sample *NbNb25*(-,100)#1 are shown in figure 3.11 for several temperatures (excepted for figure 3.11(b) which shows the derivative of figure 3.11(a)). The magnetoresistance at $T = 8.12K$ (figure 3.11(a)) shows well pronounced matching dips which are highlighted in the derivative represented in figure 3.11(b). Accordingly to the phase diagram, matching effect are observed for fields $H_n = n \cdot H_1$ corresponding to $n = 1/4, 1/2$ and $3/4$. However, an additional matching effect can be made out at $n = 1/3$ as a shallow but reproducible change of slope.

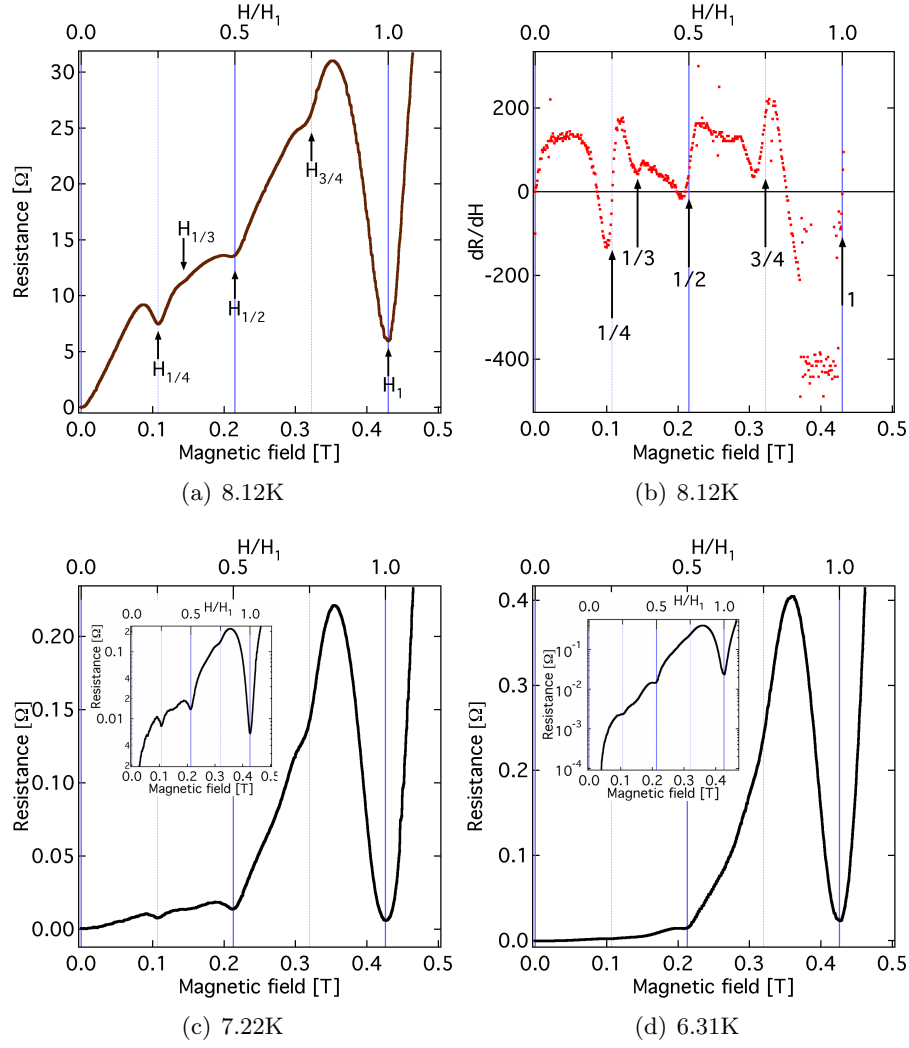


Figure 3.11: Magnetoresistance curves for sample NbNb25(-,100) at different temperatures and currents. (a) $T = 8.12K$ and $I = 10\mu A$. (b) Derivative curve of figure 3.11(a). (c) $T = 7.22K$ and $I = 40\mu A$. (d) $T = 6.31K$ and $I = 2.5mA$. (c) and (d) Inset : log-scale on the ordinate.

Figure 3.11(c) shows the magnetoresistance curve at $T = 7.22K$ with $I_{DC} = 40\mu A$. The fractional matching effects are still present but a global change of the shape of the curve appears. The relatively constant increase of the resistance between zero field and H_1 with the intermediate fractional matching effect observed at 8.12K is replaced by a relatively abrupt increase of the resistance at $H_{1/2}$. At even lower temperature (figure 3.11(d)), the same global trend is observed with weaker fractional matching effects. Therefore, the decrease of temperature seems to have two different effects on the low field behavior. First, the attenuation of the fractional matching effects and second, the appearance of a abrupt increase of the resistance above $H_{1/2}$. The decrease of the matching peaks or dips when decreasing temperature is well-known and is due to several effects, i.e. intrinsic pinning, the increase of the pinning force, the decrease of the vortex-vortex interaction force and the critical state (as already discussed in the previous section). The abrupt increase of the resistance above $H_{1/2}$ is associated to the disappearance of the highly stable triangular vortex arrangement at $H_{1/2}$ (see figures 1.9(a) and 1.9(b), page 25). This feature has been predicted by Reichhardt et al. [54].

Figure 3.12 shows the vortex configurations for the fractional matching fields. Figure 3.12(a) shows the triangular lattice of vortex predicted at $H_{1/2}$ by Reichhardt et al. [54]. In figure 3.12 (b), (c) and (d), we propose stable vortex configurations at $H_{1/4}$, $H_{3/4}$ and $H_{1/3}$ respectively. At $H_{1/4}$, an honeycomb lattice of vortex is formed, at $H_{3/4}$ an honeycomb lattice of vortex vacancy is formed and at $H_{1/3}$ a deformed honeycomb lattice of vortex is formed.

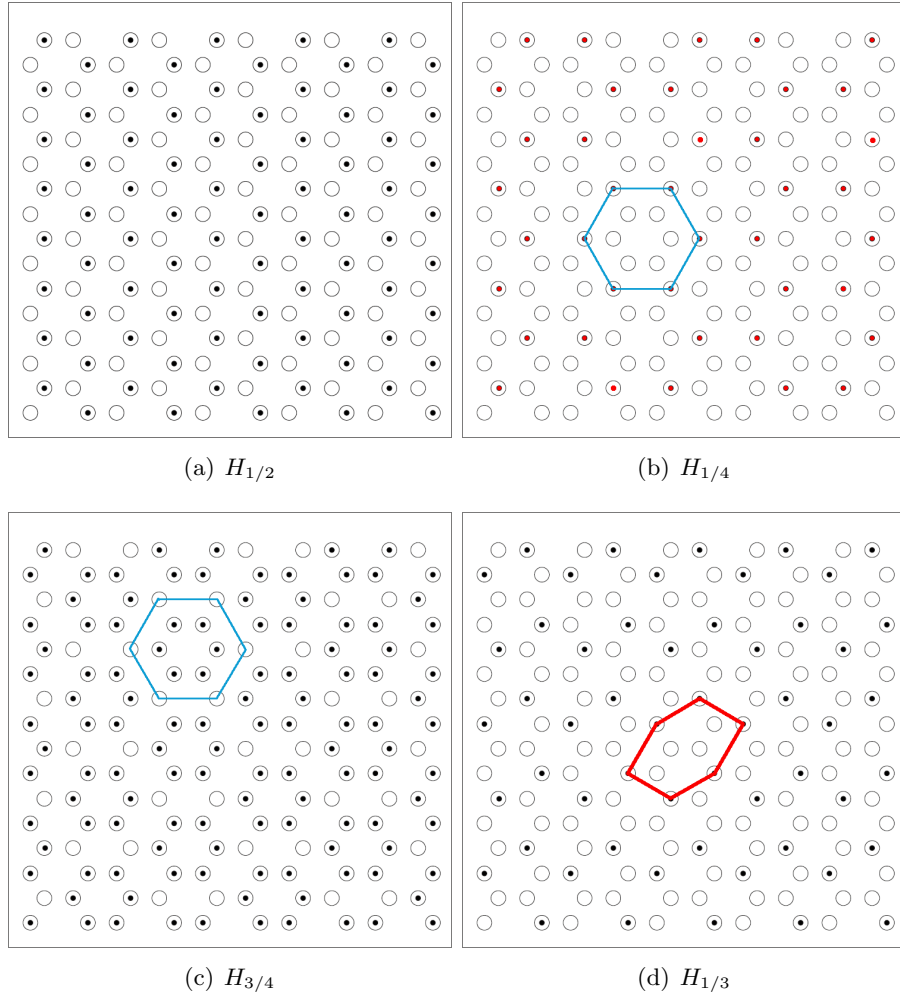


Figure 3.12: Possible vortices positions (black dots) for honeycomb lattice of pinning centers (open circles) at several fractional matching fields. (a) $H_{1/2}$. (b) $H_{1/4}$. (c) $H_{3/4}$. (d) $H_{1/3}$.

3.3.4 Influence of the pinning strength on the matching effects

The vortex pinning properties in honeycomb pinning center arrays have been predicted theoretically to depend strongly on the pinning strength [54]. Indeed, different vortices configurations and vortex motion regimes are expected to appear for different pinning strengths. We used two approaches to study these phenomena. In the first approach, a reduction of the pinning strength was achieved by depositing an insulating layer (Si) on the barrier layer prior to the deposition of the NbN film (sample sample NbNb+i25(-,100)). This layer flattens the bump relief and thus reduces the structural disorder of the NbN at the interstitial sites, which leads to a reduced pinning strength. In the second approach, changes in pinning strength were obtained considering different thicknesses of the NbN film. In particular, the goal is to study how the pinning properties evolve for lower thicknesses with expected stronger pinning strengths. Also, the temperature can be used to tune the pinning strength since it increases with decreasing temperature.

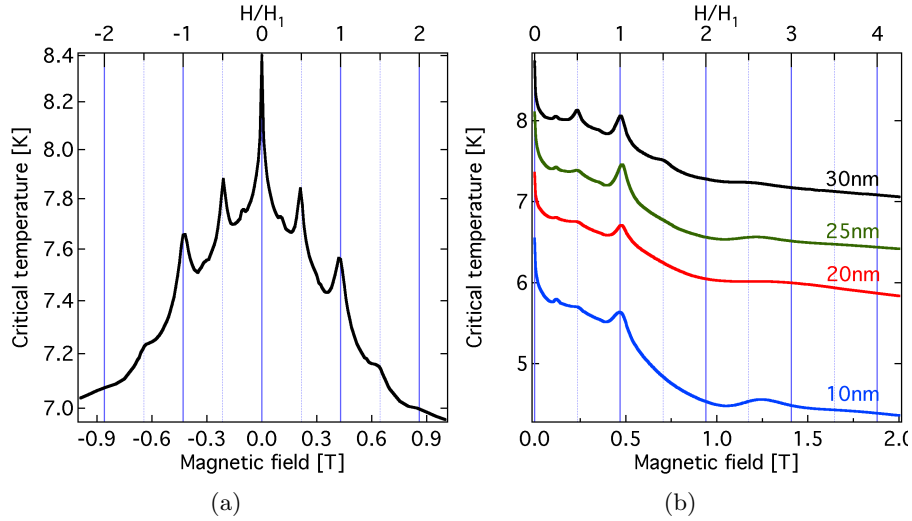


Figure 3.13: Critical temperature versus magnetic field (phase diagram) for several samples extracted from a series of $R(T)$ curves ($I_{DC} = 20\mu A$) using a resistance criterion of $R_c = 10^{-3}R_n$ (R_n is the normal state resistance). (a) NbNb+i25(-,100). (b) NbNb10(-,100), NbNb20(-,100), NbNb25(-,100)#2 and NbNb30(-,100).

Figure 3.13(a) shows the critical temperature curve as a function of magnetic field for sample NbNb+i25(-,100). In this case, lower pinning strength is expected since the insulating layer flattens the surface of the barrier layer. The critical temperature was extracted from a series of

$R(T)$ curves ($I_{DC} = 20\mu A$) using a resistance criterion of $R_c = 10^{-3}R_n$ (R_n is the normal state resistance). Thus, the criterion used probes the appearance of resistance, i.e. the onset of vortex motion. The measurement was performed with a dc current of $20\mu A$. Strong modifications are observed at low field compared to figure 3.8. Indeed, the first matching effect (H_1) is less pronounced for the smoothed surface while the fractional matching effect at $H_{1/2}$ is strongly enhanced and is even more pronounced than the one at H_1 .

Figure 3.13(b) represents the critical temperature curves as a function of magnetic field for NbN film for samples NbNb10(-,100), NbNb20(-,100), NbNb25(-,100)#2 and NbNb30(-,100), i.e. for different NbN thicknesses. By varying the thickness of the superconducting film, the pinning strength is modified. Indeed, for low thicknesses a maximum pinning strength is obtained while a negligible pinning strength is obtained for infinitely thick films. The low field evolution of the matching peaks observed when varying the film thickness is in agreement with the one observed for sample NbNb+i25(-,100) (figure 3.13(a)) and for sample NbNb25(-,100)#1 (figure 3.8). For the thinnest films, i.e. thickness less than 25nm, the matching peaks shows the same relative intensity as those observed in figure 3.8 for strong pinning centers. The peak at H_1 is well-pronounced and fractional matching features are observed at $H_{1/4}$, $H_{1/2}$ and $H_{3/4}$ with low intensities. This indicates that the pinning strength saturates for thicknesses lower than 25nm. Surprisingly, for all these samples, a matching effect is observed at $H_{2.5}$ while it was observed at H_2 for sample NbNb25(-,100)#1 (figure 3.8).

Some matching features are enhanced for lower pinning strength, i.e. NbNb+i25(-,100) and NbNb30(-,100). In particular, the $H_{1/2}$ peak becomes more pronounced and a shoulder also develops at $H_{1.5}$. On the contrary the matching effect at H_1 is less pronounced than for sample NbNb25(-,100)#1. These modifications are related to the increase of the vortex-vortex interaction force relatively to the vortex-pinning center interaction force. Indeed, the enhancement of the vortex-vortex interaction force favors the triangular arrangement of vortex compared to the hexagonal arrangement. The corresponding vortices arrangement have been discussed in subsection 1.1.4.5 (page 23) together with their stability (critical depinning force). At $H_{1/2}$, the depinning force is equal to the pinning strength independently of this strength due to the fact that the vortex lattice is triangular. At H_1 , for low pinning strength, a deformed triangular lattice is formed by placing two vortices at interstitial positions of the honeycomb pinning center lattice (see figure 3.15). The resulting vortex lattice is only partially pinned which results in a lower depinning current as thus a lower matching effect [54].

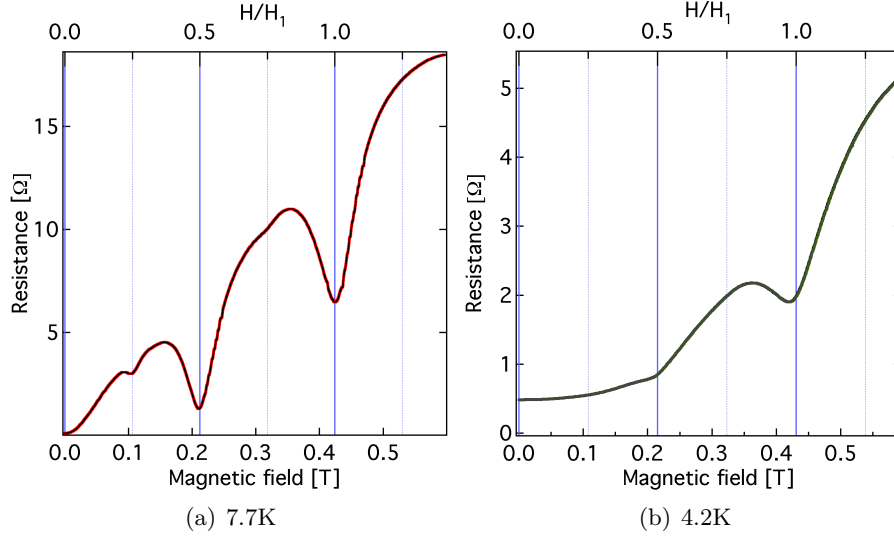


Figure 3.14: Magnetoresistance curve for sample NbNb+i25(-,100). (a) $T = 7.7K$ and $I = 20\mu A$. (b) $T = 4.2K$ and $I = 2.4mA$. At low temperature, the first matching field is more pronounced (proportional to $H_{1/2}$) than at high temperature due to the enhancement of the pinning strength.

The effect of the pinning strength on the matching effects can also be probed by varying the temperature. To illustrate this, figure 3.14 shows magnetoresistance curves for sample NbNb+i25(-,100) at $T=7.7K$ (figure 3.14(a) - $I_{dc} = 20\mu A$) and at $T=4.2K$ (figure 3.14(b) - $I_{dc} = 2.4mA$). At high temperature, the matching peaks are similar to those present in the critical temperature (figure 3.13(a)) with strong matching peaks at $H_{1/2}$ and H_1 . Smaller matching features are observed at $H_{1/4}$ and $H_{3/4}$. However, at low temperature, the matching peak at $H_{1/2}$ has almost completely disappeared. It is only observed as a change of the magnetoresistance slope. This is due to an increase of the pinning strength when decreasing the temperature which leads to the relative enhancement of the first matching effect compared to the other matching effects. However, a global reduction of all the matching effects is also observed due to the appearance of intrinsic pinning and to the appearance of the critical state.

3.3.5 Conclusion of section 3.3

In this section, we demonstrated the feasibility to use the barrier layer to create an hexagonal array of pinning centers in NbN thin films. The main advantage of this approach is to create twice more pinning

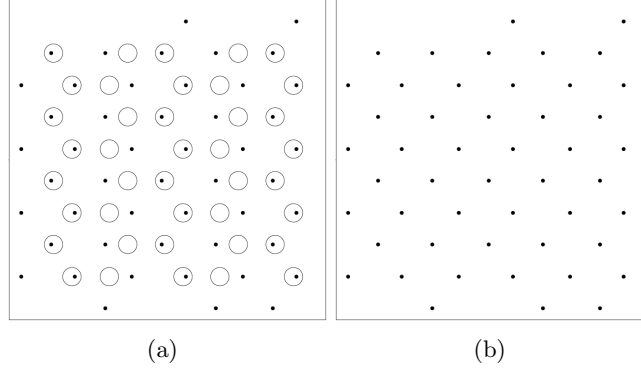


Figure 3.15: (a) Vortices positions (black dots) and pinning site location (open circles) at $H = H_1$ for honeycomb pinning center lattice with low pinning strength. (b) Same as figure 3.15(a) with the vortices positions only. The vortices tends to form a triangular lattice due to the large vortex-vortex repulsive force compared to the vortex-pinning center attractive force. Theoretical results obtained from molecular dynamics taken from Reichhardt et al [54].

centers than with the triangular array of holes. Moreover, the caging potential of an hexagonal array is more efficient than the one of a triangular array. The critical temperature curves as a function of magnetic field indicates that even at high temperature, the vortex regime is probed. Also, the sharp peak observed at H_1 and several small peaks at $H_{1/4}$, $H_{1/2}$ and $H_{3/4}$ indicates that the pinning strength is relatively important. At higher fields, the matching features are weaker indicating the appearance of interstitial vortices. However, at low temperatures, the high field matching features are enhanced.

The modulation of the pinning strength and its influence on the matching peaks have been investigated considering three different approaches. First, a 25nm thick insulating layer was deposited prior to the NbN film deposition in order to flatten the barrier layer surface and to reduce the pinning strength. Second, layers of various thicknesses of NbN have been deposited on the barrier layer. For the lowest pinning strength, the matching effect is enhanced at $H_{1/2}$ compared to H_1 , in good agreement with the theoretical predictions. This is due to the relative increase of the vortex-vortex interaction compared to the pinning force. At low temperature, the opposite effect is observed (the matching effect at H_1 is more pronounced than at $H_{1/2}$). This is due to the increase of the pinning strength compared to the vortex-vortex interaction.

3.4 Vortex pinning in Nb by a quasi-hexagonal lattice of blind holes

In the previous section, we have shown that the hexagonal array of pinning centers results from localized weakened superconducting regions in the NbN film at the interstitial positions of the bump lattice. In this section, we discuss the case of thin Nb films deposited by MBE on the barrier layer. In this case, we show that the angle of deposition is a critical factor for the observation of matching effects. Six different samples have been studied varying the thickness (30nm and 50nm) and the angle of deposition (0° , 30° and 60° from the normal to the surface). In the case of the deposition at 30° from the normal to the surface, a quasi-hexagonal array of pinning centers is obtained. However, the deposition at 60° leads to a discontinuous film ($R \rightarrow \infty$)

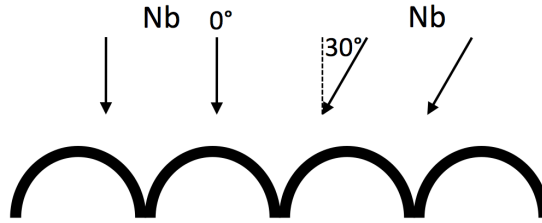


Figure 3.16: Schematic representation of the MBE deposition of Nb on the barrier layer surface of alumina template. The perpendicular and 30° from normal depositions are represented.

3.4.1 Vortex pinning in Nb thin films deposited perpendicularly

The critical current curves as a function of magnetic field obtained at different temperatures are presented in figure 3.17(a) for sample Nbb30(p)(-,100) and in figure 3.17(b) for sample Nbb50($\perp$)(-,100). These curves were extracted from a series of V-I curves measured at different magnetic field by taking a resistive criterion of $R = 0.1 \cdot R_n$ (R_n is the normal state resistance), corresponding to the onset of the resistive state, i.e. the vortex motion. The critical current curves have been normalized to the zero-field critical current.

Contrary to the results shown in figure 3.13 (page 87), i.e. for NbN sputtered on the barrier layer, no matching peaks are observed for both thicknesses. However, a close look to figure 3.17(a) (sample Nbb30(p)(-,100)) and figure 3.17(b) (sample Nbb50(p)(-,100)) reveals a small change in the slope of the critical current curves at H_1 and $H_{1/2}$ respectively.

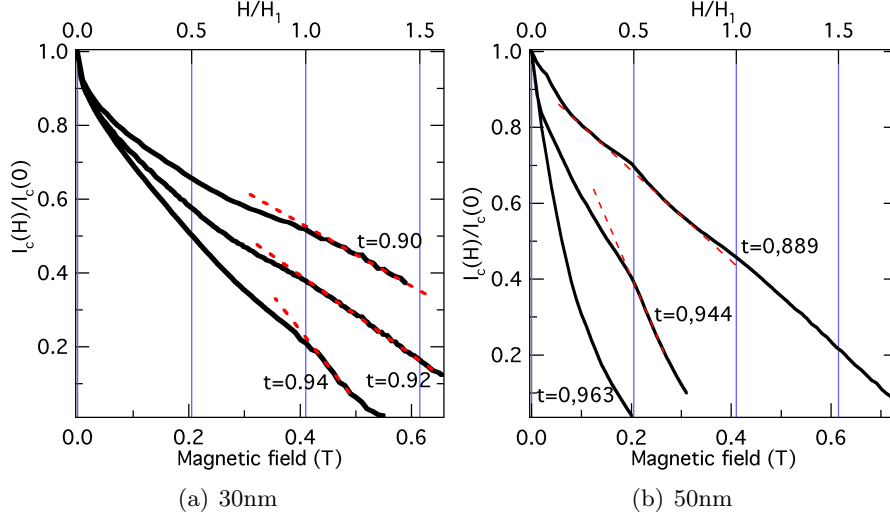


Figure 3.17: Critical current curves as a function of magnetic field at different temperatures (a) for sample Nbb30(p)(-,100) and (b) for sample Nbb50(p)(-,100). The critical current is extracted from a set of voltage vs current curves using a low resistive criterion ($R = 0.1 \cdot R_n$, R_n is the normal state resistance), i.e. corresponding to the onset of vortices motion. The critical current curves have been normalized to the zero-field value. The reduced temperatures of the measurements are indicated on the graphs.

Dashed lines have been added to highlight this change of slope. Therefore, a weak pinning force seems to be present at the interstitial sites. Therefore, it seems that the deposition technique (sputtering or MBE) and/or the superconductor (NbN or Nb) play a major role in the observation of matching effects. On the one hand, the MBE deposition of Nb is performed at a high rate and at room temperature which leads to an amorphous structure of the Nb film. Therefore, the quality of the film is quite poor which leads to important intrinsic pinning. On the other hand, the sputtered NbN thin film is highly granular (see subsection 2.3.1, page 56) and very sensitive to the surface of the substrate. Apparently, the structural quality of the NbN deposit at the sharp interstitial sites is much reduced compared to the one of the Nb deposited by MBE. The larger coherence length of Nb can also account for the lower pinning strength since pinning centers with dimension lower than the coherence length are inefficient [41, 42].

3.4.2 Vortex pinning in Nb thin films deposited at 30° from the normal

The angular depositions at 30° and 60° were achieved simultaneously with the 0° deposit by placing several pieces of the same substrate on tilted holders. On the one hand, it has the advantage that the films deposited at different angles have the same quality. On the other hand, the effective thicknesses of the samples are lower than the one of the perpendicular deposit, i.e. 30 or 50nm, multiplied by a factor $\cos 30^\circ$ or $\cos 60^\circ$. Therefore, the samples deposited simultaneously but with different angles do not have the same effective mean thickness. The samples deposited with a 60° angle were discontinuous ($R \rightarrow \infty$), which demonstrates that the angular deposition induces a strong nanostructuration of the Nb deposit. However, the samples deposited under 30° were superconducting. A schematic representation of the resulting Nb thin film after MBE deposition at 30° on the barrier layer surface is shown in figure 3.18. The Nb is highly nanostructured due to the shadowing effect of the bumps. The thinnest regions of Nb will act as pinning centers (blind holes) due to the lower loss of condensation energy [3].

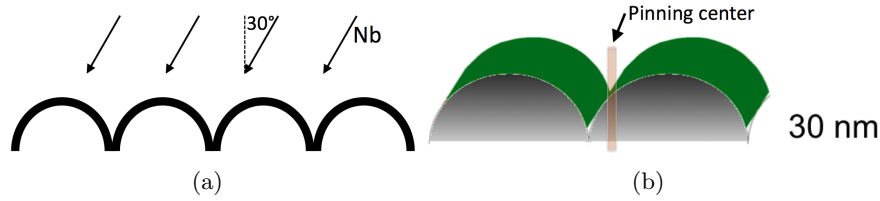


Figure 3.18: Schematic representation of (a) a deposition at 30° from the normal and (b) the resulting Nb layer (transversal view). Pinning centers are localized in the regions with the lowest thickness.

The critical current curves as a function of the magnetic field for sample Nbb30(30°)(-,100) and sample Nbb50(30°)(-,100) are represented respectively in figure 3.19(a) and in figure 3.19(b). These curves were also extracted from a set of V-I curves using a low resistance criterion ($R = 0.1 \cdot R_n$). Strong matching effects are visible for both thicknesses which proves that strong modifications were brought to the Nb deposit. For both samples, matching peaks are observed at multiples of 0.21T. This is qualitatively similar to the results obtained for sample NbNb+i25(-,100), i.e. with the low pinning strength (see figure 3.13(a)). The matching effects are stronger for sample Nbb50(30°)(-,100) which indicates that the thickness modulation is more pronounced in this case.

In this case, the origin of the matching effect is not a localized sup-

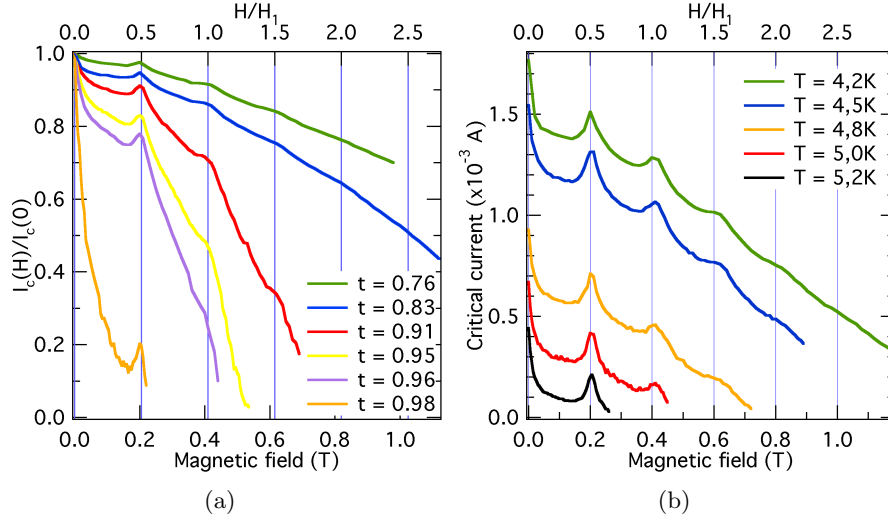


Figure 3.19: Critical current curves as a function of magnetic field at different temperatures (a) for sample Nbb30(30°)(-,100) and (b) for sample Nbb50(30°)(-,100). The critical current is extracted from a set of voltage vs current curves using a low resistive criterion ($R = 0.1 \cdot R_n$, R_n is the normal state resistance), i.e. corresponding to the onset of vortices motion. (a) The critical current curves are normalized to the zero-field critical current.

pression of superconductivity but rather a film thickness modulation (so-called blind hole pinning [3]). Indeed, the weak matching effects observed for perpendicular deposition in opposition with the strong matching peaks observed for the angular deposition indicate that the mobility of the Nb atoms is limited on the barrier layer. Otherwise, similar curves would have been obtained independently of the angle of deposition. Therefore, the Nb deposit will be mainly determined by the shadowing effect of the bumps as sketched in figure 3.18(b). Due to the angular deposition, the shape of the deposit depends on the relative orientation of the triangular lattice of bumps compared to the direction of deposition. Therefore, the array of pinning centers is not strictly speaking an hexagonal array but rather consists in a quasi-hexagonal or a double triangular array. Indeed, as represented in figure 3.20, the shadowing effect will be different if the deposition direction is aligned with an hexagon side (green arrow) or with the bumps apex (red arrow). The six-fold symmetry of our lattice allows us to consider only the directions in the 30° angle formed by these two directions. In practice, due to the multi-domain state of the array of bumps, the direction of deposition will be different for each ordered domain but always somewhere in-between these two limits.

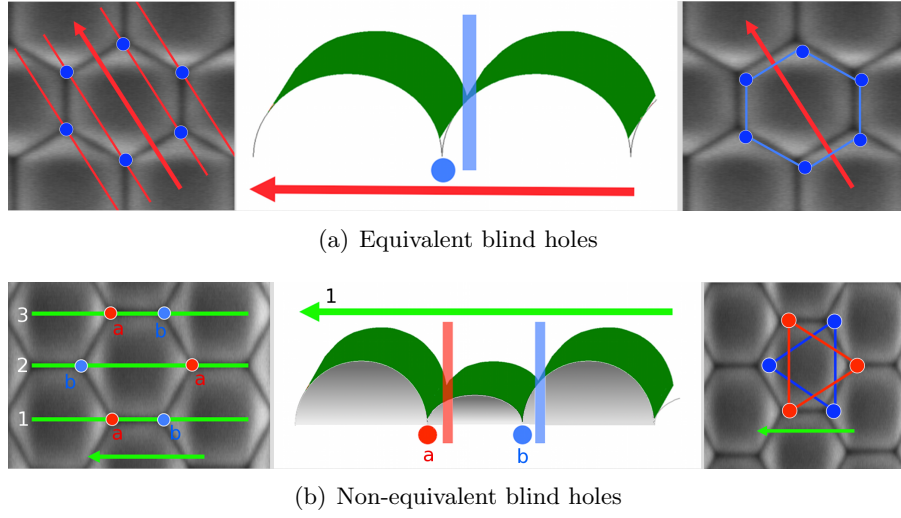


Figure 3.20: Schematic representation of the direction of deposition (left panel), of the shadowing effect on the deposit (central panel) and of the location of the pinning centers (right panel) for two different directions of deposition : (a) Parallel to the bumps row and (b) parallel to the hexagon sides. In (a), the induced pinning centers are all equivalent and form an hexagonal array. In (b), two types of pinning centers with different pinning strength form a double triangular array.

Figure 3.20(a) shows the shadowing effect obtained when the deposition direction is aligned with the bumps rows (red lines and arrow). On the left panel, the interstitial sites are located as blue dots and the direction of deposition as red lines. In this case, all the interstitial sites are equivalent in the sense that they undergo the same shadowing effect. This is represented in the central panel, in which the interstitial site is located with a blue dot, the bumps are drawn in grey and the deposit in green. The most favorable position for a vortex is located with a blue cylinder. In this case, a hole or blind hole is obtained at a position near the interstitial site. An hexagonal array of pinning centers is obtained, as shown on right panel, which is slightly shifted from the array of interstitial sites.

When the deposition direction is aligned with the hexagon sides, each interstitial site doesn't undergo an identical shadowing effect. This is represented in figure 3.20(b). In this case, the sites indicated as 'b' are behind a big bump while the sites noted 'a' are behind a small bump. Therefore, the shadowing effect generates a more pronounced thickness modulation on the 'b' sites than on the 'a' sites (see the central panel). The vortices will experience a strong pinning force on the 'b' sites while a weaker pinning force is present at the 'a' sites. As shown on the right

panel of figure 3.20(b), a double triangular lattice is formed with different pinning strength.

In practice, the majority of the ordered domains are in an intermediate situation because the direction of deposition lies somewhere in between the two directions considered here. In this case, one can assume that a mix between an hexagonal and a double-triangular lattice is obtained, i.e. a double triangular lattice with a small pinning strength difference between the two underlying lattices. Therefore, it is expected that the critical current curves display a behavior similar to an hexagonal lattice of weak pinning centers. Indeed, at $H_{1/2}$, only the stronger pinning centers are occupied. A very stable triangular lattice of vortices is formed which corresponds to a strong matching effect (as seen on figures 3.19(a) and 3.19(b)). At H_1 , the weaker triangular array of pinning centers is filled with vortices. The resulting matching effect is lower intensity due to both the lower pinning strength of the second triangular array and also due to the lower stability of the hexagonal array compared to the triangular array. The matching effects observed at higher field corresponds to similar caging effects of the interstitial vortices observed for the hexagonal array of pinning centers.

3.4.2.1 Vortex flux flow at high field

The magnetoresistance curves for sample Nbb50(30°)(-,100) at $T = 4.6K$ with a dc-current of $500\mu A$ is shown in figure 3.21(a). The upper graph of figure 3.21(b) shows the same curve in a logarithmic scale and the lower graph of figure 3.21(b) represents the derivative of the magnetoresistance curve. The $H_{1/2}$ and H_1 matching effects are clearly observed as dips. Above H_1 , the matching features are less marked and can be mainly characterized as change in the slope of the curve. Another feature of figure 3.21 is the linear dependence of the resistance above H_1 . As is shown by red lines on both figure 3.21(a) and the lower graph of figure 3.21(b), different resistive regimes are reached in between the different matching fields. These features are maintained up to $H_{2.5}$ while for higher fields the matching effects fade away.

The observation of this linear regime coincides with the appearance of interstitial vortices caged in the hexagons. However, due to the large current ($I_{DC} = 500\mu A$), some interstitial vortices can move from cell to cell. This linear behavior is reminiscent of the flux flow [30] (see subsection 1.1.4, 15) for which the electric field is given by :

$$E = \frac{Bj\phi_0}{\eta} \quad (3.1)$$

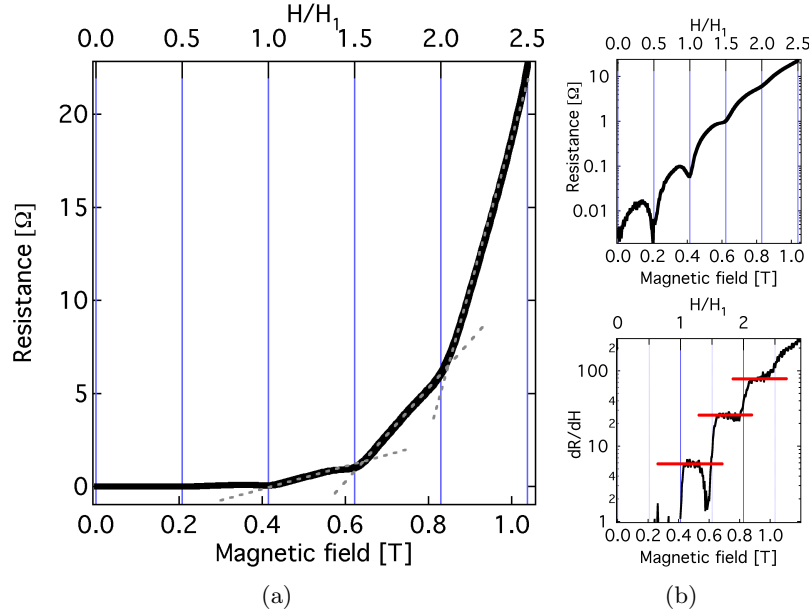


Figure 3.21: (a) Magnetoresistance curve at $T = 4.6K$ and $I_{DC} = 500\mu A$ for sample Nbb50(30°)(-,100). (b - upper graph) Same curve with logarithmic resistance scale. (b - lower graph) Derivative showing the successive linear $R(H)$ behavior obtained between the integer matching fields.

where η is the vortex friction parameter. The resistivity of the sample is by definition : $\rho = \frac{E}{j}$ which leads to $\rho = \frac{B\phi_0}{\eta}$. Thus, the resistivity depends linearly on the magnetic field as observed experimentally in figure 3.21(a). This linear dependence of the $R(H)$ curve is due to the linear increase of the number of moving vortices with increasing magnetic field. The change of the slope of the magnetoresistance curve at each matching effect is due to the change of the flow regime induced by the change of the positional order of the vortices. At fields higher than $H_{2.5}$, the linear dependence on the magnetic field is lost. We believe that for such high fields, the number of vortices becomes too large to preserve matching effects and also that the resistance corresponds to a non-negligible fraction of the normal state resistance ($R_n \approx 250\Omega$).

3.4.3 Conclusion of section 3.4

The superconducting properties of thin Nb films deposited by MBE on the barrier layer side of AAO templates have been discussed. Six different samples have been studied, consisting in 30 and 50nm thick Nb films deposited along three different angles (0°, 30° and 60°) from the

normal to the surface. The perpendicular deposition of Nb doesn't lead to significant matching effects compared to those previously observed for sputtered NbN thin films. In contrast, the deposition at 30° revealed significant matching effects for both thicknesses. In this case, the lattice of pinning centers results from shadowing effects due to the bumpy surface. Therefore, blind holes are formed close to the interstitial sites. Due to the directional deposition, the hexagonal symmetry is partly broken and the lattice of pinning center is a double triangular lattice with different pinning strengths instead of an hexagonal array. Finally, different flux flow regimes have been observed at high field ($H > H_1$), corresponding to the motion of interstitial vortices.

3.5 General conclusion of chapter 3

In this chapter, we have investigated the different possibilities that offer anodized aluminum oxide templates to create periodic lattices of pinning centers in superconducting thin films. In addition to the triangular array of antidots created by deposition of thin films on the porous face of the template, we have shown that the barrier layer side of the template, which is generally considered as undesirable (and more frequently eliminated to make porous alumina membranes) can be used to create hexagonal and double-triangular lattices of pinning centers. The magnetoresistance curves for the three different pinning center lattice symmetries are compared qualitatively in figure 3.22.

In the first section, we have investigated the superconducting properties of NbN thin films deposited by sputtering on the triangular array of holes, i.e. containing a triangular array of antidots. We have shown that the matching effects can be strongly enhanced by the appropriate tuning of film thickness and hole diameter. First, we briefly discussed the results obtained on a sample made considering pore interdistance of 100nm and pore diameter of 60nm. Then, a sample made considering pore interdistance of 50nm and pore diameter of 30nm was more deeply studied. At high temperatures, the one-dimensional wire network behavior was observed due to the narrow holes spacing (about 20nm). Only four matching effects were observed due to the short-range order of the lattice. At lower temperature, a cross-over to the vortex regime was observed due to the decrease of the coherence length. In this case, only matching effects at H_1 and $H_{1/2}$ were observed.

In the second section, we have investigated the properties of NbN films deposited by sputtering on the barrier layer face of AAO templates. In this case, pinning centers are created at the interstitial sites

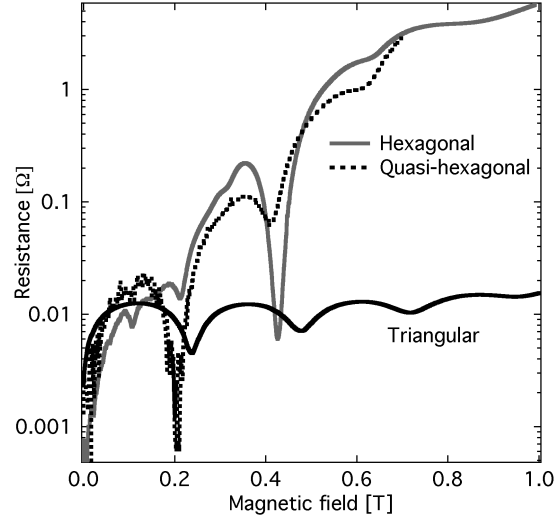


Figure 3.22: Comparison of the magnetoresistance curves of the three different lattice symmetries obtained using the AAO templates. Only the trend of the curves should be compared since the conditions are not identical.

of the lattice of bumps due to local disorder in the film. The pinning centers form an hexagonal array and are twice more numerous than the holes on the other face of the corresponding alumina template. Therefore, twice more vortices can be pinned compared to the case of the pinned triangular lattice of antidots. At high temperatures, a sharp matching peak is observed at H_1 and three smaller peaks are seen at $H_{1/4}$, $H_{1/2}$ and $H_{3/4}$. Matching effects are also observed at $H_{1.5}$ and H_2 . At low temperatures, the matching effect at H_2 becomes more pronounced and a matching effect at H_3 is also visible. We investigated in two ways how the matching features evolve while varying the pinning strength. First, an Si insulating layer (25nm) was deposited prior to the NbN deposition in order to flatten the surface. In this case, the fractional matching effect at $H_{1/2}$ is favored. This is due to the enhanced vortex-vortex repulsive force compared to the vortex-pinning center attractive force which favors the triangular arrangement. Second, different thicknesses of NbN, ranging from 10 to 30nm, have been deposited on the barrier layer. Similarly, an enhancement of the $H_{1/2}$ peak is observed for increasing thicknesses.

In the third section, the superconducting properties of Nb thin films deposited by MBE on the barrier layer of alumina template were investigated. Six different samples were studied by varying the thickness (30 and 50nm) and the angle of deposition (0° , 30° and 60°) from the normal to the surface. For the 0° deposits, no clear matching effects have been

observed. Therefore, the Nb deposited by MBE is less sensitive to the sharp angles present at the interstitial sites. On the contrary, the deposits at 30° shows strong matching features for both thicknesses of the Nb film. The pinning center lattice results from thickness modulations (blind holes) located close to the interstitial sites due to shadowing effects. In this case, the pinning center lattice is not hexagonal but double triangular. This results from the dependence of the shadowing effect on the direction of the deposit. The global behavior of such array is similar to the one of an hexagonal array with low pinning strength. At fields higher than H_1 and at low temperature, different flux flow regimes (linear magnetoresistance) are observed between the successive matching fields.

As a final conclusion, the major advantage of the barrier layer surface is the twice larger number of pinning centers compared to the hole surface. However, contrary to the triangular array of holes, it is not possible to enlarge the pinning centers on the barrier layer surface and thus allow multi-quanta vortices to be pinned.

Chapter 4

Vortex pinning in NbN thin films using dense ferromagnetic nanowire arrays

4.1 Introduction

4.1.1 Context

In the previous chapter, we considered the core pinning of vortices using alumina membranes. In this chapter, we will discuss the possibility to use these membranes to perform magnetic pinning of vortices. The study of the magnetic interaction between ferromagnets and a superconductor has attracted a lot of attention during the last decade. The way has been paved by Otani and coworkers [109], who investigated the superconducting properties of a Nb thin film with a superposed GdCo dot array with in-plane magnetization (i.e. parallel to the superconducting film). The stray magnetic field of the magnetic dots was acting as pinning center for the vortices. Since then, many experimental [8–11, 71, 72, 111, 129–132] and theoretical [83, 133–136] works have been performed on ferromagnets-superconductors hybrids¹. Notably, Hoffmann et al. [71] compared experimentally the vortex pinning in superconducting films deposited on top of square arrays of magnetic and non-magnetic dots of different sizes. The enhancement of the superconducting properties due to the presence of the stray field of the

1. for a review see Vélez et al. [81], Lyuksyutov et al. [82] and Aladyshkin et al. [137].

ferromagnetic dots rather than to a local thickness modulation has been proved. Indeed, for equal size of the dots, the number and the amplitude of the matching effects were more important with ferromagnetic dots. Another significant advance was made by Lange et al. [9] who obtained field-induced superconductivity (FIS) in similar hybrid structures than Hoffmann et al. The shift of the corresponding phase diagram $T_c(H)$ takes its origin in the creation of vortex - anti-vortex pairs (V-AV) in the superconductor due to the stray field of the dots. Gillijns et al. [129] tuned this field-induced superconductivity by modifying the magnetic state of the ferromagnetic dots. They were able to modify the number of V-AV pairs generated by each dot and therefore to shift at will (by discrete steps) the phase diagram up to a maximum value. Recently, Villegas et al. [111] manipulated the superconductivity of an Al film using a very dense array of ferromagnetic dots synthesized using porous alumina membranes. This opens new possibilities to achieve ferromagnets/superconductors hybrids avoiding the lithographic techniques. However, the small dimensions of the magnetic dots limit the strength of the magnetic interaction between the superconducting film and the array of dots.

4.1.2 Motivation

However, the majority of the studies considering ferromagnet - superconductor hybrids were either limited to low magnetic field or to low T_c superconductors. Therefore, an hybrid ferromagnet-superconductor system allowing a strong magnetic pinning of vortices at high field and for high T_c superconductors is still missing. Also, it is not clear yet, whether strong vortex pinning can be achieved using very dense pinning center arrays. Indeed, the overlap of the magnetic field between neighboring magnetic structures and the overlap of the screening currents in the superconductor should lead to the disappearance of the vortex pinning for very dense arrays [138].

In this chapter, a new approach to create strong vortex pinning at high fields of NbN thin films using very dense arrays of ferromagnetic nanowires ($5.7 \cdot 10^9 \text{cm}^{-2}$) is demonstrated. For this purpose, an AAO membrane is filled with Ni using electrodeposition and a NbN film is placed nearby the extremities of the ferromagnetic nanowires (see figure 4.1). This technique allows us to avoid lithographic techniques and to obtain lattices with much smaller periodicities.

The chapter is organized as follows : in section 4.2, the magnetic properties of the dense Ni nanowire arrays are discussed. Then, the influence of the surface corrugation of the alumina membrane on the supercon-

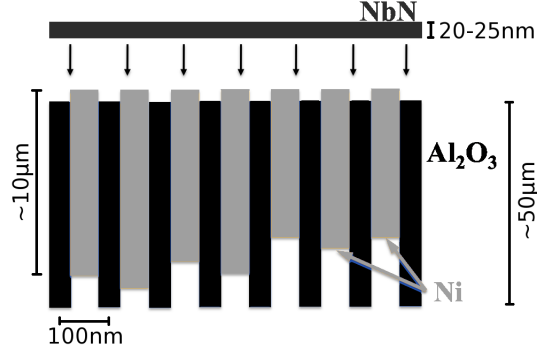


Figure 4.1: Schematic representation on the NbN deposition on the surface of an alumina membrane filled with Ni. The dimensions of the different components are indicated.

ducting properties is discussed in section 4.3. The phase diagram of superconductivity of non-perforated films is discussed in section 4.4. In section 4.5, the vortex pinning properties as a function of the magnetic state of the Ni nanowire array are analyzed. Finally, the conclusions are drawn in section 4.6.

4.2 Magnetic properties of the Ni nanowire array

4.2.1 General properties

The magnetic properties of such dense array of nanowires are complex and depend on numerous geometrical parameters as well as on the nature of the ferromagnetic material. Therefore, it is far beyond the scope of this thesis to make an exhaustive overview of the underlying topic. In this subsection, we present the basic properties of the Ni nanowires in order to give to the reader the basis to understand the properties of our ferromagnet-superconductor hybrid. The reader is referred to specialized literature for the details see references [76–78, 139, 140] and references therein.

The general properties of ferromagnets are dictated notably by the domain wall thickness d_w . In order, to reduce the magnetostatic energy, the magnetization splits into domains with different orientations. Two domains are separated by a domain wall in which the spins progressively switch from one orientation to the other. The domain wall width results from a compromise between the exchange energy (A_{ex}) and the anisotropy energy (K_1) and is given by the formula : $d_w = \pi\sqrt{A_{ex}/K_1}$ (see

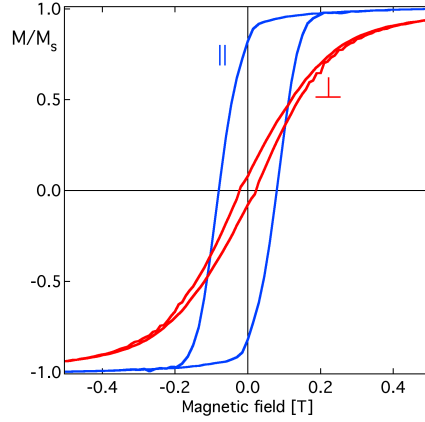


Figure 4.2: (a) Magnetization hysteresis loop for sample NbNNW20(40,100) at $T=15\text{K}$ with the external magnetic field parallel (blue curve) and perpendicular (red curve) to the nanowire axis. The packing of the nanowire array is $P = 14.5\%$. M_s is the magnetization at saturation.

subsection 1.2.3.4, page 36). Nanowires with diameters thinner than the domain wall thickness will exhibit a single-domain behavior. This limit corresponds approximately to a diameter of 100nm for Ni ².

Single- and multi-domain nanowire arrays exhibit very different magnetic behaviors. The multi-domain nanowires present a very low remanence, a low coercive field and a high saturation field. The magnetic state is dictated by the complex reorganization and rotations of the magnetic domains in the nanowires which are driven by both the external magnetic field, the stray fields of neighboring nanowires and the exchange interaction. On the contrary, the single-domain nanowires have a bistable state corresponding to a magnetization vector either parallel or anti-parallel to the wire axis. Therefore, the magnetization hysteresis is dictated both by the external field and the dipolar interactions between neighboring nanowires. Figure 4.2 shows the magnetization curves for sample NbNNW20(40,100) at $T=15\text{K}$ with the field perpendicular ($\perp$) and parallel ($\parallel$) to the nanowires axis. For a parallel field, a high remanence ($0.82M_s$), a coercive field of 0.08T and a saturation field of 0.21T are observed. In perpendicular field, a low remanence (8%), a coercive field of 0.02T and a saturation of almost 1T is observed. These striking differences in the magnetization hysteresis loops for parallel and perpendicular magnetic fields are due to the strong shape anisotropy and dipolar coupling that occur in such dense array of single domain nanowires.

2. Note that it is considerably smaller for Co , around 15nm

4.2.2 Array of bistable ferromagnetic nanowires

In this work, we only report on the magnetic interaction between a superconducting film and an array of single-domain nanowires. Therefore, we will discuss in more details the behavior of these single-domain nanowire arrays.

The magnetic anisotropy of an array of thin magnetic wires results from the interplay of several contributions to the anisotropy [77]. In the case of a single domain wire, three contributions should be considered : (1) the macroscopic demagnetization field, (2) the shape anisotropy and (3) the magnetocrystalline anisotropy energy. For Ni, the magnetocrystalline anisotropy can be neglected [76]. The shape anisotropy is very important and tends to align the magnetization parallel to the nanowires axis. The demagnetization field is also dependent on the shape of the ferromagnet. When the magnetic field is applied parallel to the nanowires axis, the demagnetization field is zero. However when nanowires are placed close to each other, a demagnetization field can be imputed to the array due to the dipolar coupling. This demagnetization field is proportional to the packing of the array [139]. Therefore, the magnetic anisotropy decreases with the packing (P) of the array³.

The magnetization hysteresis loop for sample NbNNW25(60,100) at 15K is presented in figure 4.3 for a parallel magnetic field. Schematic representation of the magnetic state of the array are indicated for several points of the loop. At high positive magnetic field, the magnetization of all the nanowires is aligned with the magnetic field (point A). When decreasing the field intensity, due to large dipolar coupling, the magnetization of some nanowires starts to reverse (point B). Therefore, in the remanent state (point C) the magnetization is not at its saturation value (in this case $M_r \approx 0.6M_s$). The coercive field is reached at about -0.09T (point D). The external field must be increased up to -0.3T to reach again the saturation (point E). The similar behavior is obtained while sweeping the field in the opposite direction leading to the hysteresis curve. This hysteresis reflects the magnetic energy that should be transferred to the ferromagnetic array in order to complete the cycle.

In order to characterize the different magnetic states of the Ni nanowire array, a MFM measurement was performed on the surface for sample NbNNW20(70,100) in regions without the NbN deposit. Although, the measurement was performed at room temperature, it reflects quite well the low temperature behavior because the Ni nanowires are also single-domain at 300K. The magnetic state in the remanent state is

3. $P = \frac{\pi}{2\sqrt{3}} \left(\frac{D}{d}\right)^2$, with D the diameter of the nanowires and d their interdistance.

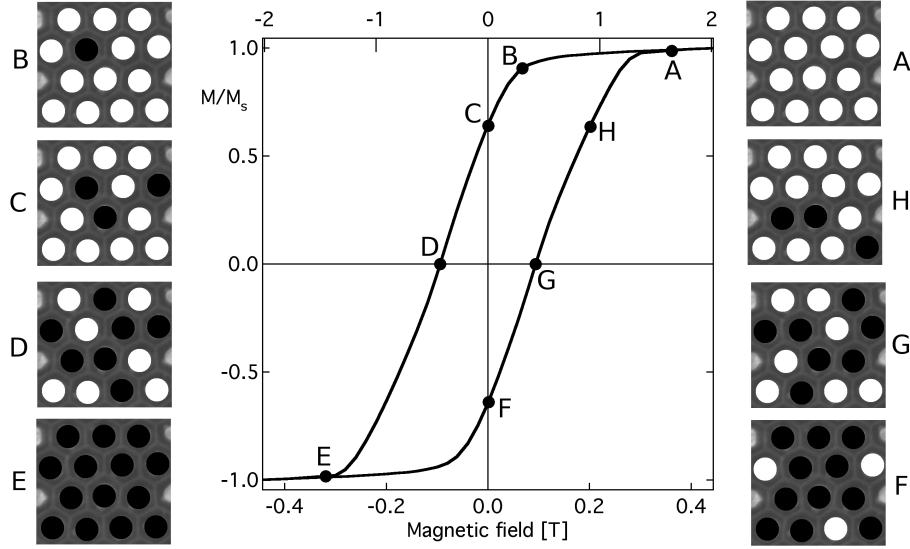


Figure 4.3: Magnetization hysteresis loop for sample NbNNW25(60,100) (central graph) at $T=15\text{K}$ in parallel magnetic field. The left and right panels show schematic representations of the local magnetic state of the array at different points of the cycle. The packing of the nanowire array is $P = 33\%$. M_s is the magnetization at saturation.

shown in figure 4.4(a)⁴. This image is the superposition of an AFM image and a MFM image taken at the same location. The AFM is the back image. Some basic image treatment was performed on the MFM image before the superposition. The image has been transformed from a grayscale image to a black and white image. Then, the black pixels were made fully transparent while the white pixels were made 50% transparent. This allows us to see the perfect match between the magnetic poles and the nanowires location, thus proving the single-domain state. Unfortunately, it was not possible to image the magnetic state of the array with an external magnetic field⁵. This may result from the high packing of the array as well as the resolution of our MFM equipment. However, the magnetic state at different position on the hysteresis loop of a Ni nanowire array with diameter 30nm and interdistance 100nm was stu-

4. Measurement performed by W. Gillijns at KUL.

5. We tried to image the magnetic state of the array at different positions of the hysteresis loop : first, the sample was fixed on a calibrated amount of small magnets in order to apply a magnetic field to the sample during the measurement. Then, the saturation field was applied to the sample using a 1T electromagnet. Finally, the MFM measurement was performed on the sample scotched on the magnets. Unfortunately, no MFM contrast was obtained with this procedure.

died by Nielsch et al. [78]. Figure 4.4(b) shows the magnetic state at the coercive field, where both alignment are equally present. A complicated pattern of clusters of nanowires with the same orientation is present. This clustered pattern is similar to the one observed in figure 4.4(a) for sample NbNNW20(70,100) because of the low remanence of our sample ($M_r/M_s \approx 0.3$).

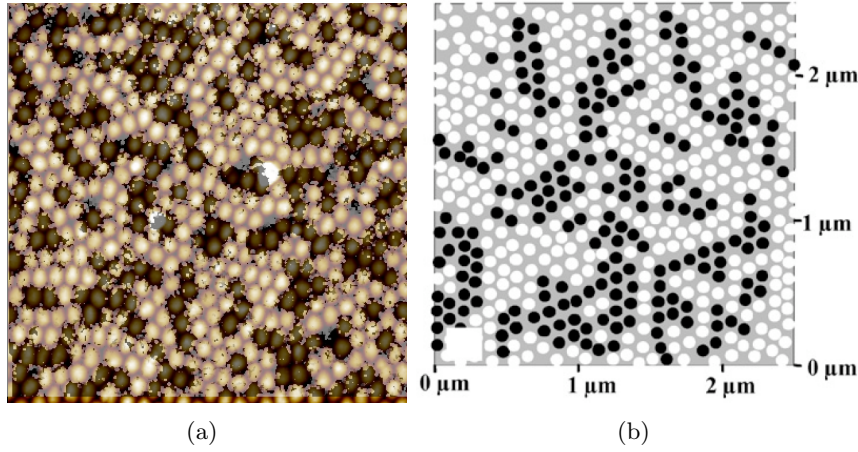


Figure 4.4: (a) Superposition of an AFM and MFM measurement performed at room temperature showing the magnetization arrangement in the remanent state for sample NbNNW20(70,100) ($P = 44\%$). With dots are parallel to positive field. (b) MFM measurement taken from Nielsch et al. [78] at the coercive field of a triangular array of Ni nanowires with interdistance 100nm and diameter 30nm ($P = 8\%$). A clustered arrangement of the Ni nanowires with the same magnetization orientation is observed in both cases.

4.2.3 Influence of the packing and minor M-H cycles

The packing of the array has a major influence on the magnetization hysteresis loop. The higher the packing, the higher the dipolar interactions in the array and the lower the effective field H_{eff} . Therefore, the key properties of such array of ferromagnets, i.e. the remanence, the coercivity and the saturation field, are expected to strongly depend on the packing. Figure 4.5(a) represents the magnetization curves of nanowire arrays with different packing. Both samples are Ni nanowire array in an alumina membranes with an interdistance of 100nm but one has a nanowires diameter of 50nm (green curve; $P = 23\%$) and the other of 70nm (blue curve; $P = 44\%$). The sample with the larger packing has a higher saturation field because the inter-wire coupling is stronger. For the same reason, it leads to a lower remanence. In contrast,

for the low packing sample, the hysteresis is closer to the one expected for non-interacting nanowire array (square cycle). It is clear that these different magnetization cycles impact differently on the properties of superconducting thin films.

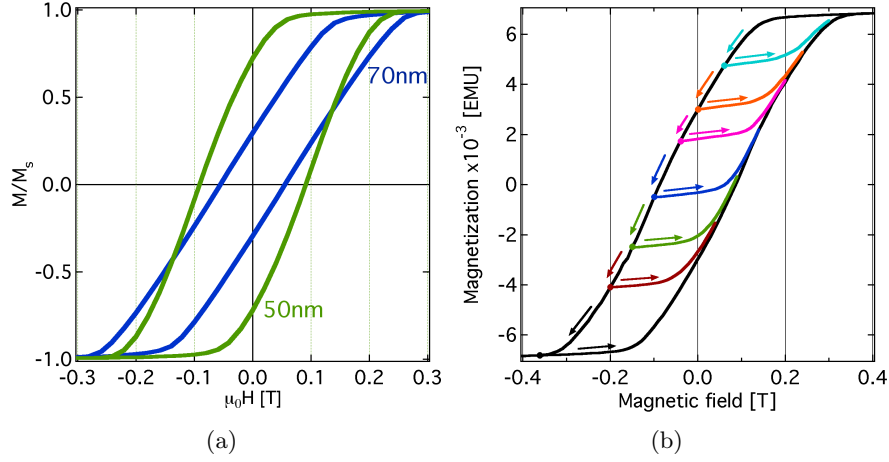


Figure 4.5: (a) Magnetization hysteresis loops normalized to the saturation magnetization for two Ni nanowire array with different packing. The green curve corresponds to sample NbNNW20(50,100) ($P = 23\%$) and the blue curve to sample NbNNW20(70,100) ($P = 44\%$). (b) Magnetization hysteresis loops and minor cycles for sample NbNNW20(60,100)#2 ($P = 33\%$). The magnetic field sweep direction is indicated by arrows.

Intermediate magnetic states can be reached by performing minor cycles. These cycles are obtained by sweeping the field back and forth from the saturation field down to an intermediate field in the hysteretic part of the magnetization curve. Several minor loops are represented in figure 4.5(b). New magnetic states of the array are reached in the inner part of the loop which allow us to study the impact of the magnetic state of the array on the superconducting film at a given magnetic field. Moreover, these new magnetic states are relatively stable over a certain field range fixed by the two major branches. Indeed, the magnetization only slightly varies in these regions. Therefore, such features also allow us to study the impact of the external magnetic field on the superconducting properties for a fixed magnetic state of the Ni nanowire array.

4.3 Influence of the surface topography

As was discussed in subsection 2.2.4 (page 52), the surface topography can be modified by a selective chemical etching of the alumina mem-

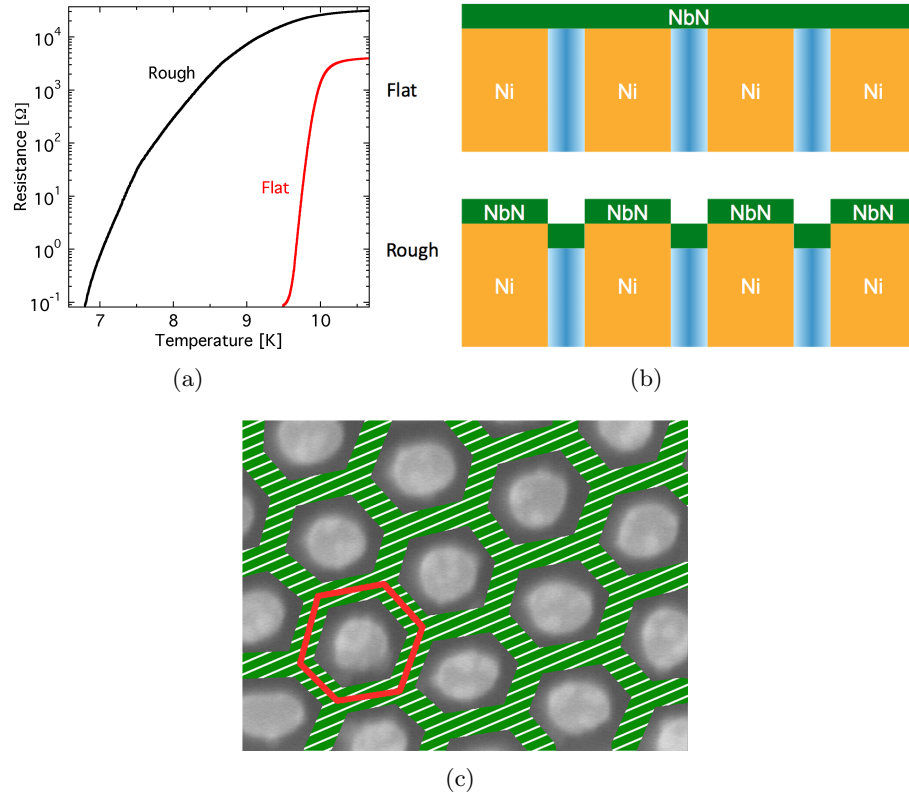


Figure 4.6: (a) Resistance as a function of the temperature for sample NbNNW20(70,100) with a rough surface (black curves) and with flat surface (red curves). The resistance is measured with a dc current of $10\mu A$ in both case. (b) Schematic representation of an ideal NbN deposit on the flat and rough substrates. The red curve corresponds to the upper scheme and the black curve to the lower scheme. (c) Schematic representation of the conductive region of the NbN deposit on the rough surface (green - white hatched region). The conductive region is much reduced in the case of rough surface.

branes using a 0.2M NaOH solution. The resulting relief has a strong impact on the pinning properties, mainly for two reasons : first, it creates core pinning (antidots or blind holes). Second, it changes the magnitude of the magnetic interaction between the ferromagnetic nanowire array and the superconducting thin film. The superconducting transitions for sample NbNNW20(70,100) with a flat surface and with a rough surface (5 minutes of etch) are schematically represented in figure 4.6(a). Each curve was measured with a dc current of $10\mu A$ at zero field with the nanowire array in the remanent state. In the case of the rough surface, the NbN thin film is perforated by the Ni nanowire array. Indeed, a strong increase of the normal state resistance (a factor 8) is observed. This increase is related to the reduction of the section in which the current can flow as is schematically shown in figures 4.6(b) and 4.6(c). The deterioration of the structural quality of the deposit is also responsible for the increase of the resistivity of the film as is suggested by the decrease of the RRR (residual resistivity ratio), the decrease of the critical temperature and the increase of the superconducting transition width. The effective conducting region strongly depends on the etching time and much less on the nanowires diameter as schematically represented in figure 4.7. Indeed, the inhomogeneous etching speed of the alumina (much faster near the nanowires) reduces the effective superconducting region of the NbN deposit. This is particularly pronounced for the smallest diameter (as represented in figure 4.7(b), the red dashed frame highlights the effective superconducting region) for which a hole can be present between the NbN deposit and the Ni nanowires.

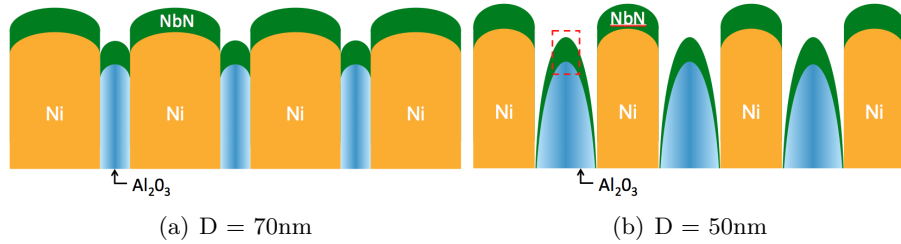


Figure 4.7: Schematic representation of a 20nm thick NbN deposit on AAO substrates filled with Ni nanowires after partial chemical etch (0.2M NaOH) of the alumina membrane. (a) $D = 70\text{nm}$ and (b) $D = 50\text{nm}$. For $D = 50\text{nm}$ (b), the alumina in contact with the Ni nanowires is etched much faster than for $D=70\text{nm}$. Therefore, the effective superconducting NbN section is smaller than expected (as indicated by the red dashed frame).

Magnetostatic simulations have been performed in order to determine the stray field profile and amplitude of the Ni nanowire array in

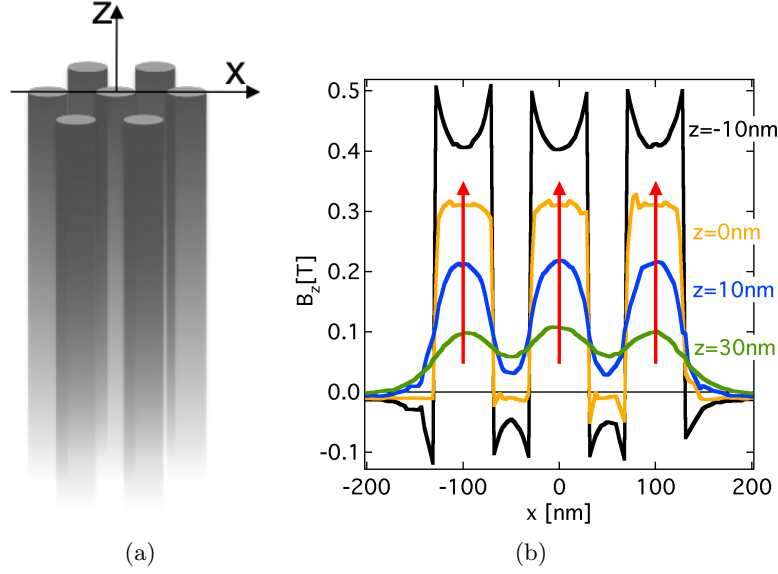


Figure 4.8: (a) Schematic representation of the 7 nanowire array used for the magnetostatic calculation of the stray magnetic field. Nanowires have a diameter of 60 nm, a length of 1.2 μm and an interdistance of 100 nm. The axis system is also represented. (b) z -component of the stray field along the x -axis of the Ni nanowire array represented in figure 4.8(a) for several z values ($z = 0\text{ nm}$ corresponds to the nanowire tips). The arrows show the nanowire location along the x -axis as well as the direction of magnetization.

the NbN thin film for the different surface corrugations. We considered a triangular lattice of cylinders with interdistance $d = 100\text{ nm}$ and diameter $D = 60\text{ nm}$ with magnetization fixed to $\vec{M} = M_s^{Ni} \cdot \vec{e}_z$ with $M_s^{Ni} = 5.1 \cdot 10^5\text{ A/m}$. A schematic representation of the model system is shown in figure 4.8(a) for 7 nanowires with length of 1.2 μm . Simulations were also performed considering up to 80 nanowires. The increase of the number of nanowires leads to a decrease of the positive stray field on top of the nanowires and to an increase of the negative field in-between the dots. Different calculations have been performed for different lengths of the nanowires. For wire length larger than 600 nm (i.e. 10 times the diameter), no significant modification of the stray field is observed. Figure 4.8(b) shows the B_z component of the stray magnetic field along the x direction, i.e. along a row of nanowires, for different distances z from the nanowire tip. For the NbN film perforated by the Ni nanowires, the magnetic field profile corresponds approximately to the $z = -10\text{ nm}$ case (see figure 4.8(b)). In this case, the magnetic flux above each nanowire is about $\phi \sim 0.6\phi_0$ (see appendix A.1, page 153).

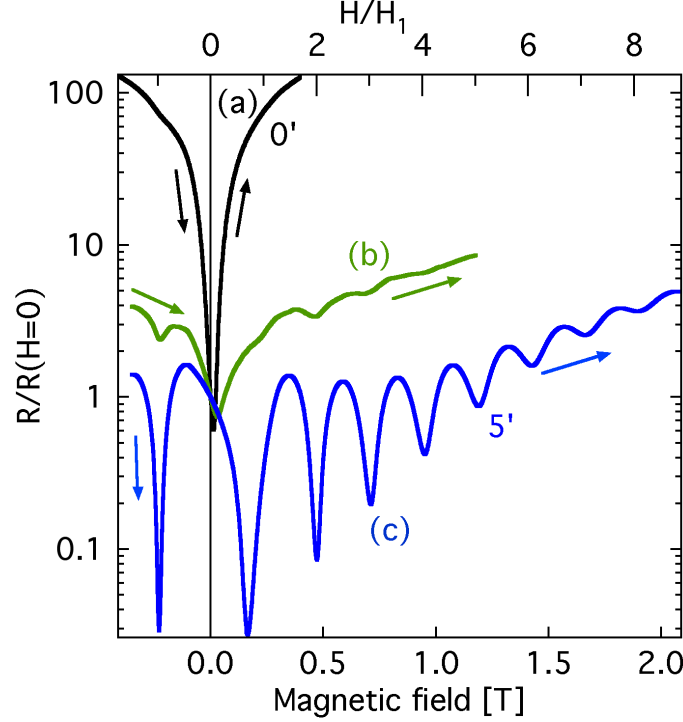


Figure 4.9: Magnetoconductance curves for sample NbNNW20(60,100)#1 for different corrugation of the surface. The measurement were performed at temperatures close to the onset of resistance (T_c^{onset}) with $I_{DC} = 10\mu A$. The field was swept from negative to positive fields. The curves were normalized to their zero field resistance $R(H=0)$ and were all lying in the range $2 - 5\Omega \sim 10^{-3}R_n$ with R_n the normal state resistance. The corrugation of the surface increases from (a) to (c). Curve (a) corresponds to a flat surface (no etch) while (c) corresponds to a perforated film (5' etch). Curve (b) corresponds to intermediate corrugation obtained with the polishing with Syton.

It is clear that in the case of perforated films, the magnetic interaction between the stray field of the nanowires and the vortices is not the only pinning mechanism as core pinning of the vortices can not be neglected. Holes are always attractive for the vortices (core pinning) while the magnetic interaction can be either attractive or repulsive depending on the magnetization orientation. Therefore, tuning the magnetic pinning and/or the core pinning is expected to modify the superconducting properties of the NbN film. Unfortunately, magnetic and core pinning cannot be modified independently because they both depend on the level of perforation.

The magnetoconductance curves for sample NbNNW20(60,100)#1 with different surface corrugation is shown in figure 4.9. The measurements were performed at temperatures close to the onset of resistance (T_c^{onset})

with $I_{DC} = 10\mu A$. The field was swept from negative to positive fields. The curves were normalized to their zero field resistance $R(H=0)$ (all $R(H=0)$ values are in the range $2 - 5\Omega \sim 10^{-3} - 10^{-4}R_n$ with R_n the normal state resistance). It should be emphasized that the same substrate have been used to elaborate the different samples in a successive way. After each full characterization, the substrate was polished once again with water to flatten the surface. Then the chemical etch with NaOH was performed during a different time to modify the surface corrugation. The corrugation of the surface increases from (a) to (c). Curve (a) corresponds to a flat surface (no etch) while (c) corresponds to a fully perforated film (5' etch). Curve (b) corresponds to intermediate corrugation obtained with the polishing with Syton. Curve (a) corresponds to a flat substrate so-that only the weak magnetic pinning is effective. The magnetic interaction is weak due to the small field profile present in the superconducting film as shown by the magnetostatic calculations (see blue and green curves of figure 4.8(b)). Only a very weak hysteretic behavior is observed which denotes the weak magnetic interaction with the Ni nanowires. The resistance increases very rapidly with increasing field as is observed for continuous thin film without pinning centers. For moderate corrugation, a strong modification of the magnetoresistance curve is observed (curves (b)). A dip is observed at the theoretical first matching field $H_{-1} = -0.235T$. At $H_1 = 0.235T$, only a small change of slope is observed. The resistance at $H = H_1$ is much larger than the one at $H = 0$. When the NbN film is fully perforated by the Ni nanowire array (curve (c)), the matching effects are much more intense and numerous resistance dips are observed as the magnetic field is swept. The resistance drops by almost 2 orders of magnitude at H_{-1} . At H_1 , the peak is now shifted from its theoretical position ($+0.165T$ instead of $+0.235T$). Moreover, the resistances at H_1 and H_{-1} are much lower than at zero field which apparently refers to field-induced superconductivity.

Therefore, the magnetoresistance curves strongly depend on the level of perforation of the film. When varying the perforation, the core pinning and the magnetic pinning of the vortices are modified. In the following of this chapter, we will discuss the case of the moderate surface corrugation corresponding to the case of curve (b). The chapter 5 will be devoted to the particular case of the perforated film (curve c).

4.4 H-T phase diagram

The stray field of the Ni nanowire array strongly impacts on the superconducting transition of the superconducting thin films [9, 72]. In

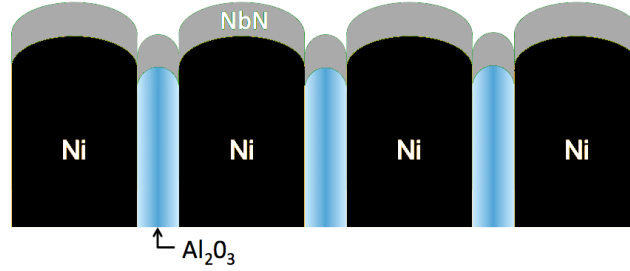


Figure 4.10: Schematic representation a highly corrugated NbN thin film after the deposition on a rough substrate.

in this section we discuss in more details the superconducting transition for sample NbNNW25(75,100) and for its reference sample (i.e. a sample obtained by depositing the same NbN thin film on a sapphire substrate) and its evolution with the external magnetic field. The room temperature resistivity for the sample NbNNW25(75,100) is a factor 6 higher than for its reference sample. For temperatures just above the superconducting transition, this factor increases up to 7. The enhanced resistivity and lower RRR for sample NbNNW25(75,100) compared to its reference sample are due to the higher granularity and large surface corrugation of the membrane. It is worth emphasizing that the NbN thin film is likely to be strongly corrugated but not completely perforated by the Ni nanowire array since the AFM measurement (see figure 2.11(a), page 55) revealed a corrugation of about 15nm.

The superconducting transition for sample NbNNW25(75,100) (with the Ni array in the remanent magnetic state after saturation) and for its reference sample are shown in figure 4.11(a) on a linear resistance scale and in figure 4.11(b) on a logarithmic resistance scale. For both samples, the resistance was measured with a dc current of $10\mu A$. The curves were normalized to the normal state resistance for convenience. A wide transition ($\Delta T_c \sim 3.5K$) is observed for the sample NbNNW25(75,100) in contrast to the sharp transition of the reference sample ($\Delta T_c < 1K$). Also, the transition to the superconducting state is shifted to lower temperature for sample NbNNW25(75,100). The high granularity of the NbN thin film leads to a percolative transition to the superconducting state [141, 142]. This phenomenon is enhanced in the case of the alumina substrate and leads to the broadening of the transition. The critical temperature is therefore very inhomogeneous in the NbN film. The critical temperature of the NbN films, extracted from the mid-transition temperature, are respectively 9.45K and 10.67K for sample NbNNW25(75,100) and for its reference sample. The heterogeneous pro-

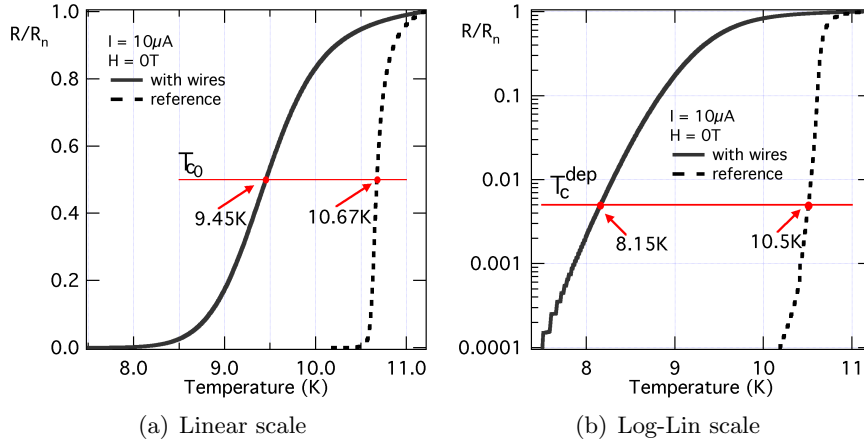


Figure 4.11: Superconducting transition curves for sample NbNNW25(75,100) (solid curve) and for its reference sample (dotted curve). The curves were measured at zero field starting from the superconducting state. The Ni nanowire array for sample NbNNW25(75,100) was in the remanent magnetic state. (a) Linear resistance scale and (b) logarithmic resistance scale. Curves have been normalized to their normal state resistance (R_n) for clarity. The mid-transition temperature is indicated (T_{c0}) as well as the depinning temperature T_c^{dep} ($R = 0.005 R_n$).

properties of granular superconductors (T_c , electron mean free path,...) lead to heterogeneous coherence length and London penetration depth. Therefore particular care should be taken when using these superconducting lengths [143, 144].

The major impact of the nanostructuration appears in the low resistive part of the transition. Indeed, sample NbNNW25(75,100) shows a long tail spreading down to 7.5 K. This is emphasized in figure 4.11(b) showing the superconducting transition in a logarithmic scale on the ordinate. This is very different from the reference sample for which the perfect superconducting state (within our resolution) was reached at 10.1 K. Such long low resistance tail was already observed for NbN films deposited on the porous face of the alumina template (see figure 3.2, page 68) and should therefore be principally attributed to the deterioration of the quality of the NbN film leading to a percolative transition.

Due to the large superconducting transition, the phase diagram of superconductivity for sample NbNNW25(75,100) is strongly influenced by the resistive criterion used to determine it. The critical temperature as a function of the field is represented in figure 4.12(a) for both sample NbNNW25(75,100) and its reference sample using two different criteria. These curves have been extracted from a set of resistance versus temperature curves at different magnetic fields by taking the temperature

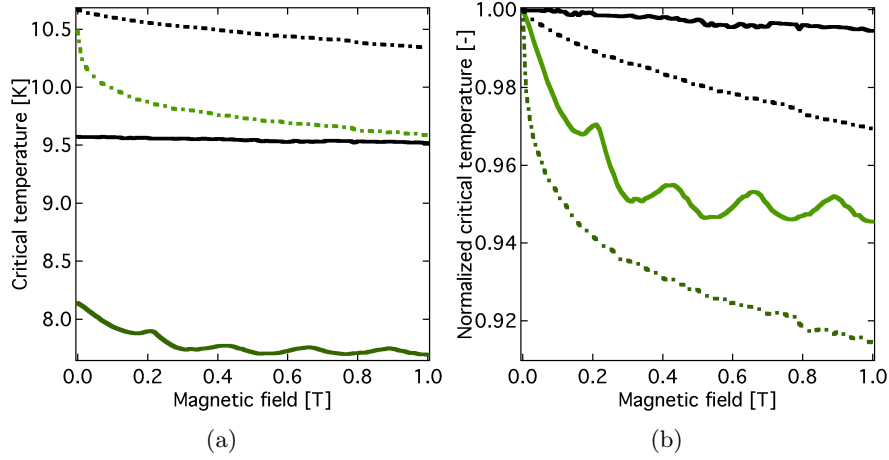


Figure 4.12: (a) Critical temperature as a function of magnetic field (phase diagram) for sample NbNNW25(75,100) (solid curves) and for its reference sample (dotted curves). The green curves are obtained with a low resistive criterion ($R = 5 \cdot 10^{-3} R_n$) probing the onset of vortex motion. The black curves have been obtained considering a mid-transition criterion. (b) Normalized curves to the zero field values. The superconducting state of the reference sample is more affected by the magnetic field.

corresponding to a certain resistance. The black curves correspond to a mid-transition criterion and the green curves corresponds to a $0.5\% R_n$ criterion (R_n is the normal state resistance) i.e. the onset of the resistive state. The dotted lines corresponds to the reference. The use of different criteria allows us to probe different phenomena. Indeed, the onset of resistance is generally associated to vortex motion while a mid-transition criterion is often used to determine the critical magnetic field or temperature.

The mid-transition criterion shows a linear dependence of the critical temperature with the external magnetic field which is the typical bulk behavior (see section 1.1.5). The low resistance criterion shows a more interesting behavior. First, the critical temperature is much more sensitive to the magnetic field. Second, the magnetic field behavior is not linear. Third, oscillations are present for the sample NbNNW25(75,100). These peaks originate from matching effects between the pinning centers array and the vortices. Although a strong decrease of the zero field critical depinning temperature is observed, the Ni nanowire array seems to be efficient for the vortex pinning. Indeed, the critical depinning temperature for sample NbNNW25(75,100) drops less rapidly compared to the the one for its reference sample as indicated by the normalized curves in figure 4.12(b).

Coherence length and penetration depth

From the phase diagram and the normal state resistance, the coherence length and the London penetration depth can be estimated. Using the relations 1.37 and 1.40 of subsection 1.1.5 (page 29), one obtains : $\xi(0) \approx 1.6nm$ for sample NbNNW25(75,100) and $\xi(0) \approx 3nm$ for its reference sample. The coherence length for the reference sample is in agreement with coherence lengths for similar sputtered NbN films obtained by S. Adam et al. at UCL [95]. The lower coherence length for sample NbNNW25(75,100) is due to the higher resistivity of the film [95]. As will be discussed in the next sections, such low coherence length allows to pin multi-quanta vortices in each pinning center.

The London magnetic penetration depth is calculated using equation 1.41 (page 29). One obtains : $\lambda(0) \approx 2\mu m$ using $\rho_N \sim 4000\mu\Omega cm$ and $T_c = 9.57K$. Moreover, a correction should be made for thin film to obtain the effective penetration depth [28] : $\lambda_{eff} = \frac{\lambda^2}{d}$ where d is the thickness of the film, leading to $\lambda_{eff}(0) \sim 200\mu m$. Therefore, the vortices interact with each other and with pinning centers over very large distances. Sample NbNNW25(75,100) is clearly in the extreme type-II limit since $\kappa \sim 10^5$.

4.5 Vortex pinning properties

4.5.1 High field matching effects

The magnetoresistance curve for sample NbNNW25(75,100) at $t = \frac{T}{T_c} = 0.75$ is shown in figure 4.13. The measurement was performed with a dc-current of $10\mu A$, from zero field (in the remanent magnetic state of the Ni nanowire array when coming from positive fields) to 2.5T. Numerous matching effects are present with a period of about 0.225T, in good agreement with the theoretical matching fields for a triangular array of pinning center of 100nm of interdistance : $H_n^\Delta = n \times 0.2388T$. The matching effects are visible up to 2.5T (H_{11}), which is about 1-2 orders of magnitude higher than the matching fields obtained with ferromagnets-superconductor hybrids using conventional lithography techniques [81, 82, 137]. The first matching effect is missing which is related to the magnetic origin of the vortex pinning and will be discussed in the next subsection.

Generally, such numerous oscillations are attributed to the collective regime (1DWN limit, see subsection 1.13, page 32 and references [71, 72]). This collective regime of superconductivity appears when the inter-space between the pinning center is of the order of 2ξ . In fact, this limit is rea-

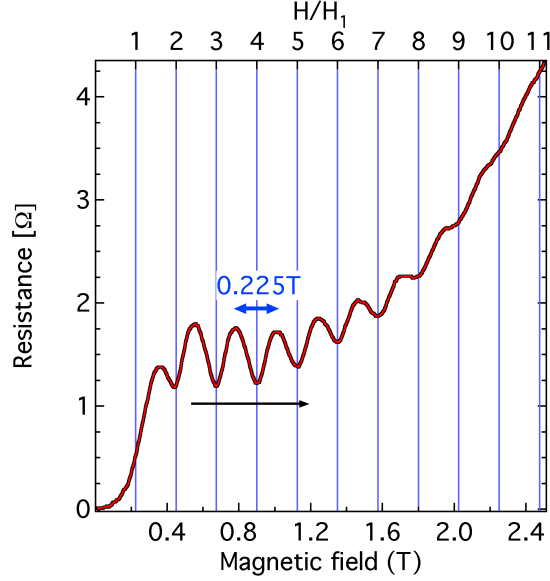


Figure 4.13: Magnetoresistance curve for sample NbNNW25(75,100) at 7K ($t = \frac{T}{T_c} = 0.75$) and $I_{DC} = 10\mu A$. The field was swept from zero field (in the remanent magnetic state of the Ni nanowire array when coming from positive field) to positive fields.

ched for all samples close to T_c due to the diverging dependence of ξ with temperature. In this case, vortices cannot be placed in-between the pinning centers (interstitial vortices) due to the small dimensions of the superconductor compared to the coherence length. Taking the previously calculated value of $\xi(0) = 1.6nm$ and a strand width of 25nm ($w_s = d - D$), this condition becomes : $t = T/T_c > 0.987$. This is far above the temperature of the magnetoresistance measurement ($t = 0.75$) shown in figure 4.13. Indeed, at $t=0.75$, the coherence length is about 3.2nm. The choice of T_c (i.e. the choice of the resistance criterion) doesn't change the conclusion that the collective regime is not present. Indeed, choosing the appearance of resistance at zero field with a small current as critical temperature, the critical temperature becomes about 7.5K as seen on figure 4.11(b). In this case, the reduced temperature is $t = 0.93$, which is also below the 1DWN limit condition. Moreover, the critical temperature of a 1D wire network has a very different temperature dependence compared to the bulk superconductor. It is expected to display a quadratic dependence with temperature : $1 - \frac{T_c}{T_{c0}} \propto H^2$ (see subsection 1.13, page 32). This quadratic behavior is not observed in the critical temperature curves (see figure 4.12). Therefore, the collective regime can be ruled out as the origin of the observed matching

effects.

The magnetoresistance oscillations thus result from matching effects between the vortex lattice and pinning center array [4, 5]. Numerous matching effects are attributed to giant vortices pinning in each pinning center. The magnetic pinning, i.e. the magnetic interaction between the vortices and the ferromagnetic nanowires, is the preponderant factor of the vortex pinning. Core pinning is also present in our hybrid system due to the strong surface corrugation but it appears to be lower than the magnetic pinning as will be discussed in the next section. Core and magnetic pinning have been extensively studied, however, while analytic results about the maximum number of vortices that can be pinned by core pinning have been derived [45, 46, 145], their equivalent in the case of magnetic pinning is still missing. This is clearly due to the difficulty to make valid approximations since the number of parameters is much larger than for core pinning. Therefore, we first use oversimplified theories and then discuss the results.

The stable vortices configurations in superconducting films with a triangular lattice of pinning centers have been widely studied both theoretically [52, 53, 146] and experimentally [12, 73, 147]. The advantage of the triangular arrangement of pinning centers compared to other symmetries, is that, at the first matching field, the energy of elastic deformation of the vortex lattice is zero. For small pinning centers, interstitial vortices appear when the field is higher than H_1 (see subsection 5.1, page 131). As a result, a strong decrease of the superconducting properties is observed due to the high mobility of these vortices. In contrast, large pinning centers can pin more than one vortex. Using the model of Buzdin [44] (see subsection 5.1, page 131), which is based on a triangular lattice of columnar defects, one can determine the minimum diameter for which a vortex lattice with vorticity 2 can be pinned. This corresponds to $D^3 > 8\xi^2(t)d$, with d the columnar defect interdistance. $D = 17nm$ is found for $t = 0.75$ ($T_c = T_c^{mid}$) and $D = 33nm$ for $t = 0.95$ ($T_c = T_c^{dep}$). Thus, in both cases the minimum diameter for multi-quanta vortex stability is lower than the diameter of the nanowires. As expected, multi-quanta vortices are likely to be pinned at each pinning centers. The maximum number of vortices that can be pinned in an isolated columnar defect (the so-called saturation number) has been calculated by Mkrtchyan and Schmidt [46]. This saturation number is given by $n_s(t) = \frac{D}{4\xi(t)}$ where D is the diameter of the hole and $\xi(t)$ the coherence length (t is the reduced temperature). For $D = 75nm$, at $t = 0.75$, one obtain : $n_s(0.75) \approx 6$. However, up to 10-11 matching effects are observed experimentally (figure 4.13). Doria et al. [45] have extended the calculation of Mkrtchyan and Schmidt to

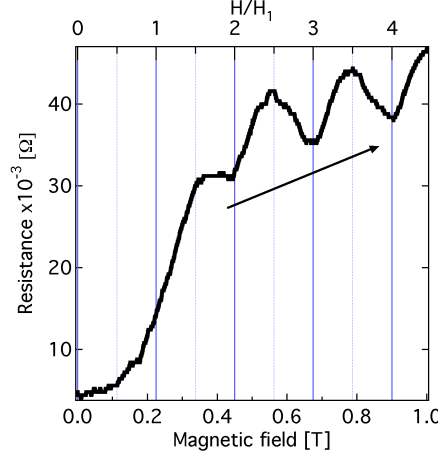


Figure 4.14: Magnetoresistance curve for sample NbNNW25(75,100) at $t = \frac{T}{T_c} = 0.53$ and $I_{DC} = 250\mu A$. The field was swept from zero field (in the remanent magnetic state of the Ni nanowire array when coming from positive field) to positive fields.

the case of array of columnar defects and magnetic defects. The presence of interstitial vortices was also considered. First, they found that the repulsive interaction between the vortices can lead to higher saturation number than for isolated columnar defect. Second, in the case of magnetic defects the saturation number was always found higher. Therefore, the saturation number found using the Mkrtychyan and Schmidt model very likely underestimates the real saturation number. This should be particularly pronounced in our case since the inter-vortex interaction is strong ($\lambda \gg d$). Therefore, the magnetic pinning allows to pin more vortices at each pinning center in agreement with the calculations of Doria et al.

The magnetoresistance curve for sample NbNNW25(75,100) is represented in figure 4.14 ($t = \frac{T}{T_c} = 0.53$ and $I_{DC} = 250\mu A$). Remarkably, the matching effects are still well pronounced at low temperature. This result indicates that the pinning force due to the ordered array of ferromagnetic nanowires is large because, generally, matching effects vanish at low temperature due to the appearance of intrinsic pinning [6, 39, 70, 73, 115] and of the Bean critical state [47]. At lower temperature, the saturation number is larger and thus multi-quanta vortex pinning is favored.

4.5.2 Hysteretic magnetoresistance

The low field hysteretic behavior is the major feature of a ferromagnet. In this subsection, we discuss how the hysteretic behavior of the nanowire array is transferred to the superconducting film.

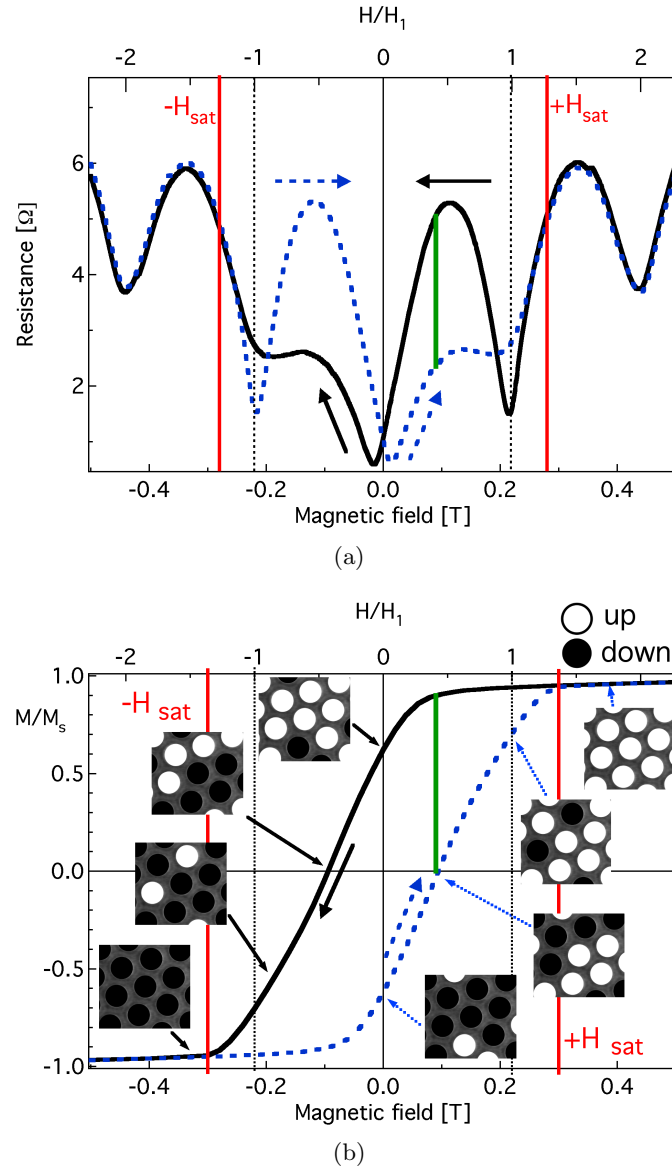


Figure 4.15: (a) Magnetoresistance curve for sample NbNNW25(60,100) at $t=0.8$ with $I_{DC} = 10\mu A$ in the low field region. The field sweep direction is indicated by the arrows. (b) Magnetization hysteresis loop for the same sample at $T=15K$. Schematic representations of the local magnetic state of the Ni array are shown for several points of the hysteresis loop.

The magnetoresistance curve for sample NbNNW25(60,100) at $T/T_c = 0.8$ is shown in figure 4.15(a). The external field was swept from positive to negative fields (solid curve) and then back to positive fields (dotted curve). An hysteretic behavior is clearly observed at low field, demonstrating the strong influence of the magnetic state of the nanowire array on the superconducting properties of the NbN film.

Figure 4.15(b) shows the magnetization hysteresis loop measured at $T=15\text{K}$. Schematic representations of the magnetic state of the nanowire array are indicated for several particular points of the hysteresis loop. The reversal of the magnetization of the entire array from one saturated state to the other corresponds to the successive reversal of individual nanowires. In the remanent state, about 19% of the nanowires already experienced a magnetization reversal due to the strong interwire dipolar couplings (see figure 4.15(b)).

At the first positive matching field ($H_1 = 0.22T$), a difference in the overall magnetization state of the nanowire array leads to a change in the resistive state of the superconducting thin film. Indeed, as the field is reduced down to H_1 from positive saturation, a pronounced drop in the resistance is observed. In this case, all the nanowires are magnetized up, which results in a strong periodic pinning potential. In contrast, if the field is increased from negative saturation to the positive first matching field H_1 (dotted curve), only a small decrease of the resistance is observed around $H = 0.22T$. From the hysteresis loop shown in figure 4.15(b), it appears that about 15% of the nanowires are still magnetized down, giving rise to a strong perturbation to the periodic pinning potential and leading to a weakened matching effect, as shown in figure 4.15(a). The strongest difference between the resistive states of the two major branches is observed at the coercive field (indicated by a green bar) due to the largest difference between the corresponding magnetic states.

A similar effect may be responsible for the disappearance of the first matching effect for sample NbNNW25(75,100). Figure 4.16(a) shows a zoom on the low field part of the magnetoresistance curve for sample NbNNW25(75,100). In this case, the field is swept from positive saturation (2.5T) to zero field (remanent state - green curve) and then back to high positive fields (orange curve). The corresponding magnetization loop is represented in figure 4.16(b). Sweeping down from positive saturation, the sample is still in the saturated state at H_1 leading to a matching dip (green curve). In contrast, when sweeping up the field from the zero field remanent state, about 20% of the nanowires are still magnetized down at the first matching field, leading to a strongly disordered pinning potential. In this case, no matching dip is observed because the disordered potential avoids any local increase of the vortex

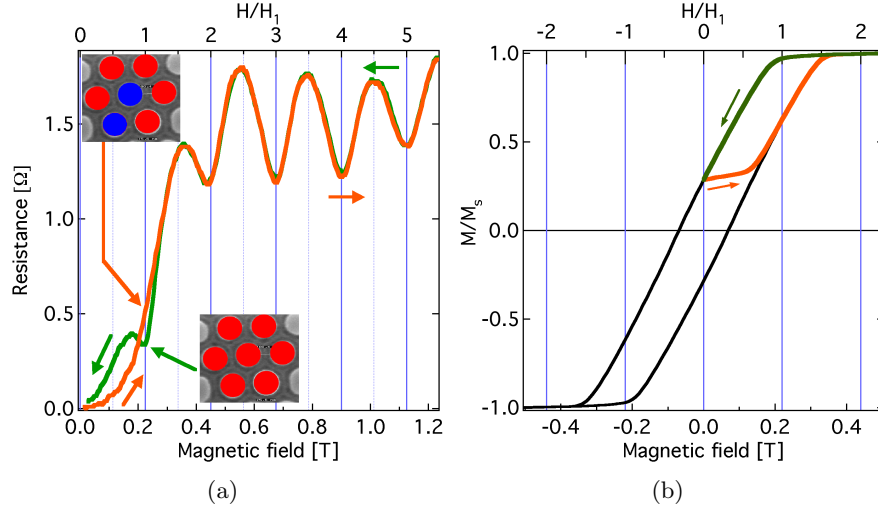


Figure 4.16: (a) Low field part of the magnetoresistance curves and (b) magnetization curves for sample NbNNW25(75,100). The magnetic state at H_1 is schematically represented for both field sweep cases, i.e. from zero field (in the remanent state) to positive fields and from high positive fields to zero field.

lattice stability.

A similar system was studied by Villegas et al. [148]. Indeed, the impact of the magnetic state of single-domain Co dots on the superconducting properties of an Al thin film was investigated. In the demagnetized state of the array, no matching effects were observed. However, the advantage of our system is the larger density of pinning centers and the larger vortex pinning strength (more matching effects are observed).

Rosen et al. [149] have studied the impact of positional disorder in a Co dots lattice on the matching features of a superconducting film in close proximity with Co dots. The matching features tends to disappear when increasing the disorder. However, the first matching feature was found to be quite robust with respect to the disorder. A shift to lower field of the first matching field was also observed for increasing disorder. Such shift of the first matching field seems to be present in figure 4.15(a). However, this will be discussed in detail in the next chapter.

4.5.3 Vortex depinning current

Arrays of pinning centers generally lead to an increase of the critical current or at least to an increase of the critical depinning current of the superconductor. The critical depinning current as a function of magnetic field for sample NbNNW20(60,100) and its reference sample

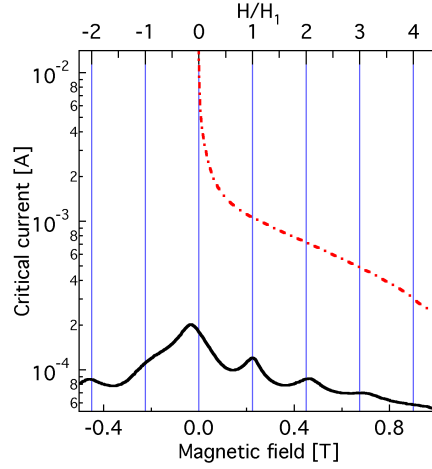


Figure 4.17: Critical depinning current curves for sample NbNNW20(60,100)#2 (black curve) and for its reference sample (red dashed curve) at $t = 0.76$. The criterion used was $10^{-4}R_n$ with R_n the normal state resistance). A logarithmic scale is used on the ordinate. A strong decrease of the critical depinning current is observed on the whole range of measurement. For sample NbNNW20(60,100)#2 the section for the current flow is 2.5 times lower than for the reference sample if one considers than the current flows between the pinning centers.

are shown in figure 4.17 at $t = 0.76$ (a logarithmic scale is used on the ordinate for clarity). This curve was obtained by adjusting the applied current in order to reach a resistance criterion of $R = 10^{-4}R_n$. Therefore, it is clearly the onset of vortex motion that is probed. Prior to each measurement (i.e. for each magnetic field value) a 'reset' of the sample was performed by applying a current higher than the instability current. This procedure drives the sample to a temperature higher than T_c for a short time. The sample has thus undergone some type of "field cooling procedure" prior to each measurement.

Critical depinning current peaks are clearly observed at the matching fields in perfect agreement with the previous results. These enhanced depinning currents result from a higher stability of the vortex lattice at these magnetic fields due to the absence of elastic deformation of the vortex lattice [150].

Nevertheless, the most striking result is the strong difference in critical current between sample NbNNW20(60,100) and its reference sample. Indeed, the critical current of the continuous reference film is higher over the entire magnetic field range by at least one order of magnitude. The most pronounced difference is at zero field, where sample NbNNW20(60,100) has a critical current 2 orders of magnitude lower than its reference sample. The global behavior of the critical current

curve is also very different. The critical current of the reference sample drops of about 1 orders of magnitude at low field (from 0 to 0.2T) and then decreases more slowly. The zero-field sharp peak is due to the absence of vortices⁶. At high field, the critical current reflects the onset of vortices motion which requires much lower currents and is dictated by the intrinsic pinning. On the contrary, the hybrid sample shows a quite low zero-field critical current. Then, the critical current decreases less rapidly than for its reference sample. However, in the magnetic field range of interest, i.e in the magnetic field range over which matching effects are observed, the critical currents are always much smaller.

The reduced critical current of the hybrid sample compared to its reference sample can originate from several factors :

- **The reduction of the effective cross-section for the current flow**

The presence of the Ni nanowire array reduces significantly the effective section in which the superconducting current can flow. Indeed, these nanowires induce a strong relief of the substrate (see figure 4.6(b), page 109) that reduces the electrical contact between the NbN deposited on top of the nanowires and in the surrounding regions. Moreover, the superconducting state of the NbN on top of the nanowires is weakened by the local magnetic stray field of the nanowire. Therefore, the current mainly flows in between the pinning centers.

This issue refers to the problem of the ideal pinning center size. Indeed, large pinning centers have large vortex pinning strength but they reduce the effective section for the current flow, while small pinning centers have large section for the current but lower vortex pinning strength. It was shown by Moshchalkov et al. [5] that the ideal pinning center size depends on the magnetic field range over which the superconducting properties have to be optimized. Increasing the pinning center size leads to better properties at high field but poorer at low field. Due to the large size of the pinning centers in our sample, improved properties compared to the reference sample are anticipated at high field rather than at low field.

- **The granularity**

The NbN films fabricated by sputtering are granular (see subsection 2.3.1). The grain boundaries constitute weak and narrow

6. The zero field critical current can be limited by either the depairing current [30] or the motion of thermal and current-induced vortex-antivortex pairs [30, 151] or by the critical current of the Josephson junctions present at the grains boundary [121] as is generally the case in granular superconductors.

superconducting regions in which high current densities flow. Therefore, these regions have the lowest critical current. The higher granularity of the NbN film for the hybrid sample compared to its reference sample leads to lower critical current due to weaker inter-grain electrical contacts (see figure 2.12). As a result, NbN films are usually modeled by Josephson Junction Arrays (JJA) [121, 152].

– **The stray fields of the Ni nanowires**

The reference sample exhibits a very strong dependence with external magnetic field, especially at low fields. Therefore, the presence of a Ni array with relatively high stray field reduces the zero-field critical current of the hybrid sample. This effect can partly explain the reduced critical current at low field (but not the high field one) for the hybrid sample.

4.6 Conclusion

We have investigated a new method to design ferromagnet - superconductor hybrids composed of a thin superconducting film of NbN deposited perpendicularly on a very dense triangular array of Ni nanowires (interdistance of 100nm and adjustable diameter in the range 35-80nm). This approach is based on the filling of a nanoporous alumina membrane with a ferromagnetic material using electrodeposition. Then, a thin superconducting film is deposited perpendicularly to the ferromagnetic nanowire array. Thanks to their small diameter, the electrodeposited Ni nanowires are single-domain. The nanowires exhibit a bistable magnetic state with the magnetization pointing either up or down along the nanowires axis. Therefore, the nanowires with magnetization parallel to the external magnetic field will act as pinning centers while the other constitute repulsive centers (barriers).

First, we have studied the impact of the surface corrugation of the alumina membrane filled with Ni nanowires on the superconducting properties of NbN thin films. For the smallest corrugation, the magnetic interaction was too weak to pin vortices and an abrupt increase of the resistance is observed when applying an external magnetic field. For the largest corrugation, the NbN is perforated by the Ni nanowire array. The normal state resistance is increased of almost one order of magnitude and the critical temperature is decreased with a global enlargement of the transition. In this case, numerous matching effects are observed reflecting the strong vortex pinning. At low field, an hysteretic behavior is observed which is a signature of the magnetic origin of the vortex pinning. For intermediate corrugation, intermediate situations between

these two cases are observed. Overall, the number and intensity of matching effects increase with the corrugation of the surface.

The case of intermediate corrugation, i.e. not sufficient to perforate the thin film, have been investigated in more details. Matching effects have been observed up to 2.5T for sample NbNNW25(75,100). For non-saturated magnetization states, different resistive states can be reached at a given magnetic field depending on the magnetic history. The largest difference is obtained at the coercive field of the array because the magnetic states associated to the two major branches of the magnetization curve differ mostly. The first matching effect is less pronounced when the ferromagnet deviates from the saturated state due to the presence of repelling centers which induce disorder in the pinning centers array.

The critical depinning current of hybrid samples is lower than the one of the reference sample. Several factors could be responsible for this reduction of the depinning current : first, the reduced cross-section over which the current is flowing due to the large size of the pinning centers. Second, the enhanced granularity of the superconducting film on the Ni nanowire array compared to the reference sample. Third, the stray fields of the nanowires which reduces the low-field critical current.

Chapter 5

Tunable matching effects and misleading field-induced superconductivity in NbN thin films

5.1 Introduction and motivation

In the previous chapter, we demonstrated the feasibility to produce dense arrays of magnetic pinning centers for superconducting thin films using a templating approach. The importance of the corrugation of the template surface on the vortex pinning has been presented. In particular, NbN films perforated by dense arrays of single domain ferromagnetic nanowires (see figure 5.1) display intense and numerous matching effects. In this chapter, we investigate in more details the properties of these NbN films perforated by arrays of Ni nanowires. In this particular case, both core pinning and magnetic pinning take place. As it was pointed out in the previous chapter, the first matching effect can be shifted from its expected value considering only geometrical aspects. Moreover, the resistance at the matching fields is lower than the one at zero field, which may be seen as a misleading field-induced superconductivity.

This chapter is organized as follows : in section 5.2, we discuss the possibility to tune the position of the first matching field. Section 5.3 is devoted to the origin of the superconductivity enhancement observed for the shifted matching fields. The origin of field-induced superconductivity

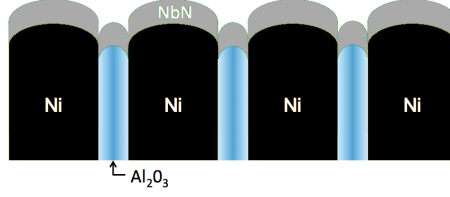


Figure 5.1: Schematic representation of a 20nm thick NbN film perforated by a Ni nanowire array with diameter $D = 70\text{nm}$.

is addressed in section 5.4. Finally, the conclusions are drawn in section 5.5.

5.2 Tunable first matching field

In this section, we investigate in detail the low field part of the magnetoresistance curves of NbN thin films perforated by ordered arrays of Ni nanowires. It was notably seen on curve (c) of figure 4.9 (page 112) that the first positive matching dip when coming from negative saturation field is shifted to lower fields. In this section, we first discuss the origin of this effect. Second, we show how the position of the first matching field can be tuned. Finally, we discuss the impact of the nanowire diameter and packing of the array on the shift of the first matching effect.

5.2.1 Origin of the shift of the first matching field

In the following discussion, we will use the notation H_{anb} to designate the various matching fields. 'a' = + (-) refers to initial positive (negative) saturation field of the measurement. 'n' refers to the index of the matching effect. 'b' = + (-) refers to the positive (negative) sign of the matching field. For example : H_{-1+} refers to the first matching effect at positive field when sweeping the field from negative saturation. Figure 5.2(a) shows the low field behavior of the magnetoresistance curves of sample NbNNW20(70,100) at $t=0.75$. This graph illustrates the notations of the matching effects. The solid curve corresponds to the field sweep from negative saturation to positive saturation. The dotted curve corresponds to the field sweep from positive saturation to zero field. Starting at a negative saturation (solid curve), the first matching dip is observed at $H_{-1-} = -0.23T$. This value corresponds to a nanowire interdistance of 102nm which is in very good agreement with the estimated 100nm from the anodization procedure. The first matching dip at

positive fields is observed at $H_{-1+} = 0.14T \approx 0.6 \cdot |H_{-1-}|$. This shift is due to a decrease of the number of pinning centers. Indeed, the so-called matching effect in a superconductor occurs when the number of vortices is equal to the number of pinning centers. A reduction of the number of pinning centers therefore leads to a reduction of the matching field. At H_{-1+} , some nanowires still not experience magnetization reversal and thus have their magnetization antiparallel to the external magnetic field. In this case, the number of pinning centers is reduced because the nanowires with magnetization anti-parallel to the magnetic field act as repulsive centers. As the number of pinning centers can be modified by the magnetic history, the matching effect also depends on the magnetic history of the nanowire array.

The position of the first matching field can be roughly determined from the normalized magnetization hysteresis curve of the array. If one considers a unit cell of the lattice, the matching condition, for which the number of vortices is equal to the number of pinning centers, takes the form :

$$f_p = f_v \quad (5.1)$$

where f_p is the "fraction" of pinning center per unit cell, i.e. the fraction of nanowires parallel to the external field and f_v is the fraction of vortex per unit cell (see red hexagon in figure 2.11(f)). For positive fields, $f_p = f_{up}$ (f_{up} is the fraction of nanowires parallel to a positive field) and for negative fields $f_p = f_{down}$ (f_{down} is the fraction of nanowires parallel to a negative field). f_{up} and f_{down} can be extracted from the hysteresis loop (M(H) curve of the nanowire array, see figure 5.2(b)) which was obtained from SQUID measurements at low temperature. Indeed, considering that the nanowire magnetization has only two possible orientations, one has :

$$\frac{M(H)}{M_s} = f_{up}(H) - f_{down}(H) \quad (5.2)$$

where M_s is the magnetization at saturation and f_{down} refers to repulsive pinning centers in positive fields. Since $f_{up}(H) + f_{down}(H) = 1$, Eq. 5.2 becomes :

$$f_{up}(H) = \frac{1}{2} \cdot \left[1 + \frac{M(H)}{M_s} \right] \quad (5.3)$$

On the other hand, the fraction of vortex per unit cell can be well approximated by :

$$f_v(H) = \frac{H}{H_1} \quad (5.4)$$

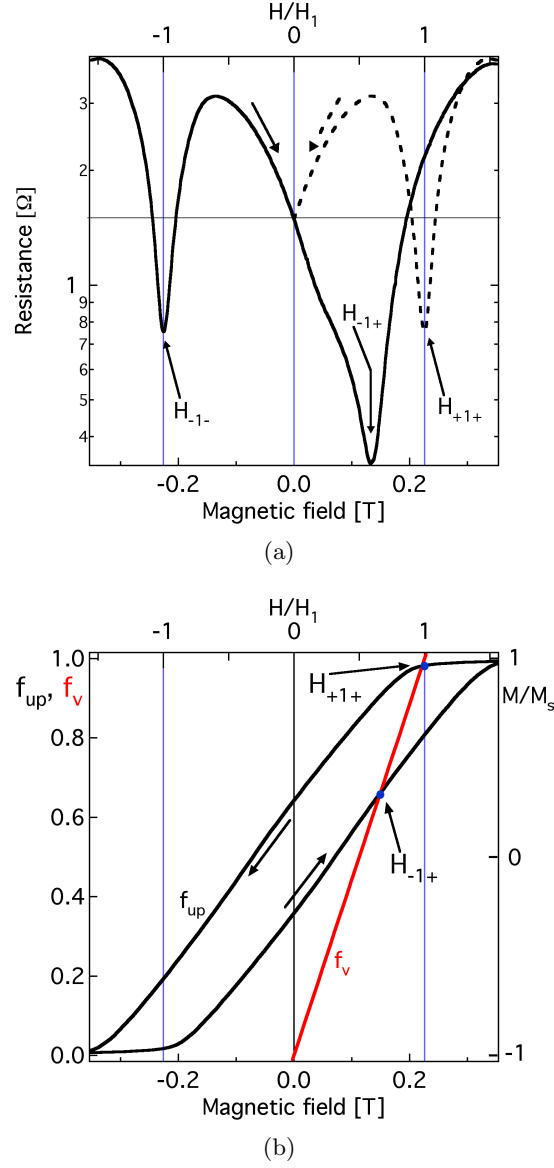


Figure 5.2: (a) Magnetoresistance curves for sample NbNNW20(70,100) at $t = 0.75$ and $I_{DC} = 10\mu A$. The solid curve refers to the field sweep from negative to positive saturation and the dashed curve to the sweep from positive saturation to zero field. (b) Fraction of nanowires with magnetization parallel to a positive field per hexagonal unit cell for sample NbNNW20(70,100) as a function of external magnetic field ($f_{up}(H)$) and fraction of vortices per hexagonal unit cell as a function of external magnetic field ($f_v(H)$). The $f_{up}(H)$ have been extracted from a magnetization curve (see text) performed at $T=15K$. The intersections of $f_{up}(H)$ and $f_v(H)$ correspond to the matching conditions for positive fields in qualitative agreement with the minima of the magnetoresistance curve.

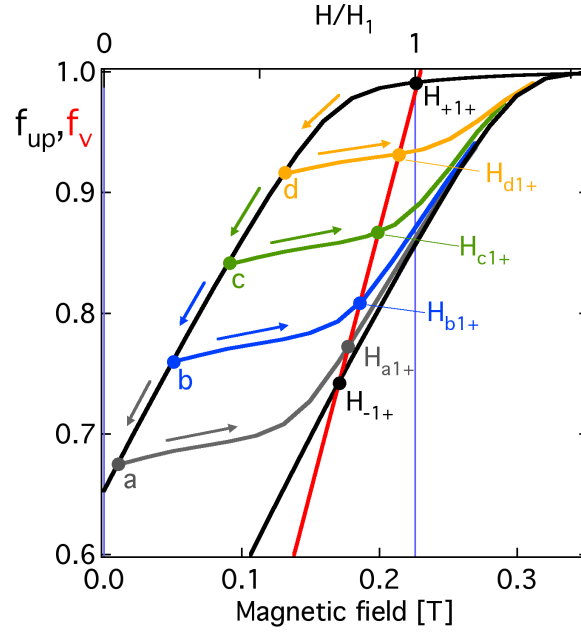
where $H_1 = 0.23T = H_{+1+} = -H_{-1-}$ for our particular sample. The evolutions of $f_{up}(H)$ and $f_v(H)$ are sketched in figure 5.2(b). The black curve corresponds to the $f_{up}(H)$ loop while the red straight line corresponds to $f_v(H)$. The intersection between these two curves gives the solutions of the equation 5.1, i.e. the matching condition, for positive fields. Contrary to the usual experiments with pinning center arrays, there are here two solutions for the first matching condition because the number of pinning centers can take two values at low magnetic field. The first one is realized at $f_{up} = 1$, i.e. when all the nanowires are parallel to the field. As the positive saturation is maintained down to about 0.1T, it is possible to observe the matching effect at its theoretical value, namely $H_{+1+} = 0.23T$ (by sweeping down the field from positive saturation). Sweeping the field from negative saturation to positive fields, the first positive matching condition is fulfilled at $H_{-1+} \approx 0.14T < H_{+1+}$ (see figure 5.2(b)). The minimum of resistance reported on figure 5.2(a) is in qualitative agreement with the intersection of the f_v and f_{up} curves shown on figure 5.2(b). This point will be discussed in the next subsection.

5.2.2 Intermediate shifts of H_1

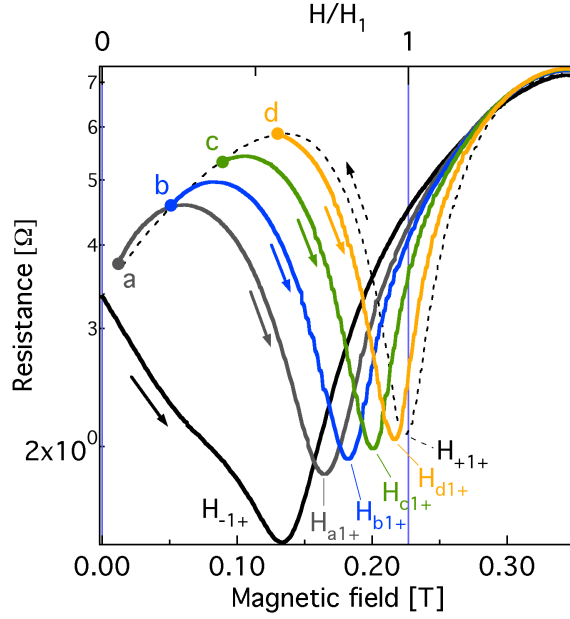
From figure 5.2, one sees that varying the magnetic configuration of the bistable nanowire array allows us to tune the position of the first matching dip. The matching fields can be varied in the range $[H_{-1+}, H_{+1+}]$ and $[H_{-1-}, H_{+1-}]$, delimited by the intersection between the linear function $f_v(H)$ and the two major branches of the function $f_{up}(H)$ (for positive fields) or $f_{down}(H)$ (for negative fields). As the magnetic state of the nanowire array (or equivalently the $f_{up}(H)$ curve) can be modified by performing minor cycles (see subsection 4.2.3, page 107), intermediate values of f_{up} between the two major branches can be reached. Figure 5.3(a) shows that the matching condition can be achieved at any intermediate magnetic field between H_{+1+} and H_{-1+} by performing such minor cycles. The notation H_{anb} has been extended to the minor cycles, using letters referring to the point at which the minor loops starts (e.g. H_{a1+} , see figure 5.3(a)). Accordingly, a tunable shift of the first matching peak in the magnetoresistance is observed (see figure 5.3(b)). Intermediate values of resistance at the matching conditions are also reached. These intermediate resistive states are directly related to the number of repelling centers obtained in these minor cycles. Therefore, modifying the magnetic history of the nanowire array allows us to tune at will the first matching field position (and also the resistivity) over a field range determined by the magnetic properties of the single-domain nanowire

array (i.e. mostly governed by the packing).

Figure 5.4 shows the evolution of the matching field $H_m = H_{i1+}$ as a function of the fraction of pinning centers $f_p = f_{up}$. The blue dots are experimental data extracted from a set of minor cycles similar to those shown in figure 5.3. The black line shows the theoretical evolution of the matching field corresponding to $H_{i1+}/H_{+1+} = f_{up}$. For high f_{up} (i.e. for low shift of the matching field with respect to H_{+1+}), the experimental data are in very good agreement with the expected behavior. For this particular sample, the matching effect is maintained down to a pinning center fraction of 0.66, i.e. for a relatively large disorder of the pinning array. This reflects the fact that the pinning force is very strong. However, the relative matching field deviates from the expected linear behavior for f_{up} lower than ~ 0.8 , i.e. for large disorder of the pinning potential. A similar observation was made by Rosen et al. [149] for pinning centers array with controlled positional disorder. Indeed, they observed that the matching field of triangular lattice of magnetic pinning center with controlled positional disorder of the pinning centers is shifted to lower field as the disorder increases. They ascribed this shift to the decrease of the local stable configurations of the vortex lattice in the disorder pinning center, which leads to enhanced vortex mobility (e.g. flux creep). In our case, the situation is somewhat different because the pinning centers positions is not modified by the reversal of the magnetic state of the nanowires, instead some of the pinning centers disappear and become repulsive. However, for a significant amount of repulsive centers in the lattice, some pinning centers become inactive due to the screening of the neighboring repulsive centers.



(a)



(b)

Figure 5.3: (a) Fraction of nanowires up (parallel to a positive field) for sample NbNNW20(70,100) as a function of magnetic field for different minor cycles ($f_{up}(H)$) and fraction of vortex by hexagonal unit cell of the nanowire lattice (f_v). (b) Magnetoresistance curves for sample NbNNW20(70,100) at $t = 0.75$ ($I_{DC} = 10\mu A$) for different minor cycles. A relatively good agreement is found between the position of the matching fields and the intersection of f_v and f_{up} .

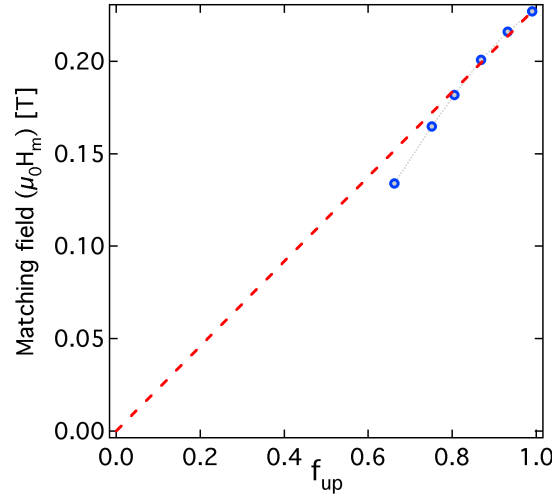


Figure 5.4: Matching field evolution ($H_m = H_{i1+}$) for sample NbNNW20(70,100) as a function of the pinning center fraction ($f_p = f_{up}$). The dots corresponds to the experimental data extracted from a set of minor cycles and the dotted line to the theoretical evolution of the matching field corresponding to $H_{i1+}/H_{+1+} = f_{up}$.

5.2.3 Influence of the nanowire packing on the shift

We have shown that the evolution of the matching field (H_{i1+}) is qualitatively determined by the intersection of the f_{up} and f_v curves. This intersection depends on the magnetic history, i.e. on the minor loop, but also on the shape of the magnetization curve which is mostly determined by the packing of the Ni nanowire array (see subsection 4.2.3, page 107). For our particular samples, the change in the packing of the nanowire array only originate from modification in wire diameter. The increase of the packing leads to a higher saturation field and a lower remanence. In order to study the influence of the packing of the nanowire array, we fabricated different samples with the same nanowire interdistance but with different nanowire diameter. Four different diameters have been studied : 40, 50, 60 and 70nm. The corresponding packing values are 14.5%, 22.7%, 32.6% and 44.4% respectively. Figure 5.5(a) shows the magnetization curve of samples with diameter 50nm and 70nm. As the saturation field increases with the diameter of the nanowires, the intersection between the lower branch of the hysteretic f_{up} curve and $f_v(H)$ (i.e. H_{-1+}) is lower for larger diameters. Figure 5.5(b) shows the evolution of the matching minimum as a function of the initial magnetic field of the minor loop for the different diameters. The ratio H_{i1+}/H_{+1+} increases with decreasing diameter : it falls down to 0.58 for the 70nm diameters, to 0.72 for the 60nm diameter and to 0.96 for

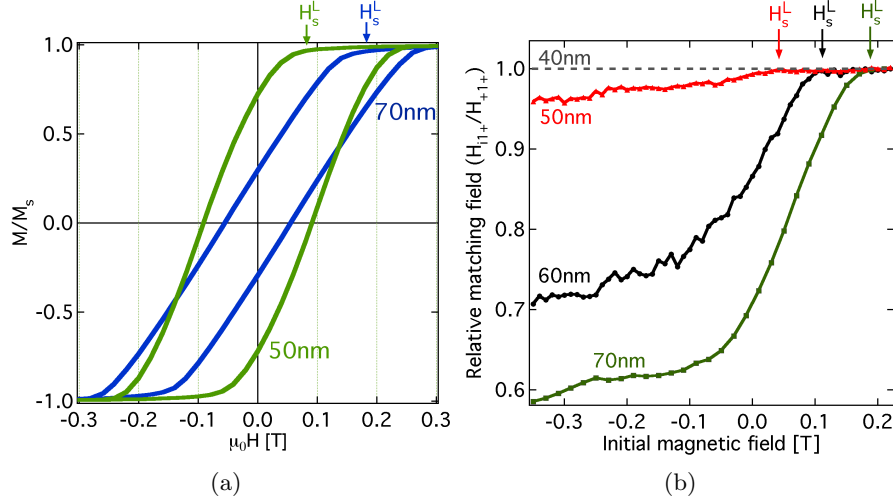


Figure 5.5: (a) Normalized magnetization hysteresis curves for sample NbNNW20(70,100) (blue curve) and for sample NbNNW20(50,100) (green curve). (b) Evolution of the matching field as a function of the initial magnetic field of the minor cycle for samples with different Ni nanowire diameter (i.e. different packing). Dashed line = NbNNW20(40,100), red curve = NbNNW20(50,100), black curve = NbNNW20(60,100) and green curve = NbNNW20(70,100). For the samples considered here, the magnetic field range, over which the matching effects can be observed, increases with the diameter of the nanowires, i.e. the packing of the array. This is due to the increase of the saturation field. H_s^L refers to the lower saturation field as indicated on figure a.

the 50nm. The 40nm diameter sample doesn't show any shift because the saturation field is lower than the first matching field. Therefore, the evolution of the matching field as a function of the starting magnetic field is represented as the horizontal dashed line at $H_{i1+}/H_{+1+} = 1$ (see figure 5.5(b)).

The sample NbNNW20(50,100) is of particular interest. Indeed, as it is shown in figure 5.6(a), the matching condition is only strictly realized very close to the saturation field. This leads to a very small shift of the first matching peak, i.e. $H_{-1+} \approx 0.95H_{+1+}$ as shown in figure 5.5(b). The figure 5.6(b) shows the magnetoresistance curves for NbN50 considering different minor cycles. It should be emphasized that an additional dip is present at low field (H_{add}). It is located close to zero for the major branch, then shifts to higher field and progressively vanishes from minor cycles (1) to (5) (see figure 5.6(b)), i.e. as smaller minor cycles are performed. Strictly speaking, these matching effects do not correspond to a theoretical matching condition (see figure 5.6(a)). Indeed, at these

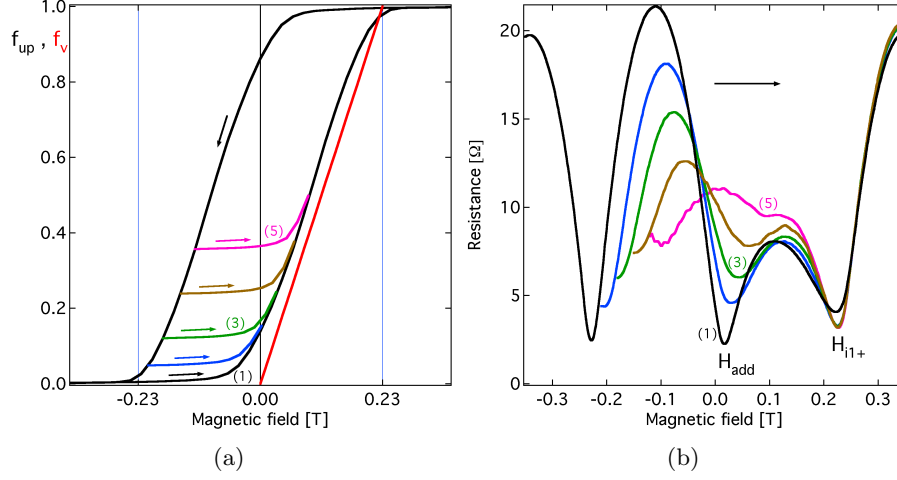


Figure 5.6: (a) Fraction of nanowires with magnetization parallel to a positive field $f_{up}(H)$ (black curve) for sample NbNNW20(50,100) and fraction of vortex $f_v(H)$ (straight line). Both fraction are expressed by hexagonal unit cell. (b) Magnetoresistance curves for sample NbNNW20(50,100) at $t = 0.77$ and $I_{DC} = 10 \mu A$. Minor cycles are represented in both figure and labeled from (1) to (5). The field sweep is indicated by arrows. H_{add} refers to the additional matching effect observed between $H=0$ and H_{i1+} .

fields, the number of pinning centers somewhat exceeds the number of vortices. Nevertheless, we believe that these minima can be ascribed to matching effects, due to the close proximity between the two curves especially considering the low concentration of pinning centers. Therefore, both minima (located at H_{i1+} and H_{add}) can be considered as first matching effects and therefore there are two successive first matching effects for two different pinning center density. Both first matching effects are tunable, although the one at $\sim 0.23T$ (H_{i1+}), can be modified only over a very narrow field range. These intermediate minima clearly reflect the strong and complicated magnetic interaction between the superconducting film and the Ni nanowire array in the non-saturated region. Vortex imaging experiments as well as theoretical works would shed some light on this interesting effect.

5.3 Superconductivity enhancement at H_{-1+}

Figure 5.3 clearly shows that the lowest resistive state at a matching field is obtained at $H_{-1+} = 0.14T$ instead of $H_{+1+} = 0.227T$. This enhancement is the most pronounced for low resistive state (i.e. just above the resolution of our setup) while it progressively disappear at higher

resistive states (together with the disappearance of the matching dips). Therefore, it is related to the vortex depinning and motion. Two factors influencing the vortex motion and the vortex pinning are competing at H_{-1+} . First, the vortex lattice is not perfectly ordered due to the presence of repelling centers. Therefore, elastic deformation of the vortex lattice is present contrary to the case at H_{+1+} where the vortex lattice is not deformed. This term reduces the depinning current compared to a perfectly ordered array of pinning centers. Second, the repelling centers act as barriers for the vortex motion. Indeed, the presence of such barriers around the pinning centers increases the critical depinning force and hinders the vortex motion leading to a reduced vortex channeling.

One of these competing factors will get the best of the other depending on the relative intensity of the pinning force and the vortex-vortex interaction. In the case of perforated films, the pinning force clearly overcomes the vortex-vortex interaction force. Actually, such lower resistive state at H_{-1+} was only observed for superconducting films perforated by the array of nanowires. As shown in figure 4.15 (page 121), for lower pinning strength, the resistance at H_{-1+} is higher than at H_{+1+} and the matching effect is much less pronounced.

MFm measurements presented in figures 4.4(a) and 4.4(b) (page 107) show that despite the presence of repelling centers, the pinning center lattice remains ordered on a short length scale. The pinning centers form clusters separated by clusters of repelling nanowires. Therefore, the small clusters of pinned vortices are caged by the surrounding repelling centers which increase their stability.

This caging effect has been theoretically investigated by Chen et al. [153]. Using molecular dynamics they calculated the critical current of a thin superconducting film in close proximity with ferromagnetic dots. As for our system, these magnetic dots were single-domain with bistable magnetization states perpendicular to the superconducting film. Three different magnetic configurations were investigated : ferromagnetic arrangement, anti-ferromagnetic arrangement and 4×4 anti-ferromagnetic arrangement. The caging effect for the antiferromagnetic arrangement clearly leads to an increase of the depinning current.

Moreover, it has been also demonstrated numerically [55, 154, 155] and experimentally [7, 156, 157] that partly disordered lattices [55] or quasiperiodic [154, 155] pinning arrays exhibit enhanced superconducting properties compared to perfectly periodic arrays and randomly distributed pinning centers. This is generally explained by the lower vortex channeling.

5.3.1 Impact of the magnetic state on the resistance

Figure 5.7(a) shows the magnetoresistance curves for a full set of minor loops for sample NbNNW20(70,100) at $t = 0.75$ and $I_{dc} = 10\mu A$. This graph refers to the range of resistive states that can be reached for a given magnetic field by changing the magnetic state of the ferromagnetic nanowire array. For example at zero field, the resistance of the sample can be varied between 3.35 and 4.11Ω , thus representing a variation of 23%. However, for certain magnetic field, the range of allowed resistive states is much larger. At $H_{-1+} \approx 0.134T$ (or H_{+1-}), the resistance can be varied from 1.44Ω to 5.8Ω (variation of $\sim 300\%$). The evolution of the resistance as a function of the initial field of the minor loop (called 'initial magnetic field' which corresponds for example to points a, b, c and d in figure 5.3) is represented in figure 5.7(b) for $H = 0$, $H = H_{-1+} = 0.134T$ and $H = H_{+1+} = 0.226T$. Three different behaviors are observed. At zero field, the resistance is relatively constant for initial magnetic fields less than $-0.2T$, then at about $-0.2T$ the resistance starts to increase up to a maximum value reached for $-0.07T$ and then decreases till zero field. At $H_{-1+} = 0.134T$, the resistance slowly increases with increasing initial field till about $-0.06T$ then it starts to increase much stronger. At $H_{+1+} = 0.226T$, the resistance is at its maximum for low initial field and almost constant until zero field. Then a relatively sharp transition to a lower resistive state is observed. This lower resistive state is reached when the initial magnetic field equals the lowest saturation field, denoted by H_s^l in figure 5.7(b) and corresponding to the lowest field at which the saturated state of the nanowire array is maintained.

These different behaviors are due to the evolution of the fraction of pinning centers per unit cell when performing such different minor loops. Using the magnetization curves corresponding to each minor cycle, one can extract the fraction of pinning centers per unit cell at a given magnetic field. Therefore, the resistance as a function of the fraction of pinning center per unit cell (f_{up}) can be determined for different magnetic fields of the minor loops. This is represented in figure 5.7(c). Very different behaviors are observed when varying the fraction of pinning center for the three different magnetic fields. At zero field, the resistance is maximum for $f_{up} = 1/2 = f_{down}$. Of course, in this case, it makes no sense to talk about pinning centers since, in principle, no vortices are present in the superconducting film. This result is in contradiction with the one obtained by Villegas et al. [111] in Al thin films deposited on Fe nanodots array. In their case, a huge drop of resistance was observed at zero field when the out-of-plane magnetization of the dot was randomly distributed up and down. This was due to the reduced stray field

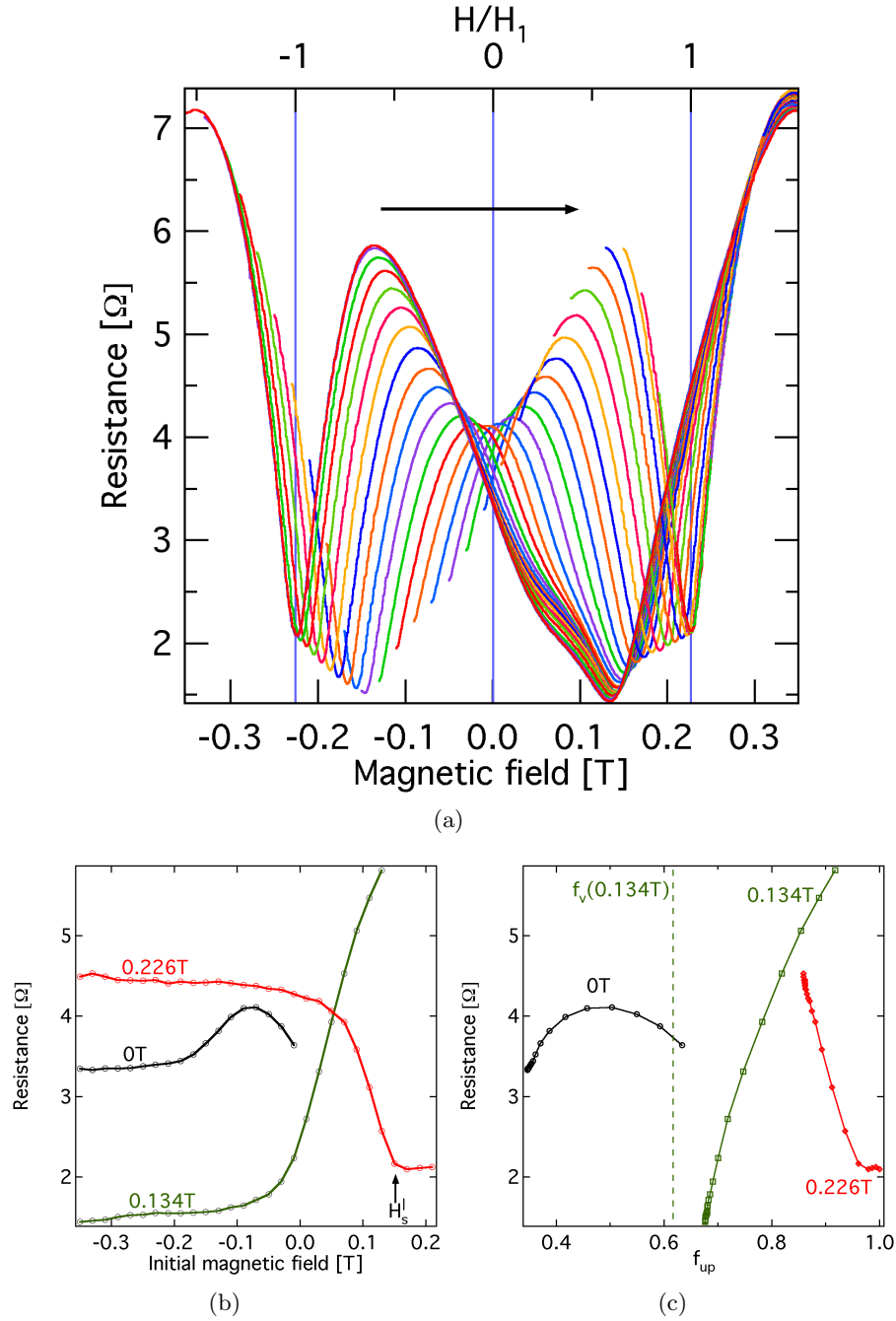


Figure 5.7: (a) Magnetoresistance curves for sample NbNNW20(70,100) at $t = 0.75$ and $I_{dc} = 10\mu A$ for all the minor loops. (b) Evolution of resistance as a function of the initial magnetic fields of the minor loops for $H = 0$, $H = H_{-1+} = 0.134T$ and $H = H_{+1+} = 0.226T$. Data have been extracted from figure 5.7(a). (c) Resistance as a function of the fraction of the pinning center f_{up} for $H = 0$, $H = H_{-1+} = 0.134T$ and $H = H_{+1+} = 0.226T$. The f_{up} values have been extracted from the corresponding minor loops of the magnetization curves.

present in the Al film in-between the dots which allowed the percolation of superconductivity. In all our samples, the opposite effect is observed but with a much lower amplitude. We argue that this difference is due to the difference of shape and size of our ferromagnets and thus to the different stray field distribution in the NbN film compared to the case of Villegas et al. Indeed, due to the large aspect ratio of our nanowires, the stray field have a much larger extend than the interwire distance.

At 0.226T, the resistance increases with decreasing f_{up} , i.e. with increasing disorder in the pinning center lattice. This can be explained by the fact that the matching effect is reached for $f_{up} = 1 = f_v(0.226T)$. Therefore, the decrease of pinning center with a fixed number of vortex leads to an increase of interstitial vortices or doubly-quantized vortices which are more mobile. At 0.134T, the opposite tendency is observed, i.e. the resistance increases with increasing f_{up} . This is due to the fact that for the lowest f_{up} , the matching condition is almost fulfilled and the number of repulsive centers is high thus leading to a strong caging potential for the vortices. Therefore, the resistance is quite low (in fact, it corresponds to the lowest resistance at this temperature). The increase of f_{up} leads to a reduction of the caging which leads to a decrease of the pinning force and thus to an increase of the resistance.

5.4 Misleading field-induced superconductivity

Field-induced superconductivity is the general term used in the literature [9–11, 72, 110, 129, 148, 158] to designate an enhancement of the superconducting state (lower resistive state) for a finite magnetic field compared to the zero field superconducting state. As the Cooper pairing is weakened by the magnetic field, field-induced superconductivity is basically generated by the compensation of the external field by a localized magnetic field. This local magnetic field mostly originated from arrays of ferromagnetic nanostructures close to the superconducting thin film [9, 11, 72, 129] although similar effects have been obtained with superconducting loops [110]. Either the creation of vortex-antivortex pairs by the stray field of the ferromagnets [9, 72, 129] or the nucleation of superconductivity in the domain or at the domain-walls of the ferromagnets have been studied [10]. The results reported in several previous figures (notably figures 5.2(a) and 5.7(a)) have already shown that in our samples, the resistance at zero field can be larger than for some applied magnetic field. In this section, we discuss the origin of this misleading field-induced superconductivity.

5.4.1 Phase diagram

The phase diagrams for sample NbNNW20(60,100)#1 and for its reference sample are shown in figure 5.8. The black solid curve corresponds to the critical depinning temperature, i.e. the temperature at which the vortices start to move. This curve was obtained by adjusting the temperature in order to maintain the resistance at $R = 10^{-4}R_n$ (R_n the normal state resistance)¹. The resistance was measured by applying a direct current of $20\mu A$, which is much lower than the instability current at these temperatures (typically several mA). The blue region (R) corresponds to the resistive state (vortex motion) and the orange region (S) corresponds to the perfect superconducting state (motionless vortices). The green region (FIS) refers to field-induced superconductivity. For this particular sample, the field-induced superconductivity is observed up to 1.2T, i.e. up to the fifth matching field. The field-induced superconductivity mainly corresponds to several magnetic field intervals (green region - FIS) centered around the matching fields, as shown in figure 5.8.

The critical temperature curve for the reference sample is presented on the same figure (dashed red curve). In order to compare the shape of the curves, the critical temperatures have been normalized to their zero field values (7.16K for sample NbNNW20(60,100) and 9.94K for its reference). As shown in figure 5.8, the curves are noticeably different for the two samples, i.e. the superconductor-ferromagnet hybrid sample is clearly less affected by the external magnetic field.

1. A higher resistive criterion has a negative impact on both the matching effects and on the misleading field-induced superconductivity. For example, the use of a mid-transition criterion to define the transition from the superconducting state to the normal state leads to the disappearance of the matching effects and thus also of the field-induced superconductivity.

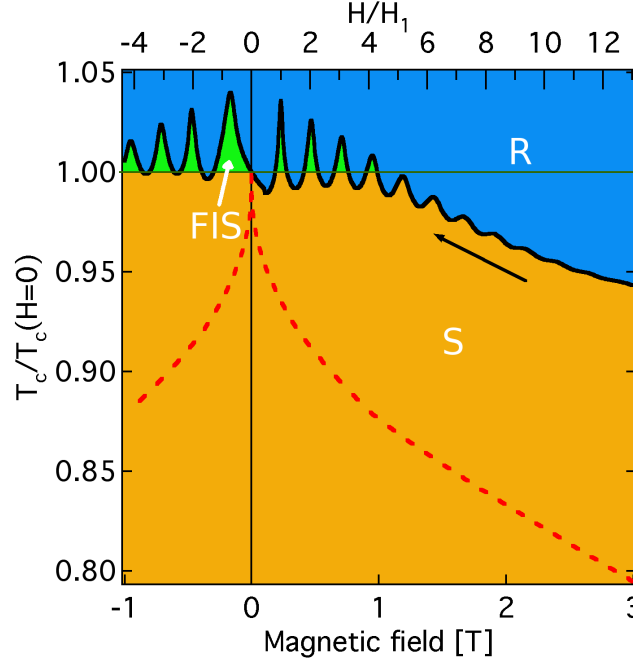


Figure 5.8: Phase diagram of superconductivity for sample NbNNW20(60,100) (black solid line) and for its reference sample (red dashed line). The criterion is the appearance of resistance ($R = 10^{-4} \times R_n$). The temperatures are normalized with respect to the zero field value (7.16K and 9.88K for sample NbNNW20(60,100)#1 and its reference respectively). The blue region (R) corresponds to the resistive state ($R > 10^{-4} \times R_n$), the green region (FIS) corresponds to field-induced superconductivity ($R < 10^{-4} \times R_n$), the orange (S) region corresponds to the perfect superconducting state of sample NbNNW20(60,100)#1 ($R < 10^{-4} \times R_n$). The field was swept from positive to negative fields (black arrow).

5.4.2 Origin of field-induced superconductivity

Generally, field-induced superconductivity is ascribed to the creation of vortex-antivortex pairs (V-AV) by the ferromagnets. In our case, the stray field of Ni nanowire array is too weak to create V-AV pairs. Indeed, The magnetic flux above each nanowire is $\phi_{NW} \sim 0.45T \times S_{NW} = 0.61\phi_0$ (estimated from the magnetostatic calculations see figure 4.8(b), page 111), where S_{NW} is the surface of the nanowire tip. Moreover, the appearance of V-AV shifts globally the phase diagram which is not the case here².

In reality, the misleading FIS observed in our samples is an artefact due to the evolution of the magnetic state of the nanowire array during the measurement. In fact, by decreasing the field from the saturated

2. More details about the phenomena leading to FIS are discussed in annexe A.

magnetic state to zero field, not only the number of vortices is changed but also the magnetic state of the array is modified which impacts on the zero field superconducting state. To convince the reader, we consider the sample with thinnest nanowires (i.e. sample NbNNW20(40,100)). Thanks to its high remanence (about 90%), it is possible to vary the zero field magnetic state to a large extent by performing minor cycles. Also, the saturation field is just below H_1 which means that $H_{-1+} = H_{+1+}$.

Several magnetoresistance curves corresponding to different minor cycles are represented in figure 5.9(a). The zero-field resistance is strongly dependent on the minor cycles, i.e. on the magnetic state of the array. This is not the case at H_1 because the sample is always in the saturated state. One can see that for some minor cycles, the resistance at zero field is higher than the one at H_1 which results in an misleading field-induced superconductivity. In this case, the magnetic flux above the dots is $\phi_{dot} \sim 0.27\phi_0$, i.e. much lower than the value of $1.29\phi_0$ calculated by Milosevic et al. [135] for the creation of a V-AV pair.

Figure 5.9(b) shows the evolution of the zero-field resistance as a function of the initial magnetic field of the minor loop for samples NbNNW20(40,100) and NbNNW20(50,100). The region corresponding to "field-induced superconductivity" is highlighted in green. Figure 5.9(c) shows the evolution of the resistance as a function of f_{up} at zero field for sample NbNNW20(50,100). For $f_{up} > 0.82$, the resistance is lower than the one at H_{+1+} which is indicated by the dashed line. When decreasing f_{up} , the resistance increases significantly and becomes higher than the one at H_{+1+} . For the majority of the f_{up} values, the resistance at $H = 0$ is larger than at $H = H_1$. Therefore, if the remanence of this array was lower, it would also lead to field-induced superconductivity.

Figure 5.9(d) shows the zero field resistance for the same sample as a function of the $f_{up} \cdot f_{down} = f_{up} \cdot (1 - f_{up})$ which reflects the disorder of the magnetic state of the nanowire array ($f_{up} \cdot f_{down}$ takes the maximum value of 0.25). Within the experimental error, the resistance is maximum when the disorder in the array is maximum.

The same dependence of the zero field resistance with f_{up} was observed for the other samples (NbNNW20(60,100) and NbNNW20(70,100)) but over a smaller range of f_{up} since the remanence is lower. Indeed, this evolution was shown in figure 5.7(c) for sample NbNNW20(70,100). The zero field resistance is directly related to the disorder of the array as is indicated by the parabolic behavior with a maximum at $f_{up} = 0.5$. Therefore, we assume that if the magnetic state of this array could be maintained in the $f_{up} = 1$ state at zero field, field-induced superconductivity would not be observed, in agreement with the absence of V-AV pairs in our system.

In conclusion, the field-induced superconductivity observed in our samples is due to the fact that two parameters are varying simultaneously : the external magnetic field and the magnetic state of the single-domain Ni nanowire array. At zero field, a higher resistive state is observed due to the modification of the magnetic state of the array compared to the one reached at high field (i.e. the saturated state). If the saturated state of the array was kept down to zero field, we argue that no field-induced superconductivity would be observed.

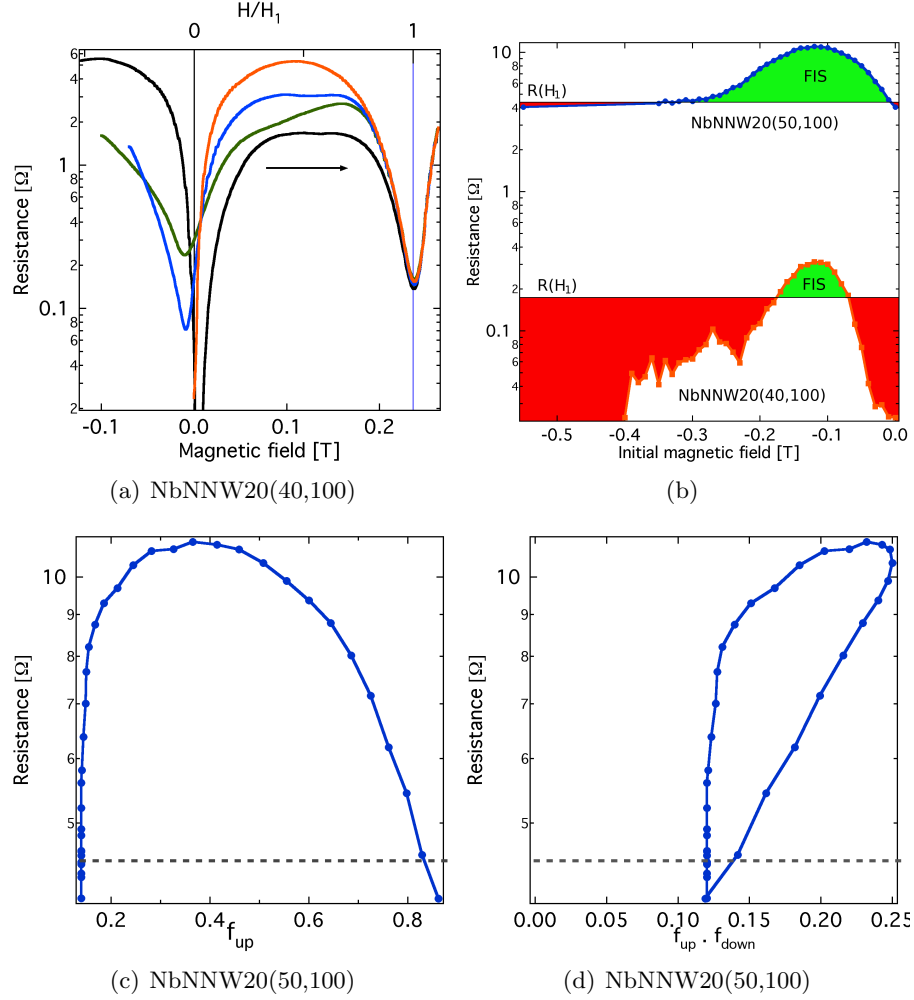


Figure 5.9: (a) Magnetoresistance curves for sample NbNNW20(40,100) for several minor loops ($I_{DC} = 10\mu A$ and $t = 0.92$). (b) Resistance at zero field as a function of the initial magnetic field of the minor loops for samples NbNNW20(50,100) and NbNNW20(40,100). Green regions correspond to misleading field-induced superconductivity. (c) Resistance at zero field as a function of f_{up} for sample NbNNW20(50,100). (d) Resistance at zero field as a function of $f_{up} \cdot f_{down}$ (parameter reflecting the disorder of the magnetic state of the nanowire array) for sample NbNNW20(50,100). The dashed line corresponds to the resistance at H_{+1+} .

5.5 Conclusion

In this chapter, we investigated the superconducting properties of NbN thin films perforated by Ni nanowire arrays. For such strong magnetic pinning, the first matching field is shifted from its theoretical value from geometrical considerations. This shift is due to field-dependent number of pinning centers. Therefore, the first matching peak can be tuned over a certain field range, by performing minor loops of the magnetization curve. Indeed, minor loops allow to modify the number of pinning centers for a given magnetic field and therefore modify the matching condition. The first matching peak position depends on the packing of the array which can be modified by varying the diameter of the nanowires. For the smallest diameters ($D = 40nm$), no shift in the first matching field is observed since the saturation field is lower than H_1 . For the largest diameters ($D=70nm$), the shift of the first matching field is in agreement with the condition $f_{up} = f_v$ excepted for the lowest values of f_{up} for which the shift is more pronounced. This effect may originate from the enhanced disorder of the pinning center array.

In addition, the superconducting state is enhanced (lower resistance) for matching effects with $f_{up} < 1$, i.e. when the first matching field is shifted to lower field than its theoretical value from geometrical considerations. This is due to the caging effect of the surrounding clusters of repelling centers.

Misleading field-induced superconductivity is observed for the perforated samples up to 1.2T. However, this effect was ascribed to an artifact originating in the evolution of the magnetic state of the single-domain nanowire array at low field.

General conclusion

In this work, we developed new approaches based on porous alumina membranes and templates to create very dense arrays of pinning centers in superconducting thin films. We focused on the electrical properties and particularly on the vortex pinning properties.

The first approach is based on the imprinting of the nanostructured alumina surfaces into superconducting thin films in order to create periodic arrays of pinning centers.

The first system consists in a NbN thin film containing a triangular array of antidots with interdistance of 50nm and pore diameter of 30nm. The sample was fabricated by the direct reactive sputtering deposition of NbN thin film on the porous face of an alumina template. At high temperatures, the one-dimensional wire network regime was identified due to the narrow inter-hole spacing (about 20nm). However, only four frustration effects were observed due to the relatively short-range order of the lattice. Nevertheless, this is much higher than what have been previously reported by other groups. At lower temperature, a cross-over to the vortex regime was observed due to the decrease of the coherence length. In this case, only the first matching effect was visible.

The second system consists in NbN thin films deposited on the barrier layer face of AAO templates. We have shown that the barrier layer side of the template, which is generally considered as undesirable (and more frequently eliminated to make porous alumina membranes) can be used to create hexagonal and double-triangular lattices of pinning centers. The major advantages of the barrier layer side compared to the hole side are the higher pinning center density and the hexagonal symmetry of the pinning center lattice. Indeed, the number of pinning center on the barrier layer side is twice that on the porous face. Moreover, the hexagonal array of pinning center allows us to cage more efficiently the interstitial vortices that appear above the first matching field compared to a triangular lattice.

In this case, the vortex regime was observed in the whole range of

temperature. At high temperatures, a sharp matching peak is observed at H_1 and three smaller peaks are present at $H_{1/4}$, $H_{1/2}$ and $H_{3/4}$ thus, indicating a strong pinning force. Matching effects are also observed at $H_{1.5}$ and H_2 in agreement with the theoretical predictions. Surprisingly, the matching effect at H_2 becomes more pronounced at low temperatures and a matching effect at H_3 also appears.

In addition, using two different approaches, we also investigated how the matching features evolve while varying the pinning strength. First, a Si insulating layer (25nm thick) was deposited prior to the NbN deposition in order to flatten the surface (thus reducing the pinning strength). In this case, the fractional matching effect at $H_{1/2}$ is favored. This is due to the enhanced vortex-vortex interaction compared to the vortex-pinning center interaction which favors the triangular arrangement. Second, different thicknesses of NbN, ranging from 10 to 30nm, have been deposited on the barrier layer. Similarly, an enhancement of the $H_{1/2}$ peak is observed for increasing thicknesses.

The third system consist in Nb thin films deposited by MBE on the barrier layer of alumina templates. Six different samples were studied by varying the thickness (30 and 50nm) and the angle of deposition (0° , 30° and 60°) from the normal to the surface. For the 0° deposits, no clear matching effects have been observed. This strongly contrasts with the strong matching effects observed for the sputtered NbN films. On the contrary, the deposits at 30° show strong matching features for both thicknesses of the Nb film. The pinning center lattice results from thickness modulations (blind holes) located close to the interstitial sites due to shadowing effects. Due to the dependence of the shadowing effect on the direction of deposition, the pinning center lattice is not hexagonal but double triangular. The global behavior of such array is similar to the one of an hexagonal array with low pinning strength. At low temperature and at magnetic fields higher than H_1 , different flux flow regimes (featured by linear magnetoresistance) are observed between the successive matching fields, due to the appearance of interstitial vortices.

The second approach consisted in the use of dense arrays of Ni nanowires grown in the alumina membranes to tailor the superconducting properties on NbN thin films.

The triangular arrays of Ni nanowires with interdistance of 100nm and ajustable diameter in the range 35-80nm were grown by electro-deposition in alumina membranes. Then, a thin superconducting film is deposited on the surface of the membrane in proximity with the nanowire array. Due to their small diameters, the Ni nanowires are single-domain.

Therefore, each nanowire exhibits a bistable magnetic state with the magnetization pointing either up or down along the nanowires axis. The nanowires with magnetization parallel to the external magnetic field act as pinning centers while the other constitute repulsive centers (barriers).

We have studied the impact of the surface corrugation of the alumina membrane filled with Ni nanowires on the superconducting properties. For the smallest corrugation, the magnetic interaction was too weak to pin vortices and an abrupt increase of the resistance is observed when an external magnetic field is applied. For the largest corrugation, the NbN thin film is perforated by the Ni nanowires array. The normal state resistance is increased of almost one order of magnitude and the critical temperature is decreased with a global enlargement of the transition. In this case, numerous matching effects are observed reflecting the strong vortex pinning. At low field, an hysteretic behavior is observed, which is a signature of the magnetic origin of the vortex pinning. For intermediate corrugations, intermediate situations between the two extreme cases are observed. Overall, both the number and intensity of matching effects increase with the corrugation of the surface.

In the case of intermediate corrugation, i.e. not sufficient to perforate the superconducting thin film, matching effects have been observed up to 2.5T for the sample with the largest nanowire diameter, i.e. $D=75\text{nm}$. At low field, different resistive states can be reached at a given magnetic field depending on the magnetic history. The largest difference is obtained at the coercive field of the array because the magnetic states associated to the two major branches of the magnetization curve differ mostly. The first matching effect is less pronounced when the ferromagnet deviates from the saturated state due to the presence of repelling centers which induce disorder in the pinning centers array. However, due to a reduced effective cross-section for the current flow and an enhanced granularity of the NbN films, the critical depinning current for hybrid samples are lower than the one for the reference samples.

NbN thin films perforated by Ni nanowires arrays have been investigated more deeply. Depending on the magnetic history, the first matching field can be shifted from its theoretical value using geometrical considerations. This shift is due to field-dependent number of pinning centers. Therefore, the first matching peak can be tuned over a certain field range, by performing minor loops of the magnetization curve. Indeed, minor loops allow to modify the number of pinning centers for a given magnetic field and therefore modify the matching condition. The first matching peak position depends on the packing of the array which can be modified by varying the diameter of the nanowires. For the smallest diameters ($D=40\text{nm}$), no shift in the first matching field is observed

since the saturation field is lower than H_1 . For the largest diameters ($D=70\text{nm}$), the shift of the first matching field is in agreement with the condition $f_{up} = f_v$ (i.e. the fraction of nanowires parallel to the field is equal to the fraction of vortex per unit cell) excepted for the lowest values of f_{up} for which the shift is more pronounced. This additional shift should originate from the enhanced disorder of the pinning center array as reported in the literature. In addition, the superconducting state is enhanced (lower resistance) for matching effects with $f_{up} < 1$, i.e. when the first matching field is shifted to lower field than its theoretical value from geometrical considerations. This is due to the caging effect of the surrounding clusters of repelling centers.

Misleading field-induced superconductivity is observed for the perforated samples up to 1.2T. This effect was ascribed to an artifact effect originating in the evolution of the magnetic state of the array at low field. This interpretation is corroborated by the possibility to tune the zero field resistance on a relatively large range by modifying the magnetic state of the array.

In conclusion, we have demonstrated in this work that alumina templates and membranes can be used to create different types of periodic arrays of pinning centers with very high densities (holes, blind holes and magnetic pinning centers) in thin superconducting films.

Appendix A

Field-induced superconductivity models

In section 5.4, we showed that the apparent field-induced superconductivity observed in our samples is due to the different magnetic state at zero magnetic field compared to the first matching field. However, in the literature, field-induced superconductivity have been ascribed to two other effects : field-compensation [9–11, 110, 129, 131, 148] (V-AV pairs creation, domain-wall or domain superconductivity,...) and disorder in superconducting 1D wire network [159, 160]. In this appendix, these alternatives models are discussed.

A.1 Field-compensation effects

Figure A.1(a) (taken from Lange et al. [9]) represent the field - compensation effect as it was previously used in several works [9, 72, 110, 129, 161, 162] to obtain field-induced superconductivity. The magnetic stray field of the dots in the inter-dot region is compensated by the external magnetic field. Therefore, the critical temperature of the inter-dot region is higher for a finite external field than for zero external field.

The origin of this phenomenon is associated to the presence of vortex-antivortex (V-AV) pairs in the superconducting thin film [9, 129]. These V-AV pairs are created by the stray fields of the ferromagnetic dots. An anti-vortex is always associated with a vortex due to the magnetic flux conservation. This leads to a global shift of the phase diagram to a magnetic field $H = nH_1$ [72, 129, 131, 135], with n being the number of V-AV pairs generated by each dot. Indeed, at $H_{ext} = nH_1$, the vortices generated by the external field cancels two by two with the antivortices created by the dots. This leads to a minimal number of vortices in the

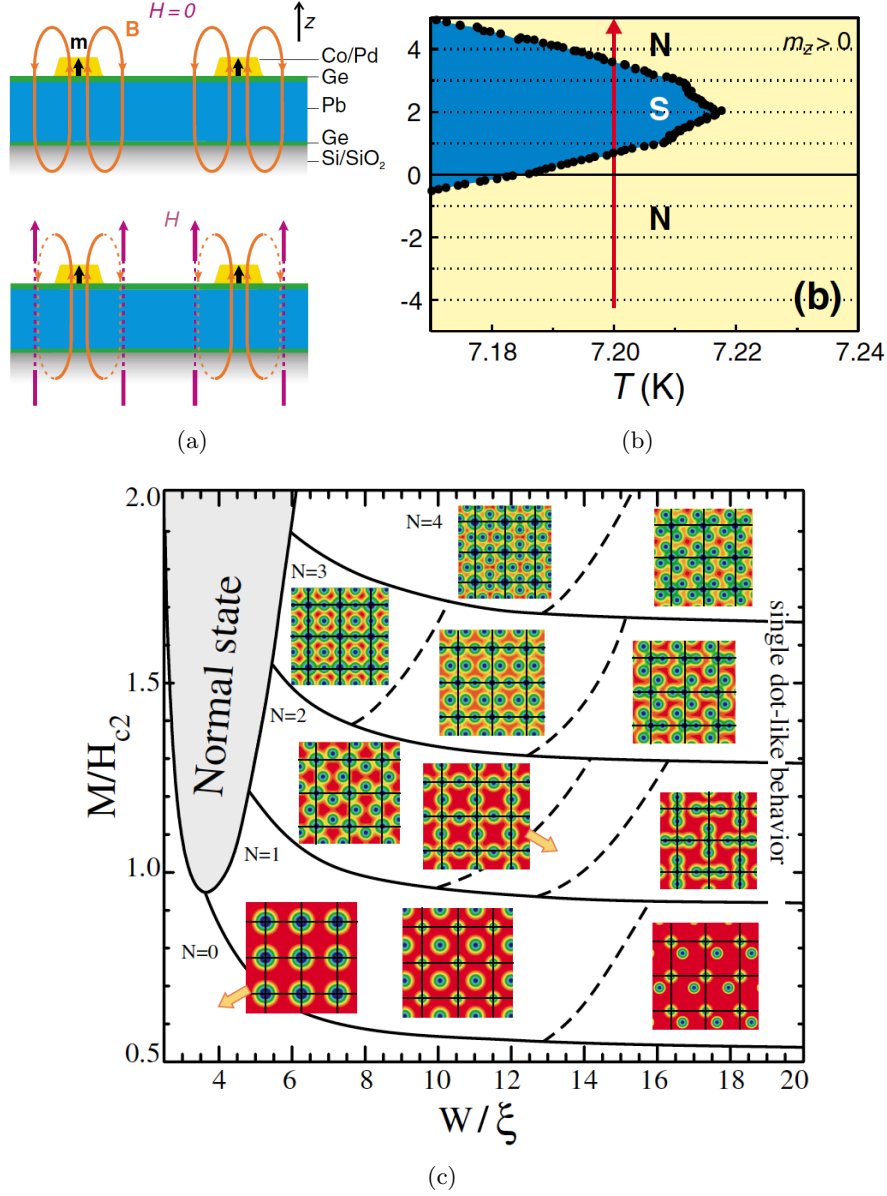


Figure A.1: (a) Schematic representation of the compensation effect of the external magnetic field by the magnetic stray field of magnetic dots (taken from Lange et al. [9]). (b) Phase diagram of superconductivity of the system shown in A.1(a). The phase diagram is shifted to positive fields (taken from Lange et al. [9]). (c) Vortices configurations at zero external field in a superconducting thin film in close proximity with a ferromagnetic dots (cubes of side length equal to 2ξ) square array as a function of the magnetization of the dots (in units of H_{c2} , the second critical field) and their interdistance w (in units of ξ , the coherence length)(taken from Milosevic et al. [135]).

film and thus to the lowest resistive state. Figure A.1(b) shows the phase diagram obtained by Lange et al. [9] for a Pb film on top of CoPt dots. The phase diagram is shifted to H_2 which means that two V-AV pairs are created by each dot.

It has been experimentally observed that the appearance of a V-AV pair requires that the magnetic flux produced on top of the dot roughly exceeds one quantum of flux ϕ_0 [129]. In this case a vortex is created above the dot and an antivortex in between the dots. However, the V-AV creation depends on many parameters, notably the stray field distribution and intensity, the interdistance between the dots, the coherence length and the London penetration depth. Simulations were performed by Milosevic et al. [135] in the framework of the Ginzburg-Landau theory for a superconducting thin film in close proximity to a square array of ferromagnetic dots A.1(c). The dots are cubes with side length of 2ξ , the vortices configurations at zero field are presented as a function of the magnetization of the dots (in units of H_{c2} , the second critical field) and their interdistance w (in units of ξ , the coherence length). A rich variety of states has been revealed by calculating the zero field superconducting ground state as a function of the magnetization of the dots and as a function of the dot interdistance. One important result of their work is that the transition line between the ground state with and without a V-AV pair corresponds to a magnetic flux above each ferromagnetic dot equal to $1.29\phi_0$. More generally, it was roughly found that the appearance of N V-AV pairs requires that the magnetic flux above the dot (in the film) to be more than $N \times \phi_0$.

Magnetostatic simulations have been performed in order to determine the stray field of the Ni nanowires array. A triangular lattice of cylinders with interdistance 100nm and diameter 60nm with magnetization fixed to $\vec{M} = M_s^{Ni} \cdot \vec{e}_z$ ($M_s = 5.1 \cdot 10^5 A/m$). A schematic representation of the model system is shown in figure 4.8(a) for 7 nanowires with length $1.2\mu m$. Simulations with up to 80 nanowires have been performed. The increase of the number of nanowires leads to a decrease of the positive stray field on top of the nanowires and to an increase of the negative field in-between the nanowires. Different calculations have been performed with different nanowire lengths. For length longer than 600nm (10 times the diameter), no significant modification of the stray field is observed. Figure 4.8(b) shows the B_z component of the stray magnetic field along the x direction, i.e. along a row of nanowires, for different distances z from the surface. For the NbN film perforated by the Ni nanowires, the magnetic field profile corresponds approximately to the $z = -10nm$ case. The magnetic flux above each nanowire is then $\phi_{NW} \sim 0.45T \times S_{NW} = 0.61\phi_0$ (S_{NW} is the surface of the nanowires'

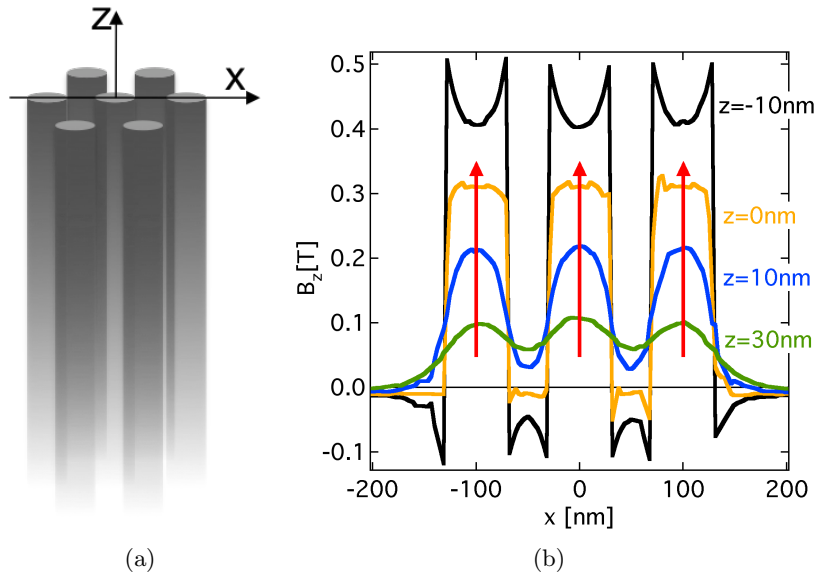


Figure A.2: (a) Schematic representation of the 7 nanowires array used for the simulation of the stray magnetic field. The axis system is also represented. (b) z-component of the stray field of the Ni nanowires array (represented in figure A.2(a)) along the x-axis and for several z values ($z = 0\text{nm}$ corresponds to the nanowire tips). The arrows indicates the nanowires location and the direction of the magnetization. Each nanowire is in the saturated state.

tip). This value is much lower than the $1.29\phi_0$ threshold found by Milosevic et al. This means that, in our case, the dots do not create any V-AV pair. Moreover, a V-AV appears if the magnetic flux between the nanowires (ϕ_{AAO}) is opposite to the one above the dots and also equal to $1.29\phi_0$. This is clearly not the case here because $\phi_{AAO} = 0.29\phi_0$. Experimentally, field-induced superconductivity is observed up to the fifth matching field, therefore, about two V-AV pairs should be created by each nanowire. Moreover, field-induced superconductivity neither manifests itself as a broad region of enhanced T_c nor as a global shift of the phase diagram, but rather as localized enhanced T_c region corresponding to the matching effects. These considerations lead to the conclusion that the FIS observed here is not related to V-AV pairs creation.

A.2 The nucleation of superconductivity

Ferromagnet-superconductor bilayers have shown theoretically and experimentally field-induced superconductivity [10, 137, 162, 163]. The samples consists of a superconducting thin film in close proximity with a ferromagnetic film in the multi-domain state. In this case, the superconducting order parameter was found to nucleate in the region where the local magnetic field was minimum. This effect is also related to compensation effect but without considering V-AV pairs since only superconductivity nucleation is considered. Therefore, this FIS can only take place at the superconducting-normal transition. The superconductivity can nucleate either below magnetic domains or at the domain-walls depending on the total magnetic field in each region. Therefore, resistive minima have been observed for finite magnetic fields. These minima can be easily tuned by changing the domain structure of the ferromagnetic film [162]. However, a resistive criterion close to the normal state is used to probe the nucleation of superconductivity. In our case, no matching dips and field-induced superconductivity is observed for resistive states close to the normal state. Therefore, nucleation of superconductivity above or in-between the nanowires should be ruled out.

A.3 The one-dimensional wire network limit

Field-induced superconductivity have been mentioned in several works on one-dimensional wire networks [159, 160]. Indeed, FIS can result from the reduction of the low field critical temperature due to the enhancement of the frustration of the network since the stray field of the nanowires is varying from one cell of the network to the other. On

the contrary, at high field, the stray field of the nanowires is equal in each unit cell. Therefore, frustration is more present at low field which can lead to apparent FIS. Simulations using the de Gennes - Alexander (dGA) model of networks have been used in order to determine the amplitude of this effect. The dGA model is based on the linearized Ginzburg-Landau equations and is therefore valid close to T_c . The network is decomposed in nodes and strands which are indexed.

The linearized Ginzburg-Landau equation on each strand is :

$$\left[\frac{-1}{\xi^2} + \left(i \frac{\partial}{\partial s} - \kappa \right)^2 \right] \cdot \Delta_{ij} = 0 \quad (\text{A.1})$$

with $s = \hat{u} \cdot \vec{r}$ and $\kappa = \frac{2\pi}{\Phi_0} \cdot \hat{u} \cdot \vec{A}$ the projection of the potential vector on the strand times a constant, Δ_{ij} the order parameter on the strand joining node i to node j.

The continuity of the order parameter at the junctions, i.e. at the nodes, gives (first boundary condition) :

$$\Delta_{ij} = \Delta_i \quad (\text{A.2})$$

The Kirchhoff matching condition on the derivatives of the order parameter at the junctions gives :

$$\left[\sum_j \left(i \frac{\partial}{\partial s_{ij}} - \kappa_{ij} \right) \cdot \Delta_{ij}(s) \right]_i = 0 \quad (\text{A.3})$$

The linearized Ginzburg-Landau (equation A.1) and the two boundary conditions (equations A.2 and A.3) applied to a network with N nodes leads to a set of N linear equations with N unknowns :

$$-\Delta_i \sum_{j=1}^N \cot \theta_{ij} + \sum_{j=1}^N \Delta_j \frac{e^{i\gamma_{ij}}}{\sin \theta_{ij}} = 0 \quad (\text{A.4})$$

with $\gamma_{ij} = \int_i^j \kappa_{ij} ds = \frac{2\pi}{\Phi_0} \int_i^j \vec{A} d\vec{l}$ and $\theta_{ij} = \frac{L_{ij}}{\xi(t)} = \frac{L_{ij}}{\xi(0)} \cdot \sqrt{1-t}$. The set of equations only deals with the order parameter at the nodes. Such set of N equations with N unknowns can be easily solved numerically using basic matrix operations. For N = 1 or 2, it is easily solved analytically. For exemple, a single cell (which can be solved with an arbitrary number of nodes) leads to the following critical temperature :

$$\frac{T_c}{T_{c0}} = 1 - \left(\frac{\pi \xi(0)}{L} \right)^2 \cdot \left(n \pm \frac{\phi_{ext} + \phi_{NW}}{\phi_0} \right)^2 \quad (\text{A.5})$$

with T_{c0} the zero field critical temperature, ϕ_{ext} the magnetic flux of the external field through the cell, ϕ_{NW} the magnetic flux of the stray field of the nanowire through the cell and $\xi(0)$ the zero temperature coherence length. This result is identical to the classical Little-Parks effect with the total magnetic flux being the sum of the external field flux and stray field flux.

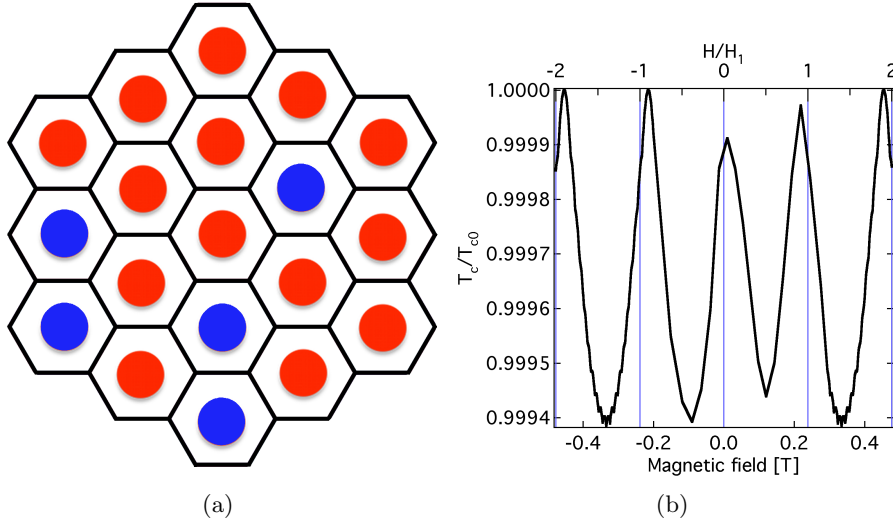


Figure A.3: (a) Schematic representation of the 19-cell honeycomb network used to calculate the phase diagram of superconductivity as a function of the nanowires magnetization. (b) Critical temperature as a function of magnetic field calculated with the dGA model with the stray magnetic flux in each cell equal to $0.1\phi_0$.

Calculations with increasing number of cells have been achieved to validate our program and are in agreement with previous works (see reference [60] and references therein). A network containing 19 cells have been used for the final simulation. The nanowires stray field have been simulated by a narrow gaussian magnetic field distribution (with a given total magnetic flux) located at the center of each cell. This method have been chosen for its simplicity because the stray field distribution inside a cell doesn't impact on the results. Indeed, calculations with different field extensions but with the same total magnetic flux have been performed and identical results were obtained. Figure A.3(a) represent the 19-cell network that was used for the calculations. The blue and red dots corresponds to the nanowires in different magnetization orientation. The number of nanowires with magnetization up or down at a given magnetic field is determined by the magnetization curves of the different samples. The different magnetic states were chosen arbitrarily.

The critical temperature of our virtual network can only be determined for 18 different fields between the two saturation fields due to the limited number of nanowires. The use of a bigger network allows us to get more point in-between the two saturation fields but is more time consuming.

The calculations performed with the 19 cells network corroborate our previous statement : our hybrid sample is not in the 1DWN limit. Indeed, in the saturated state of the nanowire array, the stray field of the nanowires induces to a shift of the $T_c(H)$ curve of a field $H_{shift} = \frac{\phi_{NW} H_1}{\phi_0}$. Such shift is not observed experimentally which can be interpreted either by the fact that the magnetic flux of the nanowire array is weak or by the fact that the sample is not in the 1DWN regime. The strong impact of the nanowires on the superconducting properties as well as the magnetostatic simulations indicates that the magnetic flux of the nanowires is not negligible. Figure A.3(b) shows the calculated phase diagram for a stray magnetic flux in each cell of $0.1\phi_0$. First, a shift of the matching peaks of $0.1H_1$ is observed at high field. Second, at low field (when the nanowire array is not in the saturated state), the first matching peak is not shifted and rather important peak is observed at zero field. It should be mentioned that the low field behavior is better explained using larger magnetic flux for the nanowires but in this case, the high field peaks are strongly shifted from the experimental values. Therefore, it is clear that the observed FIS as well as the first matching field shift can not be explained by the 1DWN regime.

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