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Effects of Hydrostatic Pressure on Critical Current Density & Pinning Mechanism of Iron Based Superconductors and MgB$_2$

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Institute for Superconducting and Electronic Materials

Effects of Hydrostatic Pressure on Critical Current Density & Pinning Mechanism of Iron Based Superconductors and MgB$_2$

Babar Shabbir

This thesis is presented as part of the requirements for the Award of the Degree of Doctor of Philosophy of the University of Wollongong

December 2015
Declaration

I, Babar Shabbir, declare that this thesis, submitted in partial fulfilment of the requirements for the award of Doctor of Philosophy, in the Institute for Superconducting & Electronic Materials (ISEM), Faculty of Engineering, University of Wollongong, Australia, is wholly my own work unless otherwise referenced or acknowledged. This document has not been submitted for a qualification at any other academic institution.

Babar Shabbir
December 09, 2015
**ABSTRACT**

The iron based superconductors have revealed distinctive superconducting properties, including reasonable critical temperature ($T_c \approx 56$ K), extremely high upper critical field ($H_{c2}$) on the order of 100 T, high intrinsic pinning potential, low anisotropy values, generally between 1-8, high irreversibility field ($H_{irr}$), and high critical current density ($J_c$), which is of vital importance for low and high field application. Enhancement of $J_c$ or flux pinning has always been an important focus of superconductor research over the past decade. Usually, three approaches have been used to increase the $J_c$ in cuprates, MgB$_2$, and iron based superconductors:

- Texturing processes to reduce the mismatch angle between adjacent grains and thus overcome the weak-link problem in layer-structured superconductors;
- High energy ion implantation or irradiation to introduce point defect pinning centres; and;
- Chemical doping, which can induces more point pinning centres.

Weak-links and weak flux pinning are the predominant factors causing low $J_c$ values, specifically at high fields and temperatures in pnictide polycrystalline bulks, and these problems must be overcome. In order to enhance the $J_c$ in granular superconductors, we have to increase/improve: i) grain connectivity; ii) point defects inside grains; and iii) $T_c$ enhancement, which can increase the effective superconducting volume as well as $H_{irr}$ and $H_{c2}$. Furthermore, elimination of weakly linked grain boundaries (GBs) is a prerequisite for improving the $J_c$ values. Moreover, the nature of the pinning mechanism plays an important role in obtaining good $J_c$ field dependence values along with a
suitable pinning force, which can boost pinning strength and, in turn, leads to higher values of $J_c$. Although chemical doping and high energy particle irradiation can induce point defects, they can only enhance $J_c$ either in high fields or low fields, with degradation of $T_c$. Therefore, the most ideal approach should be one which can improve grain connectivity (in the case of polycrystalline bulks and wires/tapes), induce more point defects, increase superconducting volume and $T_c$, and especially enhance $J_c$, at both low and high fields.

It has been reported that hydrostatic pressure has a positive effect on $T_c$ in cuprates and pnictide superconductors. Hydrostatic pressure can reduce anisotropy, improve the grain connectivity (in the case of polycrystalline bulks and wires/tapes), induce more point pinning centres, and increase the superconducting volume, $H_{irr}$, and $H_{c2}$. Therefore, the hydrostatic pressure approach was chosen for investigation of its effects on $J_c$ and the pinning mechanism in granular superconductors, single crystals, and tapes.

We found that hydrostatic pressure up to 1.2 GPa can not only significantly increase $T_c$ from 15 K (underdoped) to 22 K, but also significantly enhances the irreversibility field, $H_{irr}$, by a factor of 4 at 7 K, as well as the critical current density, $J_c$, by up to 30 times at both low and high fields for Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ polycrystalline bulks. It was found that pressure can induce more point defects, which are mainly responsible for the $J_c$ enhancement. In addition, pressure can also transform surface pinning to point pinning in Sr$_4$V$_2$O$_6$Fe$_2$As$_2$.

Hydrostatic pressure can also significantly enhance $J_c$ in NaFe$_{0.972}$Co$_{0.028}$As single crystals by at least tenfold at low field and more than a hundredfold at
high fields. Significant enhancement in the in-field performance of NaFe$_{0.972}$Co$_{0.028}$As single crystal in terms of pinning force density ($F_p$) is found at high pressures. At high fields, the $F_p$ is over 20 and 80 times higher than under ambient pressure at 12 K and 14 K, respectively, at $P = 1$ GPa. We have discovered that the hydrostatic pressure induces more point pinning centres in a sample, and the pinning centre number density found at 1 GPa is almost twice as high as under ambient pressure at 12 K and over six times as high at 14 K.

We also demonstrated strong flux pinning over a wide field range under hydrostatic pressure in optimally doped Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystal by enhancing the pinning force via the formation of giant vortexes, which can, in turn, cause significant enhancement of the critical current density in both low and high fields. Additionally, we also discuss a fundamental mechanism related to $J_c$ enhancement with unchanged superconducting critical temperature values under hydrostatic pressure. The pressure of 1.2 GPa improved the $F_p$ by nearly 5 times at 8, 12, 24, and 28 K, which can increase $J_c$ by nearly two-fold at 4.1 K and five-fold at 16 K and 24 K in both low and high fields, respectively. This study also revealed that such an optimally doped superconductor’s performance in both low and high fields can also be further enhanced by pressure.

Finally, the impact of hydrostatic pressure of up to 1.2 GPa on the $J_c$ and the nature of the pinning mechanism in MgB$_2$ have been investigated within the framework of the collective theory. We found that the hydrostatic pressure can induce a transition from the regime where pinning is controlled by spatial variation in the critical transition temperature ($\delta T_c$) to the regime controlled by spatial variation in the mean free path ($\delta \ell$). Furthermore, $T_c$ and low field $J_c$ are
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Dedicated to my family and friends
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1 INTRODUCTION

The discovery of iron-based superconductors (FeSCs) in 2006 brought new motivation to the field of high-temperature superconductivity and strongly correlated electron systems [1, 2]. The FeSCs consist of FeAs layered structures alternating with spacer or charge reservoir blocks. The FeSCs can thus be classified into the pnictide “1111” system for RFeAsO (R: a rare earth element), including LaFeAsO, SmFeAsO, PrFeAsO, etc.; into the “122” type for BaFe$_2$As$_2$, SrFe$_2$As$_2$, or CaFe$_2$As$_2$; into the “111” type for LiFeAs, NaFeAs, LiFeP, and chalcogenides; or into the “11” type such as FeSe, etc. [3-15]. The parent compound of pnictides is semimetallic, in contrast to the cuprates, as their parent compounds are Mott insulators. The pnictide superconductors exhibit distinctive superconducting properties, including reasonable critical temperature ($T_c \approx 56$ K), extremely high upper critical field ($H_{c2}$) on the order of 100 T, high intrinsic pinning potential, low anisotropy values, generally between 1-8, high irreversibility field ($H_{irr}$), and high critical current density ($J_c$), which is the predominant limiting factor for low and high field applications [16-20]. Strong pinning has been observed in pnictides, which gives rise to high $J_c$ values in both single crystals and thin films. The oxypnictide SmFeAs(O,F) superconducting films show high $J_c$ values (~$10^6$ A/cm$^2$ at 4.2 K) in low fields [21]. Similarly, SmFeAsO$_{0.7}$F$_{0.25}$ single crystals reveal a promising combination of high $J_c$ values (> $10^6$ A cm$^{-2}$ at 5 K) and nearly isotropic critical current densities along all crystal directions [22]. The $J_c$ for SmFeAsO$_{0.5}$F$_{0.15}$ crystals has been enhanced up to $10^7$ A cm$^{-2}$ at 5 K [23]. High critical current density $J_c$ of 4 MA/cm$^2$ at 4 K was obtained in Co-doped BaFe$_2$As$_2$ (BaFe$_2$As$_2$:Co) epitaxial films [24]. $J_c$ of 5 MA/cm$^2$ at low fields and
temperatures is reported for Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystals [25]. BaFe$_2$(As$_{0.66}$P$_{0.33}$)$_2$ films exhibited nearly isotropic critical current densities in excess of 1.5 MA cm$^{-2}$ at 15 K and 1 T, which are seven times higher than previously reported for BaFe$_2$As$_2$ films [26]. The point defects induced by heavy-ion and proton irradiation in Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ single crystal increased the $J_c$ up to 5 MA/cm$^2$ at 5 K and 7 T [27]. A $J_c$ value of 10$^6$ A/cm$^2$ has also been found in FeSe films at 2 K [28]. Usually, $J_c$ values in polycrystalline bulks and tapes/wires are lower as compared to single crystals and films due to weak flux pinning. $J_c$ values of nearly 10$^4$ - 10$^5$ A/cm$^2$ have been obtained at high fields and temperatures for polycrystalline bulks and tapes/wires, although they are lower than what are required for practical applications [29]. At 4.2 K and low fields, transport $J_c$ of 10$^4$ A/cm$^2$ has been found in Sn-added Sr$_{1-x}$K$_x$Fe$_2$As$_2$ superconducting tapes [30]. Similarly, (Sr,K)Fe$_2$As$_2$ superconducting wire prepared by the powder-in-tube (PIT) method combined with a hot isostatic pressing (HIP) technique provided a transport $J_c$ value of 10$^5$ A/cm$^2$ at 4.2 K under self-field [31]. $J_c$ as a function of applied magnetic field at 4.2 K for the K-doped wire exhibited $J_c$ values of nearly 10$^5$ A/cm$^2$ at low fields [32].

Although $J_c$ values at low fields and temperatures are high to some extent, the major drawbacks are that $J_c$ decays rapidly in high fields, especially at high temperatures. Enhancement of $J_c$ or flux pinning has always been a leading area of research over the past decade. Usually, three approaches have been used to increase the $J_c$ in cuprates, MgB$_2$, and iron based superconductors:

- Texturing processes to reduce the mismatch angle between adjacent grains and thus overcome the weak-link problem in layer-structured superconductors;
- Irradiation to introduce point defect pinning centres; and;
- Chemical doping, which can induce more point pinning centres.

Weak-links and weak flux pinning are the predominant factors causing low $J_c$ values, specifically at high fields and temperatures in pnictide polycrystalline bulks, so these problems must be overcome. In order to enhance the $J_c$ in granular superconductors, we have to increase/improve: i) grain connectivity; ii) point defects inside grains; and iii) $T_c$ enhancement, which can increase the effective superconducting volume as well as $H_{irr}$ and $H_{c2}$.

We have taken into account the following facts regarding the flux pinning mechanism in polycrystalline pnictide superconductors. Elimination of the weakly linked grain boundaries (GBs) is a prerequisite for improving the $J_c$ values, as the coherence length for pnictides is very short ($\xi$ of a few nm) [33]. Furthermore, the nature of the pinning mechanism plays an important role in improving the $J_c$ field dependence values along with the pinning force, which can boost the pinning strength and, in turn, leads to higher values of $J_c$. The ideal size of defects for pinning should be comparable to the coherence length [34]. Therefore, point defect pinning is required for high $J_c$ values in contrast to surface pinning, because its pinning force is stronger than that for surface pinning at low and high field, according to the Dew-Hughes model [35]. Thus, it is desirable to induce more point defects. Although chemical doping and high energy particle irradiation can induce point defects and enhance $J_c$, especially in high field, $T_c$ and low field $J_c$ deteriorate greatly for various types of superconductors. Therefore, the most ideal approach should be one which can improve grain connectivity (in the case of polycrystalline bulks and
wires/tapes), induce more point defects, and increase the superconducting volume and $T_c$.

It has been reported that hydrostatic pressure has a positive effect on $T_c$ in cuprate and pnictide superconductors. High pressure of 150 kbars can raise the $T_c$ of Hg-1223 significantly, from 135 K to a record high 153 K [36]. The $T_c$ of hole-doped (NdCeSr)CuO$_4$ has been increased from 24 to 33 K at 3 GPa [37]. The enhancement of $T_c$ for Y$_2$B$_4$C$_7$O$_{14}$ is more than 10 K at 2 GPa [38]. It should be noted that pressure also shows positive effects on $T_c$ for various pnictide superconductors. Pressure can result in improvement of $T_c$ from 28 K to 43 K at 4 GPa for LaOFFeAs [39]. For Co doped NaFeAs, the maximum $T_c$ can reach as high as 31 K from 16 K at 2.5 GPa [40]. Pressure can enhance the $T_c$ of La doped Ba-122 epitaxial films up to 30.3 K [41]. A huge enhancement of $T_c$ from 13 K to 27 K at 1.48 GPa, with even a high value of 37 K at 7 GPa, has also been reported for FeSe [42, 43]. In addition, pressure can 1) reduce the lattice parameters and cause the shrinkage of unit cells, giving rise to reduction of the anisotropy; 2) improve the grain connectivity because of compression of both grains and grain boundaries (GBs); 3) induce more point pinning centres, as the formation energy of point defects decreases with increasing pressure. Pressure can also cause low-angle GBs to migrate in polycrystalline bulk samples, resulting in the formation of giant grains and leading to the sacrifice of surface pinning centres thereafter. Therefore, a higher ratio of point pinning centres to surface pinning centres is expected due to the formation energy and migration of GBs under pressure. The significant enhancement in $T_c$ under pressure means that superconducting volumes should be increased greatly compared to $T_c$ without pressure. Therefore, it was expected that hydrostatic
pressure would increase superconducting volume, $H_{irr}$, and $H_{c2}$ due to $T_c$ enhancement, increase the pinning centre number density by inducing more point defects, improve grain connectivity (in the case of polycrystalline bulks and wires/tapes), and reduce the anisotropy in pnictide superconductors. There is some evidence for $J_c$ enhancement (not more than one order of magnitude) under pressure for some conventional and unconventional superconducting materials, which are listed in Table 1-1. The origin of the $J_c$ enhancement is still unclear, however. Therefore, detailed investigations are required to examine pnictide $J_c$ under pressure (especially to obtain the maximum in-field performance) and reveal the cause of $J_c$ enhancement.

<table>
<thead>
<tr>
<th>No</th>
<th>Material</th>
<th>Pressure effect on $J_c$</th>
<th>Pressure effect on $T_c$</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Nb-Zr Alloys</td>
<td>15% enhancement for 1.75 GPa</td>
<td>$T_c$ not changed</td>
<td>[44]</td>
</tr>
<tr>
<td>2</td>
<td>Nb$_3$Sn</td>
<td>Negative</td>
<td>Negative</td>
<td>[45]</td>
</tr>
<tr>
<td>3</td>
<td>Tl$_2$CaBa$_2$Cu$_4$O$_7$ Bulk</td>
<td>Twofold enhancement</td>
<td>Positive</td>
<td>[46]</td>
</tr>
<tr>
<td>4</td>
<td>YBa$_2$Cu$_3$O$_7$ Bulk</td>
<td>Fivefold enhancement</td>
<td>Positive</td>
<td>[46]</td>
</tr>
<tr>
<td>5</td>
<td>YBaCuO rings</td>
<td>Positive</td>
<td>Positive</td>
<td>[47]</td>
</tr>
<tr>
<td>6</td>
<td>YBCO Bulk</td>
<td>Positive</td>
<td>No effect</td>
<td>[48]</td>
</tr>
<tr>
<td>7</td>
<td>YBCO Bicrystalline ring</td>
<td>+20%/GPa</td>
<td>Positive</td>
<td>[49]</td>
</tr>
<tr>
<td>8</td>
<td>Bi-2223 Superconducting Wire</td>
<td>Negative (0.9 GPa)</td>
<td>Negative</td>
<td>[50]</td>
</tr>
<tr>
<td>9</td>
<td>FeSe$<em>{0.5}$Te$</em>{0.5}$</td>
<td>Enhanced by one order of magnitude</td>
<td>Enhanced by 7 K at 1 GPa</td>
<td>[51]</td>
</tr>
</tbody>
</table>

Table 1-1: Pressure investigations on $J_c$ and $T_c$ of some superconducting materials.

We have used the hydrostatic pressure approach to investigate its effects on $J_c$ and pinning mechanisms in granular superconductors and single crystals, which are systematically discussed in Chapters 4, 5, and 6. We also propose that the pressure approach can be used, even when all the other approaches used are
exhausted, to further improve the $J_c$ in superconductors. Moreover, we have also investigated pressure effects on pinning in MgB$_2$ (Chapter 7). The following are the summaries of our main results:

A) We found that hydrostatic pressure up to 1.2 GPa can not only significantly increase $T_c$ from 15 K (under-doped) to 22 K, but also significantly enhances the irreversibility field, $H_{irr}$, by a factor of 4 at 7 K, as well as the critical current density, $J_c$, by up to 30 times in both low and high fields for Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ polycrystalline bulks. It was found that pressure can induce more point defects, which are mainly responsible for the $J_c$ enhancement. In addition, pressure can also transform surface pinning to point pinning in Sr$_4$V$_2$O$_6$Fe$_2$As$_2$.

B) Hydrostatic pressure can significantly enhance $J_c$ in NaFe$_{0.972}$Co$_{0.028}$As single crystals by at least tenfold at low field and more than a hundredfold at high fields. Significant enhancement in the in-field performance of NaFe$_{0.972}$Co$_{0.028}$As single crystal in terms of pinning force density ($F_p$) is found at high pressures. At high fields, the $F_p$ is over 20 and 80 times higher than under ambient pressure at 12 K and 14 K, respectively, at $P = 1$ GPa. We have discovered that the hydrostatic pressure induces more point pinning centres in a sample, and the pinning centre number density found at 1 GPa is almost twice as high as under ambient pressure at 12 K and over six times as high at 14 K.

C) We also demonstrated strong flux pinning over a wide field range under hydrostatic pressure in optimally doped Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystal by enhancing the pinning force via the formation of giant vortexes, which can, in turn, cause significant enhancement of the critical current density in both low and high fields. Additionally, we also discuss a fundamental mechanism related to
$J_c$ enhancement with unchanged superconducting critical temperature values under hydrostatic pressure. The pressure of 1.2 GPa improved the $F_p$ by nearly 5 times at 8, 12, 24, and 28 K, which can increase $J_c$ by nearly two-fold at 4.1 K and five-fold at 16 K and 24 K in both low and high fields, respectively. This study also revealed that such an optimally doped superconductor’s performance in both low and high fields can also be further enhanced by pressure.

D) The impact of hydrostatic pressure of up to 1.2 GPa on the $J_c$ and the nature of the pinning mechanism in MgB$_2$ have been investigated within the framework of the collective theory. It has been demonstrated that hydrostatic pressure can induce a transition from the regime where pinning is controlled by spatial variation in the critical transition temperature ($\delta T_c$) to the regime controlled by spatial variation in the mean free path ($\delta \ell$). Furthermore, $T_c$ and low field $J_c$ are only slightly reduced, although the $J_c$ drops more quickly at high fields than at ambient pressure. The hydrostatic pressure can reduce the density of states [$N_s(E)$], which, in turn, leads to a reduction in the critical temperature from 39.7 K at $P = 0$ GPa to 37.7 K at $P = 1.2$ GPa.
1.1 References


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32. Weiss JD, et al. High intergrain critical current density in fine-grain (Ba$_{0.6}$K$_{0.4}$)Fe$_2$As$_2$ wires and bulks. *Nat. Mater.* **11**, 682-685 (2012).


51. Pietosa J et al., Pressure-induced enhancement of the superconducting properties of single-crystalline FeTe$_{0.5}$Se$_{0.5}$. J. Phys: Condens. Matt. 24, 265701 (2012).


2 LITERATURE REVIEW

2.1 History of Superconductivity

Superconductivity is a macroscopic quantum phenomenon of infinite electrical conductivity or zero electrical resistance and exclusion of magnetic fields, occurring in different elements, as well as intermetallic alloys and compounds, when cooled below a specific temperature, which is well-known as the critical temperature, $T_c$. This phenomenon was discovered by the Dutch physicist Heike Kamerlingh Onnes (Figure 2-1: Right) on April 8, 1911 in Leiden, while he was measuring the electrical resistance of mercury and found that it suddenly drops to zero at a temperature of 4.2 K, as shown in Figure 2-1: Left [1].

![Figure 2-1: Resistance versus Temperature plot for mercury obtained by Kamerling Onnes (Left). Photograph of Kamerlingh Onnes (Right).](image)

It has been found that twenty-one metallic elements and many different alloys and compound materials exhibit superconductivity. The Periodic Table in Figure 2-2 shows all known elemental superconductors and their $T_c$. 

23
There are the two characteristics of all superconductors, i.e., zero resistivity and the Meissner effect. In 1933, the German physicists Walther Meissner and Robert Ochsenfeld discovered a unique property of superconductors, that a superconductor can completely expel magnetic field from its interior during its transition to the superconductivity state from the normal state [2].

Figure 2-3: Meissner effect, the expulsion of external magnetic field from inside the superconductor in the superconducting state by creating surface current. The field is applied at (a) \( T > T_c \) and (b) \( T < T_c \) (image taken from Wikipedia).
In 1935, the macroscopic London theory explained the electrodynamics of superconductors by introducing two new equations in addition to Maxwell equations [3]. These equations can explain the experimental observations, i.e., the Meissner effect and zero resistance. Another macroscopic phenomenological theory, Ginzburg–Landau (GL) theory (1950), thoroughly explained the macroscopic properties of superconductors [4]. Nevertheless, neither theory (the London theory and the GL theory) described the microscopic origins of the superconducting properties. The first microscopic theory was proposed by Bardeen, Cooper, and Schrieffer (BCS) in 1956 [5]. Later on, Bardeen, Cooper, and Schrieffer were awarded the Nobel Prize for it. A significant contribution was also made by N. N. Bogolyubov [6]. BCS theory is based on the electron-electron attraction resulting in what is generally known as Cooper pairs (total spin of Cooper pair is zero), which occurs below $T_c$ through phonons (emitted from the vibrations of ions). L. P. Gorkov elaborated the microscopic theory further by showing that BCS theory can be interrelated to GL theory in the vicinity of $T_c$ [7]. It is noteworthy that BCS theory can only explain the pairing mechanism in conventional superconductors. A. A. Abrikosov (1957) categorized superconducting materials into two groups, depending on their behaviour in magnetic fields: type I and type II superconductors [8]. The magnetization response of type I and type II superconductors are shown in Figure 2-4.

For $H > H_c$, superconductivity is completely destroyed in type-I superconductors, where $H$ is the strength of the applied field and $H_c$ is a critical field. In contrast, the response of type II superconductors to $H$ is different. Rather, the formation of magnetic vortices occurs in a certain applied magnetic field range, i.e., between $H_{c1}$ and $H_{c2}$. Below $H_{c1}$, the magnetic field cannot penetrate inside the material, and
superconductivity disappears above $H_{c2}$. The magnetic vortex can be imagined as a cylinder which is aligned to the applied field. The superconducting order parameter is zero inside the cylinder. These vortices are surrounded by a superconducting region.

The GL coherence length, $\xi_{GL}$, can be estimated from the radius of the cylinder [2]. The vortices arrange themselves into a regular pattern known as a vortex lattice. The vortex pattern depends mostly on the structure of the material. The vortexes can be pinned by defects (due to grain boundaries, impurity particles, and or crystal imperfections) in the material, a phenomenon known as flux pinning. To prevent flux creep/motion, which can generate resistance and reduce the $J_c$ and $H_{c2}$, flux pinning plays an important role in terms of applications. The pinning effect is not effective at a certain magnetic field, commonly known as the irreversibility field, $H_{irr}$.

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In 1962, the British physicist Brian David Josephson discovered the tunnelling property of superconducting Cooper pairs and was awarded the Nobel prize in Physics [9, 10]. He demonstrated that without any voltage being applied, a current can flow indefinitely long through a Josephson junction (JJ), which consists of two superconductors separated by a thin insulating barrier (weak link). Josephson junctions have many applications, especially in superconducting qubits, superconducting quantum interference devices, and in rapid single flux quantum digital electronics.

In 1986, the IBM researchers Bednorz and Müller achieved the first milestone in modern era of superconductivity by discovering superconductivity in a lanthanum-based cuprate perovskite material ($T_c \approx 35$ K) [11]. Soon after, the replacement of lanthanum by yttrium (i.e., yttrium barium copper oxide, YBCO) increased the $T_c$ to 92 K, which is above the maximum of 30 K predicted by BCS theory [12]. Their noble discovery was aroused great hopes and motivated scientists to achieve more milestones in the field of superconductivity.

![Figure 2-6: $T_c$ of some known superconductors versus the year of discovery, as compiled by the Applied Superconductivity Group, University of Geneva, Switzerland](image)
Another conventional superconductor, MgB$_2$ (which could be explained by BCS theory) with $T_c$ of 40 K, was discovered in 2001 [13]. Recently, Hideo Hosono of the Tokyo Institute of Technology, Japan discovered superconductivity in iron based compounds, generally known as iron based superconductors [14, 15].

2.2 Pinning Mechanism

Generally, the fundamental interactions between pinning centres and vortices are the magnetic and core interactions in high $T_c$ superconductors. The interaction of surfaces between superconducting and non-superconducting material parallel to the applied field results in magnetic interactions, which are commonly very small in type-II superconductors with a high Ginzburg-Landau (GL) parameter, $\kappa$. The core interaction arises from the coupling of the locally distorted superconducting properties with the periodic variation of the superconducting order parameter, which is significantly more effective in type-II superconductors due to the high $\kappa$ value.

There are two predominant mechanisms of core pinning, i.e., $\delta T_c$ pinning, which is associated with spatial fluctuations of the $T_c$ and $\delta \ell$ pinning, which is associated with fluctuations in the charge carrier mean free path, $\ell$ [16-22].

The Dew Hughes model provides a good explanation of the pinning mechanism in terms of the vortex pinning force $F_p = J_c \times B$ [23]. D. Dew.Hughes proposed a scaling law, i.e. $F_p \propto h^m (1 - h)^n$ to determine the nature of the pinning mechanism, where $h$ is the reduced field, $h = B/B_{irr}$ (where $B_{irr}$ is the irreversibility field, and $m$ and $n$ are two basic parameters), which can indicate the origin of the pinning mechanism. In case of $\delta \ell$ pinning, $m = 2$ and $n = 2$ with no $h_{\max}$ implies volume pins; $m = 0.5$ and $n = 2$ with $h_{\max} = 0.2$ for surface/grain boundary pinning; and $m = 1$ and $n = 2$ with $h_{\max} = 0.33$ for point pinning. Similarly, in the case of $\delta T_c$ pinning, $m =$
$n=1$ with $h_{\text{max}} = 0.5$ corresponds to volume pins; $m = 3/2$ and $n = 1$ with $h_{\text{max}} = 0.6$ relates to surface/grain boundary pinning; and $m = 1$ and $n = 1$ with $h_{\text{max}} = 0.67$ indicates point pinning. D. Dew Hughes summarized his work on the nature of the pinning mechanism in a tabular form. As discussed earlier, the core pinning mechanism is more significant in type II superconductors, so only scaling parameters (i.e., $m$ and $n$) along with $h_{\text{max}}$ for core pinning need to be presented in Table 2-1.

<table>
<thead>
<tr>
<th>Types of Interaction</th>
<th>Geometry of Pin</th>
<th>Type of centre</th>
<th>Scaling parameters</th>
<th>Position of maximum</th>
</tr>
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<td>Core</td>
<td>Volume</td>
<td>$\delta l$</td>
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<td>$h=0.67$</td>
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</tbody>
</table>

Table 2-1: Scaling parameters for core pinning according to the Dew Hughes Model [23].

2.3 IRON BASED SUPERCONDUCTORS (FeSCs)

Superconductivity in FeSCs was discovered by Kamihara [14, 15]. Although superconductivity in iron related compounds (such as U$_6$Fe: $T_c \approx 3.9$ K and Lu$_2$Fe$_3$Si$_5$: $T_c \approx 6.1$ K) have been known to the scientific community for some time [24, 25], but due to the strong local magnetic moment of iron, they didn’t attract
much attention at that time. This ground-breaking discovery by Kamihara has led to the discovery of other iron based superconducting families. The iron pnictides (FePn, where Pn is As or P), including iron chalcogenides (FeCh, where Ch includes S, Se, and Te) have been widely investigated over the last few years [26]. High $T_c$ values of around 56 K have been found in 1111 type structures (discussed later), which is high in comparison with other superconductors, except for the cuprates [27]. $T_c$ around 38 K, 20 K and 14 K are also reported for 122, 111, and 11 type structures, respectively, which can be further enhanced by hydrostatic pressure, doping, etc. [26]. The superconducting pairing mechanism may be related to the coexistence of magnetism with superconductivity in the phase diagram. In FeSCs, it is generally believed that phonon mediation does not play a role in pairing. Theoretical assumptions include several electronic instabilities that could mediate the superconducting pairing, e.g., spin fluctuations or interorbital pair hopping. Besides some similarities, the properties of the FeSCs are basically different from those of the cuprates. Both have layered structure and unconventional, high values of $J_c$ and $H_{c2}$. In contrast, FeSCs are semimetallic, whereas the parent compounds of cuprates are Mott-insulators. Furthermore, FeSCs have less anisotropy as compared to cuprates. FePn forms the FeAs layered structure, with a spacer or charge reservoir block. The compounds can be classified as belonging to the “1111” system of RFeAsO, where R is a rare earth element (such as LaFeAsO, SmFeAsO and PrFeAsO etc). the “122” type, including BaFe$_2$As$_2$, SrFe$_2$As$_2$, or CaFe$_2$As$_2$; the “111” type of LiFeAs, NaFeAs, LiFeP, etc. and iron chalcogenides categorized as “11” type, such as FeSe, etc. [28].
2.3.1 Crystal Structure and Phase Diagram

Figure 2-7 shows the crystal structures of the different families of FeSCs. The elementary constituent is a quasi-two-dimensional layer consisting of a square lattice of iron atoms with tetrahedrally coordinated ionic bonds to either phosphorus, arsenic, selenium, or tellurium anions that are positioned above and below the iron lattice, forming a common blocking layer.

![Crystal structures](image)

Figure 2-7: Crystal structures (tetragonal with the c-axis pointing up) of FeSCs: FeSe, LiFeAs, LaOFeAs, and Sr$_2$VO$_3$FeAs crystallize in space group P4/nmm, and BaFe$_2$As$_2$ in I4/mmm. Iron atoms: dark grey spheres, arsenic (selenium):black spheres, and cations: light grey balls [29].

The FeAs layer is an arrangement of Fe-As covalent bonding and Fe-Fe metallic bonding [30]. It is widely believed that superconductivity emerges from the iron layers. The Fe and As/Se atoms forms Fe(As/Se)$_4$ tetrahedra, whereas the $T_c$ of FeSCs mostly depends on the angle between the FeAs/Se bonds and the height of the tetrahedra [30-32]. The 1111 compounds such as LaFeAsO, LaFePO, etc. are considered as belonging to the ZrCuSiAs type crystal structure with space group P4/nmm [33]. It comprises negatively charged FeP$_n$ layers, where the Fe atoms form
a planar square lattice, and positively charged rare earth oxide (REO) layers, with RE a rare earth element. The 122 compounds have the ThCr$_2$Si$_2$ type structure with space group I4/mmm [34]. The 111 compounds are categorized as the anti-PbFCl type [35]. The 11-FeCh compounds simply have the $\alpha$-PbO type structure with space group P4/nmm and contain a stack of FeCh$_4$ tetrahedral layers [36]. Finally, the structures of the more complicated compounds such as Sr$_2$VO$_3$FeAs are found to be of Sr$_2$GaO$_3$CuS-type [37].

The phase diagrams for different families of FeSCs are shown in Figure 2-8. For LaO$_{1-x}$F$_x$FeAs (1111 type compounds), 100% superconducting volume fraction appears as soon as the orthorhombic distortion and the spin density wave (SDW) magnetism are suppressed. The antiferromagnetic (AFM) and superconducting (SC) phases do not overlap for different doping concentrations, which is not the case for others FeSCs. High $T_c$ values found for around $x = 0.08$ and 0.10 [38]. An experimental phase diagram for the Ba-based 122 system has been compiled by

![Figure 2-8: Phase diagrams for different families of FeSCs [30, 38-40].](image-url)
Paglione et al. [30]. It consists of an AFM state that is eliminated with substitution and a superconducting dome that is nearly centred near the critical concentration where the AFM order is eliminated. The coexistence of the AFM and SC phases is considered as an intrinsic property of the generic FeSC phase diagram. The coexistence of AFM and SC over a region of $x$ that is less than 0.025 has been found for 111 type compounds. A concentration of nearly 0.03 provides high values of $T_c$ [40]. For 11 compounds, superconductivity appears through the Se doping, with $T_c$ increasing gradually up to 16 K for $x = 0.5$, by suppressing AFM [39].

2.3.2 Superconducting Properties of FeSCs

FeSCs have revealed remarkable properties in recent times. Great progress has been made in further enhancing their superconducting properties so that they can be used in practical applications, by replacing other conventional superconductors such as NbTi, etc., which have much lower $T_c$ values than FeSCs. Having layered structures, FeSCs are less anisotropic than cuprates and reveal high values of $T_c$, $J_c$, and $H_{c2}$. Details are as follows.

2.3.2.1 Critical Temperature ($T_c$)

High $T_c$ values of nearly 56 K were found in FeSCs soon after the discovering of this new family. The $T_c$ of FeSCs mostly depends on the angle between the FeAs/Se bonds and the height of the tetrahedra in most cases [30]. Kamihara et al., found superconductivity ($T_c = 26$ K) in LaFeAsO after fluorine doping [15]. Takahashi et al. increased the $T_c$ of this compound up to 43 K at $P = 4$ GPa [41]. Soon after, researchers found high values of $T_c$ (above 50 K) in compounds with the same
structure $[\text{RFeAs(O}_{1-x}\text{F}_x)]$ by doping with lanthanides such as Sm, Nd, Pr, Gd, etc. [42-45]. Researchers also reported $T_c$ values of 56 K in non-oxypnictides [46]. The superconductivity that emerges in 122 compounds is due to hole/electron/isovalent doping or by applying external pressure [47]. Superconductivity at 38 K is reported in $\text{Ba}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ [48], which is the highest among the 122 compounds. In addition, $T_c$ values of 32 K, 26 K, 26 K, and 22 K have also been reported for $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$, $\text{Sr}_{0.6}\text{Na}_{0.4}\text{Fe}_2\text{As}_2$, $\text{Ca}_{0.6}\text{Na}_{0.4}\text{Fe}_2\text{As}_2$, and $\text{BaFe}_{1.8}\text{Co}_{0.2}\text{Fe}_2\text{As}_2$, respectively [49-51]. Superconductivity has been found in undoped 111 compounds such as $\text{LiFeAs}$ at 18 K [52], and between 9-25 K for $\text{NaFeAs}$ [53, 54]. Scientists also have discovered superconductivity in simple structured FeSe compounds at $T_c \approx 8$ K [55, 56]. Furthermore, $T_c$ values of 14 K and 31.5 K have also been reported for $\text{FeTe}_{0.5}\text{Se}_{0.5}$ and $\text{K}_{0.82}\text{Fe}_{1.63}\text{Se}_2$ [57, 58].

2.3.2.2 $H_{c2}$ and $H_{irr}$

FeSCs reveals extremely high $H_{c2}$ values as compared to other superconductors (Figure 2-9). Different groups have estimated $H_{c2}$ to be between 100 and 300 T by using the Werthamer–Helfand–Hohenberg (WHH) model [59, 60] for different FeSCs. Details for the WHH model are found elsewhere [61]. Besides high values of $H_{irr}$, the $H^{ab}_{c2} (T = 0 \text{ K}) \approx 304 \text{ T}$ and $H^c_{c2} (T = 0 \text{ K}) \approx 62–70 \text{ T}$ have been found in single crystals of $\text{NdFeAsO}_{0.82}\text{F}_{0.18}$ along with anisotropy < 6, which is less than that of YBCO [60]. $H_{c2} (T = 0)$ values of 150 T and $H_{irr} (15 \text{ K}) \approx 25 \text{ T}$ have also been reported for polycrystalline $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ samples [62]. The value of $H^{ab}_{c2}$ is estimated to be well above 100 T for fluorine doped $\text{LaFeAsO}$, with anisotropy around 2.3 for 5% fluorine doping and above 50 T for bulks [63].
The nearly isotropic nature of superconductivity in the hole doped compound (Ba\(_{1-x}\)K\(_x\))Fe\(_2\)As\(_2\) has been reported [64]. The \(H_c2\) increases linearly with decreasing temperature for \(H//c\) and vice versa for \(H//ab\). Both curves are nearly the same, suggesting an isotropic \(H_c2\) in (Ba\(_{1-x}\)K\(_x\))Fe\(_2\)As\(_2\) which is a unique property for a layered superconductor. The \(H_c2\) along the \(ab\) and \(c\) planes for the electron-doped compound Ba(Fe\(_{1-x}\)Co\(_x\))\(_2\)As\(_2\) follows same trend as the hole-doped (Ba\(_{1-x}\)K\(_x\))Fe\(_2\)As\(_2\), and an almost isotropic \(H_c2\) is found in the limit of zero temperature [67, 68]. Among the FeSCs, LiFeAs shows a relatively low value of \(\mu_0H_c2(0)\), which has been reported to be 15 T for \(H//c\) and 26 T for \(H//ab\) [66]. In spite of their low \(T_c\) (~14 K), 11 compounds such as Fe\(_{1.11}\)Te\(_{0.6}\)Se\(_{0.4}\) shows a high \(H_c2\) of 45 T at zero temperature [65]. Furthermore, \(H_c2\) values above 100 T and \(H_{irr}\) (9 K) ~ 6 T have also been reported for FeSe\(_{0.5}\)Te\(_{0.5}\) [69].
2.3.2.3 Critical current density \( (J_c) \)

High values and weak field dependence of \( J_c \) are desirable for potential applications. \( J_c \) values of \( \sim \)10\(^6\) to \( 10^7 \) A/cm\(^2\) have been found in FeSCs, which are almost field independent at low temperature in many cases. Generally, \( J_c \) can be obtained for a rectangular shaped crystal with dimensions \( c < a < b \) and \( B\parallel c \) by using Bean’s model, which is as follows;

\[
J_c = 20\Delta M/ [a (1-a/3b)] \quad \ldots \ldots (2-1)
\]

where \( a \) and \( b \) are the sample dimensions perpendicular to the applied field and \( \Delta M \) is the height of the magnetization loop. Flux jumping has been observed in different superconductors. Our team reported flux jumping for the first time in FeSCs, i.e. \((\text{Ba}_{1-x}\text{K}_x)\text{Fe}_2\text{As}_2\). Generally, flux jumps can be found for large-size samples with high \( J_c \) values, small specific heat, and a sufficiently fast ramp rate of the magnetic field [70]. Flux jumping has also been reported for LiFeAs [71]. It is important to mention here that 111 type compounds have not been widely investigated in terms of \( J_c \).

A. \( J_c \) in Single Crystals

High \( J_c \) values have been reported in different single crystals, as can be seen in Figure 2-10. An in-plane \( J_c \) (almost field independent up to 7 T at 5 K) of \( 10^6 \) A/cm\(^2\) at 5 K for a SmFeAsO\(_{1-x}\)F\(_x\) crystal (1111 family) has been reported [72, 73]. 122 compounds show isotropic and high \( J_c \) in crystals. Significant fishtail peak effects and high current carrying capability up to \( 5 \times 10^6 \) A/cm\(^2\) at 4.2 K have been reported for K-doped \( \text{Ba}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2 \) single crystal [74]. The fishtail effect at low temperature has also been reported for the other 122 compounds [75, 76]. As for the 11 compounds, \( J_c \) of \( \text{FeTe}_{0.61}\text{Se}_{0.39} \) crystals was found to be well above \( 10^5 \) A/cm\(^2\) at
low temperatures. $J_c$ of nearly $10^5$ A/cm$^2$ at 5 K (field independent up to 6 T) has also been found in 111 compounds [77]

![Figure 2-10: Field-dependent $J_c$ values for 1111, 122, 111 and 11 crystals [212-217].](image)

**B. $J_c$ in Thin Films**

Thin films have not been easy to fabricate, especially in the case of the 1111 FeSCs where F and O are main dopants, as both are volatile and effectively uncontrolled in the final films [78]. Because they are free from grain boundaries and strong pinning centres, such as inclusions, surface roughness, and defects related to the growth mode, high values of $J_c$ have been found in thin films. Lida et al. reported high $J_c$ values of nearly $10^6$ A/cm$^2$ at 45 T and 4.2 K for both main field orientations, a feature that is favourable for high-field magnet applications [79]. $J_c > 10^6$ A/cm$^2$ at 4.2 K has also been reported for 122 compounds [80]. High $J_c$ values of up to 4 MA/cm$^2$ at 4 K have been also found in Co-doped BaFe$_2$As$_2$ epitaxial films grown...
directly on (La,Sr)(Al,Ta)O$_3$ substrates by pulsed laser deposition [37]. High $J_c$ values are also reported for BaFe$_2$(As$_{0.66}$P$_{0.33}$)$_2$ films with uniformly dispersed BaZrO$_3$ nanoparticles [81]. $J_c$ of about $10^5$ A/cm$^2$ was found for FeTe$_{0.5}$Se$_{0.5}$ films at 4.2 K in self-field, and the same value is retained up to 8 T [82]. In general, the $J_c$ values in thin films are higher than those of single crystals.

**C. $J_c$ in wires/tapes**

Superconductors can be used in applications in the form of wires/tapes. Ma’s group fabricated LaFeAsO$_{1-x}$F$_x$, SmFeAsO$_{1-x}$F$_x$ and Sr$_{1-x}$K$_x$Fe$_2$As$_2$ wires by using the powder-in-tube (PIT) method [83-85]. For 1111 compounds, transport $J_c$ of $10^4$A/cm$^2$ in self-field at 4.2 K has been reported in Sn-added ex-situ SmFeAsO$_{1-x}$F$_x$ tapes [86]. Usually, 122 compounds reveal high values of $J_c$ [87]. At 4.2 K, transport $J_c$ of nearly 4000 A/cm$^2$ in self-field was found for an ex-situ PIT processed Sr$_{1-x}$K$_x$Fe$_2$As$_2$/Ag wire with an Fe/Ag double sheath. The transport $J_c$ of Ba$_{1-x}$K$_x$Fe$_2$As$_2$/Ag tapes is greatly improved by a combined process of cold flat rolling and uniaxial pressing. At 4.2 K, $J_c$ exceeds the practical level of $10^5$ A/cm$^2$ in magnetic fields up to 6 T and maintains a high value of $8.6 \times 10^4$ A/cm$^2$ in 10 T [88]. Ding et al., have fabricated (Ba, K)Fe$_2$As$_2$ superconducting wires through an ex-situ powder-in-tube method. The transport $J_c$ reached $1.3 \times 10^4$ A/cm$^2$ and $10^4$ A/cm$^2$ at 4.2 K under self-field in the wires with and without Ag addition [89]. Togano et al. have reported large transport $J_c$ in Ag-added (Ba,K)Fe$_2$As$_2$ superconducting wires of $10^4$ A/cm$^2$ at 0 T and nearly $10^3$ A/cm$^2$ at 4.2 K [90]. A transport $J_c$ of $8.5 \times 10^3$ A/cm$^2$ at 4.2 K, 10 T was also found for Cu/Ag-cladded Ba$_{1-x}$K$_x$Fe$_2$As$_2$ wires [91]. Recently, a record high transport $J_c$ of up to 0.1 MA/cm$^2$ at 10 T and 4.2 K has been found in Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ tapes, as can be seen in Figure 2-11. The $J_c$ exceeded 1 MA/cm$^2$ in Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ tapes at 4.2 K [91].
D. $J_c$ in Polycrystalline bulks

$J_c$ values in polycrystalline bulks have been found to be lower due to weakly linked grains.

For 1111 compound, Wang et al. reported $J_c \approx 10^4$ A/cm$^2$ at 5 K in self-field [92]. Similar results have been found by Ding et al. [93]. At 5 K, $J_c$ values of around $10^4$ A/cm$^2$ in self-field has been obtained in K doped 122 compounds [94]. $J_c$ for FeTe$_{1-x}$Se$_x$ polycrystals, as estimated from $M$–$H$ curves, is around $10^4$ A/cm$^2$ at 5 K under zero field [95]. $J_c$ values for different polycrystalline bulks can be seen in Figure 2-12.

Figure 2-12: $J_c$ values for different polycrystalline bulks [32-35].
Low values of $J_c$ are due to weak flux pinning. Koblischka et al. have compiled the results on the pinning mechanisms for FeSCs in details [96]. The $\delta T_c$-pinning is mostly dominant in the 122- and 1111-families, whereas, the 11 and 111 compounds shows $\delta \ell$ pinning. Pinning force analysis for FeSCs was conducted by using the scaling approaches of Dew-Hughes and Kramer [23, 97]. The resulting peak positions, $h$, were found at ~ 0.3 for the 11-type materials, at ~ 0.48 for the 111-type materials, between 0.32 and 0.5 for the 1111-type materials, and between 0.25 and 0.71 for the 122-type materials. Details can be found elsewhere [236].

2.3.3 Effects of chemical doping on SC Properties of FeSCs

Chemical doping is also a useful technique for FeSCs for enhancing their superconducting properties. The parent compounds of some of the FeSCs such as SmFeAsO are not superconducting. Superconductivity appears when the AFM order is suppressed. With Co-doping in the FeAs planes, the AFM order is destroyed, and superconductivity occurs at 15.2 K [98]. Fluorine doped NdFeAsO shows superior $J_c$-field Performance of $10^6$ A/cm$^2$ at 5 K [99]. High $J_c$ values (i.e. > $10^5$ A/cm$^2$) at 45 T and 4.2 K have been obtained for epitaxial SmFeAs(O,F) thin films in both field orientations [79]. Similar results are also reported for SmFeAs(O,F) films [100]. Fluorine doped SmFeAsO single crystals showed a high and isotropic $J_c$ (> $2 \times 10^6$ A/cm$^2$) along all crystal directions [73].

Ag and Pb have been used as dopants in the development of 122 pnictide wires/tapes in order to enhance $J_c$ by improving the intergranular coupling of the superconducting grains [85]. Chemical doping with Ag of polycrystalline Sr-122 superconductor can improve grain connectivity, as it can effectively suppress the formation of the glassy phase as well as the amorphous layer. In addition, improved
$J_c$ values have also been found in Ag doped 122 wires [101]. Similarly, chemical doping with Pb can improve the grain connectivity as well [102]. It should be noted that Pb addition can predominantly increase low field $J_c$, while Ag addition can enhance the high field $J_c$ in 122 wires/tapes. Some groups have used both Ag and Pb as a dopants and found a slightly better result in the determination of transport $J_c$ in K doped 122 pnictide tapes, although weakly-linked grain boundaries still existed [103]. Although $J_c$ is improved to some extent by these approaches, the major drawback is degradation of $T_c$ in many cases. The $J_c$ found at 2 K and 0 T reaches $4.7 \times 10^6$ A/cm$^2$, and $J_c$ at 5 K remains more than $1 \times 10^6$ A/cm$^2$ at 9 T for potassium doped 122 compounds [74]. Over-doping with K has a negative effect on the $T_c$ [94]. High $J_c$ of 4 MA/cm$^2$ at 4 K was also obtained in Co-doped BaFe$_2$As$_2$ [104]. Similar results are also found elsewhere [80]. Miura et al. introduced controlled amounts of uniformly dispersed BaZrO$_3$ nanoparticles into carrier doped BaFe$_2$As$_2$, which can improves its $J_c$ values and only has a slight effect on $T_c$ [81]. Cu and Co substitution in LiFeAs has resulted in suppression of the $T_c$ value [105]. $T_c$ suppression due to doping concentrations can be seen in Figure 2-13.

![Figure 2-13: $T_c$ as a function of doping concentrations [105]](image)
Si et al., have found $J_c \sim 10^6 \text{ A/cm}^2$ in tellurium doped FeSe films [106]. $T_c$ values of sulphur doped FeSe are significantly suppressed by high doping levels, as can be seen in Figure 2-14 [107].

![Figure 2-14: $T_c$ as a function of doping concentrations for sulphur doped FeSe [107]](image)

### 2.3.4 Effects of irradiation on SC Properties of FeSCs

Although the irradiation technique can enhance $J_c$ in FeSCs, significant degradation of $T_c$ is a major disadvantage. Furthermore, it is unsuitable for large-scale applications. There have been numerous reports on how irradiation can cause $J_c$ enhancement, especially for 122 compounds, as they are more convincing in terms of their properties.

For 1111 compounds, Moore et al. studied the effects of heavy ion irradiation on the $J_c$ and the flux dynamics of polycrystalline NdFeAsO$_{0.85}$ irradiated with 2 GeV Ta ions. A little increase in $J_c$ of up to $\sim 10^5 \text{ A/cm}^2$ (factor of 3 to 4) at 10 K has been observed through the formation of continuous collinear columnar defects [108]. Recently Fang et al. planted a low density of correlated nanoscale defects into SmFeAsO$_{0.85}$F$_{0.15}$ by heavy-ion irradiation, resulting in an increase in $J_c$ values up to

42
greater than $10^7$ A/cm² at 5 K [109]. Eisterer et al. obtained enhanced $J_c$ values, together with $T_c$ deterioration, by irradiating SmFeAsO$_{1-x}$F$_x$ in a fission reactor with a fast ($E > 0.1$ MeV) neutron fluence of $4 \times 10^{21}$ m$^{-2}$ (Figure 2-15) [110]. Reduction in $T_c$ was also observed for a neutron irradiated LaO$_{0.9}$F$_{0.1}$FeAs polycrystalline sample [111].

Figure 2-15: Comparison of $J_c$ for un-irradiated and irradiated SmFeAsO$_{1-x}$F$_x$ [110].

High intrinsic pinning strength has been reported in K doped 122 single crystals due to small anisotropy (i.e. $\gamma \approx 1$-3) [64, 70]. $J_c$ values can be enhanced by irradiation through improvement of flux pinning. For example, light ion C$^{4+}$ irradiation can increase $J_c$ in BaFe$_{1.9}$Ni$_{0.1}$As$_2$ single crystal from $0.61 \times 10^5$ up to $0.94 \times 10^5$ A/cm² at $T = 10$ K and $H = 0.5$ T, but along with reduction in the $T_c$ value [112]. Nakajima et al. have introduced columnar defects into Co-doped BaFe$_2$As$_2$ single crystals by heavy ion irradiation, resulting in enhancement of $J_c$ values from $6.4 \times 10^5$ A/cm² to $4.0 \times 10^6$ A/cm² at $T = 5$ K and 0 T, as can be seen in Figure 2-16 [113]. Similarly, Kim et al. investigated the effects of heavy ion irradiation on Co and Ni doped Ba-122 compounds [114]. High $J_c$ values of nearly $1.0 \times 10^7$ A/cm² at 5
K under self-field in (Ba,K)Fe$_2$As$_2$ single crystals after irradiating with Au ions were also reported [115].

Neutron irradiation can lead to a decrease in $T_c$ and increase in $J_c$ values by a factor of 3 in Co doped Ba-122 FeSCs [116]. Karkin et al. investigated the effects of neutron irradiation on the properties of FeSe compound and also found a reduction in $T_c$ values [117]. Haberkorn et al. studied the influence of random point defects planted by proton irradiation into Co-doped BaFe$_2$As$_2$ crystal and found little enhancement in $J_c$ [118].

2.3.5 Effects of pressure on SC Properties of FeSCs

Hydrostatic pressure effects have been mostly studied with respect to $T_c$. Pressure has been revealed to have a positive effect on $T_c$ in FeSCs. Generally, $T_c$ versus pressure shows a dome-like behaviour: it increases with pressure, reaches a certain high value,
and then decreases with the application of further pressure applied, in a way which mostly depends on the doping concentration. The pressure effects (less than 5 GPa) on $T_c$ in LaFeAsO$_{1-x}$F$_x$ are positive for under-doped, optimally doped, and over-doped samples, as can be seen in Figure 2-17 [119]. The optimally and over-doped samples show nearly the same response to pressure, and $T_c$ reaches up to 43 K at 4 GPa, but decreases with increasing pressure. Low $T_c$ values were found in the under-doped sample.

![Figure 2-17: Pressure dependence of $T_c$ for under-doped, optimally-doped and over-doped LaFeAsO$_{1-x}$F$_x$ [119].](image)

Okada et al. measured $T_c$ under pressure for LaNiPO and LaNiAsO, and found that $T_c$ increased up to 3.78 K at 1.67 GPa and 3.14 K at 0.75 GPa, respectively [120]. Takeshita et al. reported the negative effect of pressure on different compositions of NdFeAsO$_{1-y}$ [121]. Similar results are also found elsewhere [122]. Lorenz et al. found that $T_c$ improves with pressure for un-doped material and is suppressed with pressure for an over-doped compound in the case of SmFeAsO$_{1-x}$F$_x$ [123]. Selvan et al. measured a GdFe$_{0.9}$Co$_{0.1}$AsO sample under pressure and found that $T_c$ decreased
from 16.7 to 10.5 K at 2.9 GPa [124]. Pressure induces superconductivity in BaFe$_2$As$_2$. For example, Alireza et al. found superconductivity at 29 K, 4.5 GPa [47]. Kimber et al. found their maximum $T_c$ in BaFe$_2$As$_2$ under pressure, where the FeAs$_4$ tetrahedra are regular, with an angle of 109.47° [125]. Igawa et al., measured $T_c$ as a function of pressure for SrFe$_2$As$_2$ with different set-ups (Figure 2-18) and found almost the same response to pressure [126].

![Figure 2-18: Pressure dependence of $T_c$ values for SrFe$_2$As$_2$ [126].](image)

Gooch et al. found a positive pressure effect on $T_c$ for under-doped K$_{1-x}$Sr$_x$Fe$_2$As$_2$ and a negative effect for their over-doped sample, whereas the $T_c$ response to pressure for the optimally doped sample was not significant [127]. Uhoya et al. found a rapid increase of $T_c$ up to 41 K at 10 GPa for EuFe$_2$As$_2$ [128]. Pressure of 0.69 GPa also induced superconductivity in CaFe$_2$As$_2$ at $T_c \approx 11$ K [129]. Park et al. investigated the pressure effect for Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ thin films and found that $T_c$ of
the thin film with optimal potassium concentration gradually increased to 40.8 K at 1.18 GPa [130].

The 111 compounds have not been widely investigated under pressure. $T_c$ was reduced linearly with a pressure coefficient of 1.5 K/GPa for LiFeAs [131], similar to LiFeP [132]. Zhang et al. reported that the $T_c$ increased from 26 K to a maximum of 31 K as the pressure increased from ambient pressure to 3 GPa for Na$_{1-x}$FeAs [133]. Wang et al. measured Co doped NaFeAs under pressure and found that the maximum $T_c$ enhanced by pressure in both under-doped and optimally-doped NaFe$_{1-x}$Co$_x$As is almost 31 K and 13 K with pressure of 2.3 GPa [134].

So far, a huge enhancement in $T_c$ by pressure has been obtained in 11 compounds. Margadonna et al. (2009b) found that the hydrostatic pressure can raise $T_c$ up to 37 K at 7 GPa from nearly 14 K [135]. $T_c$ was also found enhanced for tellurium doped FeSe [136].

Little work has been done on how pressure affects $J_c$ in FeSCs. Pietosa et al. found enhancement in $J_c$ of one order of magnitude in 11 compounds [137]. The pinning mechanism under pressure has not been investigated at all. Therefore, detailed investigations are required in this regard.

### 2.3.6 Conclusion

The combination of reasonable values of $T_c$, extremely high $H_{c2}$ on the order of 100 T, high intrinsic pinning potential, and low anisotropy (generally between 1-8) makes FeSCs worthy of investigation for applications, whereas the $J_c$ is a major limiting factor [73, 81, 91, 99, 104, 106, 109, 138-144]. Improvement in $J_c$ by using various methods has also been one of the most important topics in the field of superconductivity. Although $J_c$ can be improved by texturing procedures, ion
implantation/irradiation, and chemical doping, the major drawbacks are i) $J_c$ decays rapidly in high fields at high temperatures, or low field $J_c$ deteriorates in some cases; ii) these methods can cause degradation of $T_c$. Taking into account the positive effects of pressure, we anticipated that pressure could be used to enhance $J_c$ in FeSCs. Our detailed investigations are reported in Chapters 4-6.
2.4 MgB₂

The superconductivity of MgB₂ ($T_c \approx 39$ K) was discovered by Akimitsu in 2001 [13], although this compound was first fabricated in 1953 [145]. Generally, MgB₂ is categorized as a conventional superconductor with a unique electronic structure. MgB₂ has a low anisotropy, in contrast to the cuprates, strongly linked grains, high coherence length values, low fabrication costs, a multiple band structure, and has especially high critical current density ($J_c$) values of $10^5$-$10^6$ A/cm², which makes it a significant superconductor for applications [146-153]. Furthermore, MgB₂ can easily be drawn into wires and tapes, further supporting its potential to replace NbTi and NbSn₃ in industry.

2.4.1 Crystal structure and two-gap conductivity

MgB₂ is a simple ionic binary material possessing a simple hexagonal AlB₂-type structure (space group P6/mmm). Figure 2.19 shows the MgB₂ crystal structure [154]. It contains graphite-type boron layers, which are sandwiched between hexagonal close-packed layers of magnesium. The lattice parameters obtained for MgB₂ are $a = 3.09$ Å (equal to the in-plane Mg-Mg distance), and $c = 3.52$ Å (the distance between Mg-layers). Kortus et al. found that Mg $s$ states are pushed up by the B $p_z$ orbitals and completely donate their electrons to the boron-derived conduction bands. The electrons donated to the system are distributed over the whole crystal [155].

MgB₂ has a strong anisotropy in the B-B distances: the distance between the boron planes is larger than the in-plane B-B distance [156, 157]. There is no structural transition found for MgB₂ down to 2 K, even under high pressure of 40 GPa [154].
Numerous experiments based on heat capacity, Raman scattering, point contacts, and optical and magnetic properties measurements of polycrystalline samples or single crystals have revealed that MgB$_2$ is a multi-gap superconductor [158-165]. The $sp^3$ boron orbitals overlap, forming $\sigma$-bonds between the neighbouring atoms in the plane, whereas the rest of the $p$ orbitals spread above and below the plane and produce $\pi$- bonds in MgB$_2$. The electronic structure of MgB$_2$ contains two types of electrons at the Fermi level with different behaviours: the sigma-bonding is stronger than the pi-bonding. Figure 2-20 illustrates the Fermi surface of MgB$_2$. $\Gamma$, L, M, and A are sites in the Brillouin zone. The $\sigma$-bands form two hole-like coaxial cylinders along the $\Gamma$-A line, and the $\pi$-bands form two hole-like tubular networks near K and M, and an electron-like tubular network near H and L [155]. The anomaly of the charge distribution in the $\sigma$-bonding with respect to the in-plane boron atoms provides strong coupling of the $\sigma$-bonding state to the in-plane vibration of boron atoms, which is responsible for the superconductivity in MgB$_2$ [166]. Electrons at these different Fermi levels join into pairs with different bonding energies. Each $\pi$-bond and $\sigma$-bond has certain energy gaps at the Fermi surface. The average values of the gaps are 6.8 meV for $\sigma$-bonds and 1.8 meV for $\pi$-bonds.
2.4.2 **Superconducting Properties of MgB$_2$**

The magnetic and transport measurements show that MgB$_2$ is free of weakly linked grain boundaries, which is in contrast to high temperature superconductors (HTSCs), providing nearly the same $J_c$ values in high magnetic fields for polycrystalline bulk samples [167]. Therefore, MgB$_2$ can be used in form of wires/tapes for practical application with no degradation of $J_c$ values. $J_c$ values can also be enhanced by improving the flux pinning properties.

2.4.2.1 **Critical temperature ($T_c$)**

The $T_c$ of MgB$_2$ is found to be almost 39 K (-234 °C), which is the highest amongst the conventional superconductors [13]. It is widely believed that the $T_c$ of MgB$_2$ is
crystal structure dependent. Hydrostatic pressure, neutron irradiation, and chemical doping can change the lattice volume or structure, affecting $T_c$ values [154].

2.4.2.2 Critical fields and $H_{irr}$

MgB$_2$ behaves like a type-II superconductor under magnetic field. Being a layered structure, MgB$_2$ is an anisotropic material. The variations in the critical field values can be observed from the planes parallel ($H_{c1(c2)} \parallel ab$) to and perpendicular ($H_{c1(c2)} \perp ab$) to the $ab$-plane [168]. The anisotropy ratio $\gamma = H_{c2} \parallel ab / H_{c2} \perp ab$, is found between 1.1 and 1.7 for textured bulks and partially oriented crystallites, 1.2–2 for $c$-axis textured material, 1.7-2.7 for single crystals, and around 2 for thin films [169-177].

At 5 K, $H_{c1}$ is 0.25 T and 0.125 T parallel to the $ab$-plane and $c$-axis, respectively, and $H_{c2} \parallel ab \approx 18$ T, $H_{c2} \parallel c \approx 3.5$ T are found for single crystal [178]. For polycrystalline materials, $H_{c1}$ is found to be 425 Oe at 5 K [179]. An $H_{c2}$ value of 16 T at 0 K has been reported for polycrystalline MgB$_2$, and it is up to 74 T in thin films and up to 55 T in fibres [180-184]. The $H_{c2}$ values for MgB$_2$ can be improved by tuning of ratio of intraband to interband scattering rates through suitable doping on both Mg and B sites, which can also reduce the anisotropy of the critical fields. Furthermore, the values of $H_{irr}$ at 10 K range between 5 and 20 T for MgB$_2$ bulks, films, wires, tapes, and powders [154, 185]. Note that $H_{c2}$ in MgB$_2$ is almost two times greater than $H_{irr}$ [186].

2.4.2.3 Critical current density ($J_c$)

$J_c$ is a key parameter in superconducting applications. The $J_c$ in MgB$_2$ is mostly determined by its flux pinning, as it is free of weak links. The flux pinning is field dependent, especially in high fields, providing a quick drop of $J_c$ with increasing field. In polycrystalline MgB$_2$, $J_c \approx 10^6$ A/cm$^2$ at 0 T, $J_c \approx 10^4$ A/cm$^2$ at 6 T, and $J_c \approx$
$10^2$ A/cm$^2$ at 10 T have been reported by different groups [154]. Due to weak pinning, $J_c$ in single crystal is nearly $10^5$ A/cm$^2$ at low temperatures in self-field and drops quickly at high fields [184, 187]. $J_c > 10^7$ A/cm$^2$ has been reported in thin films, which are higher than for single crystals under self-field and low temperatures due to strong pinning at the grain boundaries [184].

At 4.2 K, the transport $J_c$ obtained for wire is above $10^6$ A/cm$^2$ in self-field and around $10^4$ A/cm$^2$ at 8 T [186]. Another study shows that the $J_c$ values at low fields and temperatures in MgB$_2$ wires are on the order of $10^5$ A/cm$^2$, but drops to the order of $10^3$ A/cm$^2$ at around 10 T [188-190]. In contrast, MgB$_2$ tapes show better results for $J_c$ at relatively high magnetic fields because of their geometrical shielding properties. At 4.2 K, H Yamada et al. found $J_c$ to be $\sim$32000 A/cm$^2$ and 14000 A/cm$^2$ for SiC doped MgB$_2$ in ethyltoluene at 10 T and 12 T, respectively [191]. Furthermore, $J_c$ values for MgB$_2$ tapes on the order of $10^4$ A/cm$^2$ have been found at 4.2 K and 10 T [192-196]. It was reported that factors such as porosity of the sample, grain boundaries, impurities, homogeneity, and fabrication and sintering conditions strongly affect $J_c$ values in MgB$_2$ [184].

**2.4.3 Effects of Chemical Doping on Superconducting (SC) Properties of MgB$_2$**

Chemical doping can enhance $H_{c2}$, $H_{irr}$, and $J_c$ by improving the flux pinning mechanism in MgB$_2$. Usually, significant enhancement in $J_c$ has been found with carbon source doping [146, 180, 197-200], but it comes along with degradation of $T_c$. Different dopants have been used to enhance $J_c$ values. Effects of dopants on superconducting properties, especially $J_c$ and $T_c$, which have similar properties, can be explained as follows:

$T_c$ values have been reduced by chemical doping with alkali metals such as Na and Li [201], and Cs and Rb [202], alkaline earth metals such as Ba [203], poor metals
such as Al [204, 205] and Zn [206], and transition metals such as Ir [207] and Ni [208], although a slight enhancement in $J_c$ was found in some of these cases. Doping of Al into Mg sites caused significant degradation of $T_c$ with increasing Al content resulting in the loss of superconductivity in MgB$_2$ [209].

$J_c$ at high fields in MgB$_2$ can be improved by doping with silicides such as elementary Si [210], ZrSi$_2$ [194], WSi$_2$ [211], Si$_3$Ni$_4$ [212], and SiO$_2$ [193], although reduction of $T_c$ is a major drawback.

Carbon-containing composites or compounds, such as nano-C, B$_4$C, carbon nanotubes (CNT), hydrocarbons, carbohydrates, and SiC, have pronounced effects on $J_c$, although $T_c$ values are greatly degraded in a few cases [213-216]. The $J_c$ in magnetic field increases significantly, but $T_c$ decreases much more slowly with carbon doping for MgB$_2$ films (Figure 2-21) [217]. Nano-C doping can also enhance $J_c$ in MgB$_2$ tapes. Xianping Zhang et al. found that $J_c$ reached $2.2 \times 10^4$ A/cm$^2$ in their 8% C doped samples at 4.2 K, 10 T [218]. Dou et al. have studied the effect of carbon nanotube (CNT) doping of MgB$_2$ on $T_c$ and $J_c$. The $J_c$ was found to be more than 10000 A/cm$^2$ at 20 K and 4 T, and 5 K and 8.5 T, respectively [219]. Similar results are also reported by other groups [220, 221].

![Figure 2-21: (Left) $T_c$ dependence on carbon concentration. (Right) Field dependence of $J_c$ at different temperatures for undoped and carbon doped samples [217].](image)
Graphite, diamond, and carbide compounds improve the $J_c$ in high fields by improving flux pinning, but with some effect on $T_c$ [222-224]. Silver doping can also degrade $T_c$ values [225].

$J_c$ in MgB$_2$ at high field can also be improved by doping with rare earth elements/oxides. For instance, elemental La doped MgB$_2$ can react with B and forms LaB$_6$ nanoparticle inclusions, which can serve as pinning centres, leading to high $J_c$ values [226, 227]. Addition of Y$_2$O$_3$ nanoparticles can give $J_c \approx 2 \times 10^5$ A/cm$^2$ at 2 T and 4.2 K, but $J_c$ drops to $\sim 8 \times 10^4$ A/cm$^2$ at 20 K. $H_{irr}$ was also reported, i.e. 11.5 T at 4.2 K and 5.5 T at 20 K [228]. Similarly, Dy$_2$O$_3$ doped MgB$_2$ can show improved flux pinning, hence $J_c$ [229]. Doping with Ho$_2$O$_3$ can form pinning centres in the form of HoB$_4$ impurities, which can improves high field $J_c$ and $H_{irr}$, with $T_c$ and $H_{c2}$ unchanged [230]. Similar results were also obtained for transition metals, such as Wb [231], Zr [232, 233], Cu [234], Ti [233, 235], and Pb [236]. Doping with poor metals, such as in Cd doped MgB$_2$, generates MgCd$_3$ impurities, which serve as effective pinning centres, resulting in the enhancement of $J_c$ values, i.e. $5.0 \times 10^5$ A/cm$^2$ (5 K, 0 T) with only a little drop in $T_c$ [237]. Magnetic elements, such as Mn [238] Co and Fe [239, 240], and Ni [208], mostly resulted in suppression of $J_c$. N Novosel et al. investigated MgB$_2$ doped with dextrin-coated magnetite nanospheres and nanorods, and found that nanospheres can enhance $H_{irr}$ and the transport $J_c$ at lower temperatures due to generation of magnetic pinning of vortices. Doping MgB$_2$ with different oxides generates different results. Doping with Al$_2$O$_3$ can decrease the $J_c$ and $H_{irr}$ with increasing Al$_2$O$_3$ level [241]. In contrast, $J_c$ is found to be increased with Co$_2$O$_3$ doping [242]. Reduction and enhancements of $J_c$ have also been reported for ZrO$_2$ [243] and TiO$_2$ doping [244], respectively. Doping with boride compounds, such as ZrB$_2$ and TiB$_2$, also led to the improvement of $J_c$ [245,
Furthermore, doping with uranium had no significant effect on $J_c$ in MgB$_2$ [247].

Chemical doping can transform the nature of the pinning mechanism in MgB$_2$. The $\delta T_c$ pinning mechanism is dominant in pure MgB$_2$ polycrystalline bulks, thin films, and single crystals [22, 248, 249]. As one example, S R Ghorbani et al. investigated the flux pinning mechanisms within the collective pinning model in nano-Si-doped MgB$_2$ and found the coexistence of both $\delta \ell$ pinning and $\delta T_c$ pinning in their nano-Si-doped MgB$_2$ samples [250]. Similar results have been found for nano-carbon doped MgB$_2$ and succinic acid doped MgB$_2$ [251, 252]. Doping with SiC can transform the pinning mechanism from $\delta T_c$ to $\delta \ell$ [253], and partial point pinning can be generated in addition to surface pinning [254, 255]. J. L. Wang et al. investigated the pinning mechanism in carbon doped MgB$_2$. Doping with C and graphene oxide can transform the pinning mechanism from $\delta T_c$ to $\delta \ell$, and carbon doping can also induce point pinning in MgB$_2$ (Figure 2-22) [256, 257].

![Figure 2-22: Transformation of pinning mechanism from $\delta T_c$ to $\delta \ell$ by graphene oxide doping [257].](image)
In summary, chemical doping can improve flux pinning and hence $J_c$, although a major drawback is the reduction of $T_c$. Therefore, we need an effective approach which can enhance $J_c$ and flux pinning, but without sacrificing $T_c$ values.

2.4.4 Effects of Irradiation on SC Properties of MgB$_2$

Irradiation is another technique that is used to enhance $J_c$ and flux pinning in superconductors. Soon after the discovery of superconductivity in MgB$_2$, several experiments were performed to enhance these parameters by artificial damage. Although irradiation can decrease the $T_c$ and low field $J_c$ in a few cases, $J_c$ in high fields can be improved.

Initial experiments with proton irradiation showed that atomic disorder created by proton irradiation can improve the flux pinning, thereby increasing $J_c$ values in high fields besides degrading $T_c$ [258]. Similar experiments have also been performed on single crystalline and bulk samples [259].

![Figure 2-23: Reduction of $T_c$ by irradiation in MgB$_2$ [258]. d.p.a. is displacements per atom.](image)

High-energy heavy ion irradiation can modify the nature of the pinning mechanism in MgB$_2$ thin films, resulting in enhancement of $J_c$, although degradation of $T_c$ has been a drawback [260, 261]. Considerable reduction of $T_c$ to 7 K has been reported in
MgB$_2$ thin films due to irradiation by alpha particles [262]. Similar results were also reported for Au ion irradiation of MgB$_2$ thin films [263]. In addition, Pb-ion irradiation can increase the $J_c$ in high fields [264].

Karkin et al. were the first to investigate the effects of neutron irradiation on the $T_c$ and the $H_{c2}$ of polycrystalline MgB$_2$ samples. The $T_c$ decreased significantly from 38 to 5 K as a result of irradiation, which is ascribed to a decrease in the density of electronic states at the Fermi level. [265]. Eisterer et al. and Wang et al. also found a reduction of $T_c$ in MgB$_2$ due to neutron irradiation [266, 267]. Enhancement in $J_c$ by neutron irradiation has been reported besides $T_c$ suppression. For instance, Zehetmayer et al. found enhancement of $J_c$ in low fields, by a factor of 5 at 5 K, along with $H_{c2}$ and $H_{irr}$ [268]. Similarly, Putti et al. also found enhancement in $J_c$; significantly, at high fields, $H_{c2}$ increased from 13.5 T to 20.3 T at 12 K, and $H_{irr}$ doubled at 5 K [269]. Tarantini et al. reported a significant improvement in the field dependence of $J_c$, reaching a value close to $10^4$ A/cm$^2$ at 10 T, along with degradation of $T_c$ by few Kelvins [270]. $T_c$ suppression under neutron irradiation can be seen in Figure 2-24. Pallecchi et al. found that there was coexistence of grain boundary and point pinning in neutron irradiated MgB$_2$ samples [271].

![Figure 2-24: $T_c$ suppression under neutron irradiation [272].](image)
Talapatra et al. also investigated the effects of 160 MeV Ne$^{16}$ ion irradiation on $J_c$, $H_{c2}$, $H_{irr}$, and flux pinning in polycrystalline MgB$_2$ [273]. MgB$_2$ polycrystalline bulks were also irradiated with gamma doses, resulting in enhancement of $J_c$ values [274].

In summary, the irradiation technique can enhance $J_c$, especially in high fields, and affect the pinning mechanism. Reduction in $T_c$ and low field $J_c$ (in some cases) are major drawbacks of this technique, however, like chemical doping. Furthermore, this technique is not significant for high field applications.

2.4.5 Effects of Pressure on SC Properties of MgB$_2$

The impact of hydrostatic pressure on the nature of the pinning mechanism and $J_c$ has not been investigated as yet. Pressure studies in the field of superconductivity generally emphasize the $T_c$ dependence, and the same is the case in MgB$_2$ investigations. Pressure studies allow us to determine the pressure dependence of $T_c$ and develop a framework in theoretical models to provide information on the pairing mechanism. A large value of $dT_c/dP$ in a material shows that the crystal structure may be tuned by applying external pressure, resulted in high values of $T_c$. This can also suggest that $T_c$ may be further improved by providing internal pressure (i.e. chemical doping). Although a positive pressure effect on $T_c$ has been predicted through reduction of the interatomic B–B distance, it is well established now that pressure can decrease the $T_c$ of MgB$_2$, as evidenced by various experimental studies [275]. The pressure dependence of $T_c$ for MgB$_2$ was first measured in a piston-cylinder clamp with a 3M Fluorinert FC 77 liquid pressure transmitting medium by Lorenz et al., as can be seen in Figure 2-13 [276].

Buzea et al. has systemically studied the pressure effect on $T_c$ [154]. Different pressure media have been used to obtain $T_c$ values. Negative pressure dependence
has been obtained, however, with different pressure coefficients [276-279]. The observed negative pressure dependence of $T_c(P)$ can be readily described in terms of simple lattice stiffening within standard phonon-mediated BCS superconductivity [280].

The pressure dependence of $T_c$ follows linear/quadratic behaviour, decreasing monotonically. Buzea et al. reported that samples with higher $T_c$ at zero pressure have a much lower $T_c(P)$ dependence ($dT_c/dp \approx -0.2 \text{K/GPa}$) than the samples with lower $T_c$ ($dT_c/dp \approx -2\text{K/GPa}$) [154]. Furthermore, samples with higher $T_c$ have a convex curvature of $T_c(P)$ dependence, changing to concave for samples with lower $T_c$ as can be seen in Figure 2-26.

Figure 2-25: Pressure dependence of $T_c$ for MgB$_2$ [276].

Figure 2-26: $T_c$ as a function of pressure through different pressure media as compiled by Buzea et al. [154] and the references therein.
Furthermore, the measurement of $H_{c2}$ under pressure was also carried out [281]. Moreover, the anisotropy of MgB$_2$ is increased under pressure, as studied by Schneider et al. [282].

2.4.6 Conclusion

MgB$_2$ is a favourable superconducting material which might replace conventional low $T_c$ superconductors in practical applications, due to its relatively high $T_c$ of 39 K, strongly linked grains, rich multiple band structure, low fabrication cost, and especially, its $J_c$ values of $10^5$-$10^6$ A/cm$^2$. Chemical doping and irradiation techniques can enhance $J_c$ either in low or high fields, but along with degradation of $T_c$. Pressure studies have shown a negative effect on $T_c$, but only by less than 2 K in MgB$_2$. This is a very insignificant reduction as compared to the other existing approaches (i.e. chemical doping and irradiation) that are mainly used for $J_c$ enhancement. Therefore, it is natural to investigate the effects of hydrostatic pressure on $J_c$ and flux pinning mechanisms in MgB$_2$. Our detailed investigations can be found in Chapter 7.
2.5 References


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3 EXPERIMENTAL

3.1 Fabrication of Samples

3.1.1 Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ Polycrystalline Bulks

For the polycrystalline Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ sample synthesis, Fe (Alfa Aesar, 99.2%) and As (Alfa Aesar, 99%) chips were sealed in an evacuated quartz tube and heat treated for 12 hours at 700°C. Later, stoichiometric amounts of V$_2$O$_5$ (Aldrich, 99.6%) + $1/2\times$SrO$_2$ (Aldrich) + $7/2\times$Sr (Alfa Aesar, 99%) + $2\times$FeAs were weighed, mixed, ground thoroughly, and pelletized in rectangular form in a glove box in high purity Ar atmosphere. The pellet was further wrapped in tantalum foil and then sealed in an evacuated (10$^{-5}$ torr) quartz tube for heat treatments at 750 and 1150°C in a single step for 12 and 36 hours, respectively. Finally, the quartz ampoule was allowed to cool naturally to room temperature.

3.1.2 NaFe$_{0.7}$Co$_{0.3}$As Single Crystals

High-quality single crystals of NaFe$_{0.972}$Co$_{0.028}$As were grown by use of the NaAs flux method. NaAs was obtained by reacting a mixture of elemental Na and As in an evacuated quartz tube at 200°C for 10 h. Then, NaAs, Fe, and Co powders were carefully weighed according to the ratio of NaAsFeCo = 4:0.972:0.028, and thoroughly ground. The mixture was put into an alumina crucible and then sealed in an iron crucible under 1.5 atm of highly pure argon gas. The sealed crucibles were heated to 950°C at a rate of 60°C/h in a tube furnace filled with the inert atmosphere and kept at 950°C for 10 h, before being cooled slowly to 600°C at 3°C/h to grow single crystals.
3.1.3 Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ Single Crystal

A high quality 122 crystals were grown by using a flux method. The pure elements Ba, K, Fe, As, and Sn were mixed in a mol ratio of Ba$_{1-x}$K$_x$Fe$_2$As$_2$:Sn = 1:45–50 for the self-flux. A crucible with a lid was used to reduce the evaporation loss of K as well as that of As during growth. The crucible was sealed in a quartz ampoule filled with Ar and loaded into a box furnace.

3.1.4 MgB$_2$

The MgB$_2$ bulk sample used in the present work was prepared by the diffusion method. Firstly, crystalline boron powders (99.999%) with particle size of 0.2-2.4 µm were pressed into pellets. They were then put into iron tubes filled with Mg powder (325 mesh, 99%), and the iron tubes were sealed at both ends. Allowing for the loss of Mg during sintering, the atomic ratio between Mg and B was 1.2:2. The sample was sintered at 800°C for 10 h in a quartz tube under flowing high purity argon gas. Then, the sample was furnace cooled to room temperature.

3.2 Experimental Equipment

3.2.1 Vibrating Sample Magnetometer (VSM)

Magnetization measurements were carried out on a QD 14T physical properties measurement system (PPMS) by using the Vibrating Sample Magnetometer (VSM) option. The Quantum Design (QD) VSM for PPMS is a fast and sensitive DC magnetometer. The VSM option includes a linear motor transport for vibrating the sample, a coilset puck for detection, and the MultiVu software application.

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The sample is attached to the end of a sample rod, as shown in Figure 3-1. The measurement is carried out by oscillating the sample near a detecting coil. The magnetic measurement can be performed for $1.9 \, \text{K} < T < 400 \, \text{K}$ and for magnetic field up to 14 T. For magnetic critical current density ($J_c$) calculation, the hysteresis loops are collected at different temperatures and magnetic fields under different applied pressure.

For a long sample with a rectangular cross-section with dimensions of $l \times w$ perpendicular to the magnetic field direction, the critical current density can be calculated using the extended Bean model:

$$J_c = 20\Delta M (l (1 - l/(3w)) \quad l < w \quad ....... \quad (1)$$

Where $\Delta M$ can be calculated by using the relation $\Delta M = M_{down} - M_{up}$, where $M_{up}$ and $M_{down}$ are the magnetization when sweeping fields up and down respectively. For the zero-field-cooling (ZFC) and field-cooling (FC) measurements,
the magnitude of applied field was 50 Oe. The transition temperature measured from the magnetization was defined by the temperature where the diamagnetic signal appeared. The H (field) is perpendicular to c-axis for all samples (in a pressure cell) measurements.

3.2.2 Hydrostatic Pressure Cell

The Quantum Design High Pressure Cell with Daphne 7373 oil as a pressure transmitting medium was used to apply hydrostatic pressure on a sample before magnetic measurements. The pressure cell construction can be seen in Figure 3-2. The following procedures for using the pressure cell have been taken from Quantum Design High Pressure Cell User Manual.

![Figure 3-2: Pressure cell construction.](image)

3.2.2.1 Inserting, Pressurizing, and Removing the Sample from the Pressure Cell

A) Teflon sample tube preparation and sample insertion

1) After selecting the Teflon tube (either 2.1 or 2.6 mm OD), a Teflon tube cutting fixture was used to cut the desired length of tube for inserting the sample. The cell kit
includes all necessary parts to accommodate either size Teflon tube. The diameter of the Teflon tube depends primarily on the volume of sample to be measured (Fig 3-3). A 4.5 to 5.0 mm length of Teflon tube is recommended. Slightly longer Teflon tubes, up to 7 mm, can be used if desired, although the maximum pressure might decrease slightly. The ends of the Teflon tube must be cut clean and perpendicular to the length of the tube. If not, there will be an increased chance of damage to the Teflon caps and maximum pressure won’t be achieved. The included Teflon tube cutting fixture was used to help get a clean cut.

![Figure 3-3: Cutting the Teflon tube.](image)

2) One end of the Teflon sample tube is sealed with a Teflon cap (Fig. 3-4). Prior to use, the Teflon caps need to be carefully inspected to verify that they are not cracked or otherwise damaged from previous use.

![Figure 3-4: Setting the Teflon cap to one end of the Teflon tube.](image)
3) The Teflon sample tube/Teflon cap combination is inserted into the centre cylinder of the pressure cell, leading with the open end of the Teflon sample tube. It is pushed in with the appropriate diameter sample push rod until the end of the Teflon sample tube is about even with the bevel of the centre cylinder (Figure 3-5). The Teflon caps can be difficult to insert into the centre cylinder the first several times they are used. For smoother insertion of the Teflon caps, the Teflon cap can be pre-installed in the centre cylinder using the sample push rod. The teflon cap should be worked back and forth several times until the cap moves smoothly, and it is then removed from the centre cylinder.

![Figure 3-5: Inserting Teflon tube + cap in centre cylinder.](image)

4) The sample, manometer, and pressure transmitting medium are inserted into the open end of the Teflon sample tube. If the included Sn or Pb wire is used for the manometer, about 1 mm length is sufficient. Enough space should be left at the top of the Teflon sample tube to allow the second Teflon cap to be inserted (Fig.3-6). The teflon sample tube should be filled with as much sample as possible, with the amount of pressure transmitting medium required to fill the teflon sample tube minimized. If the filling factor of the manometer and sample is small (perhaps less
than 60–70%), achieving 1 GPa will require significantly greater pressure cell compression.

The pressure cell kit includes Daphne 7373 oil as a pressure transmitting medium. The included syringe can be used to help apply the transmitting medium to the Teflon sample tube.

5) The second end of the Teflon sample tube is sealed with a Teflon cap (Fig. 3-6). Care should be taken to minimize any air bubbles in the Teflon sample tube.

6) Using for example a Kimwipe, any excess oil or any other foreign particles should be removed from the threads of the centre cylinder and around the Teflon caps.

7) Using the appropriate diameter sample push rod, the Teflon sample tube should be carefully pushed so that it is roughly centred within the centre cylinder (Fig. 3-7).
The Teflon sample tube and end caps form a very snug fit inside the centre cylinder. Thus, a bit of force may be necessary to push the Teflon sample tube into the centre cylinder. This is normal. Any excess grease or oil should be cleaned from around the centre cylinder.

8) The two pistons are then inserted into the centre cylinder (Fig. 3-7).

![Figure 3-7: Teflon tube is centered, setting the pistons.](image)

9) Using a toothpick or small wire, a thin coat of Teflon powder is applied to the threads of the centre cylinder (Fig.3-8).

![Figure 3-8: Applying Teflon powder, piston backup, side cylinder, and pressurizing nut.](image)

10) With the centre cylinder held horizontally, a piston backup is placed on each piston (Fig. 3-8).
11) The side cylinders are threaded finger tight to the centre cylinder. There should be no gap between the side and centre cylinders (Fig. 3-8).

12) The threads of the two cylinder pressurization nuts are then coated with Teflon powder (Fig. 3-8).

13) The cylinder pressurization nuts are threaded to the side cylinders finger tight (Fig. 3-8).

14) One of the pressurization nuts is loosened by about two turns, and the opposite pressurization nut is tightened. The nut should tighten smoothly, indicating that the pistons and Teflon sample tube are moving smoothly. Next, the tightened pressurization nut is loosened by about 3 turns, and the opposite pressurization nut is tightened. Again, the pressurization nut should tighten smoothly. The pistons and Teflon sample tube need to be worked back and forth in this manner about 3 times. When finished, both pressurization nuts are finger-tightened so that the sample is centred within the centre cylinder. This is indicated by checking that the gap between the pressurization nut and side cylinder are roughly equal on both sides (Fig. 3-9).

![Figure 3-9: Final assembly before pressurizing.](image)

The sample is now inserted in the high pressure cell and is ready for pressurization and measurements.
B) Pressurizing the sample

1) The length of the pressure cell will be used as a reference so the amount of compression can later be determined (Fig. 3-10 for the recommended dimension to measure).

2) The pressure cell is inserted into the cell clamp and cinched in place with the two hex screws. The third hex screw is used to slightly pry the cell clamp apart in case the cell does not easily slide into the cell clamp (Figure 3-10).

3) The custom spanners are attached to the pressurization nuts (Fig. 3-11).

Figure 3-10: Measuring compression and inserting pressure cell into clamp.

Figure 3-11: Setting the pressurization spanners.
4) A vice or large adjustable wrench is used to hold the cell clamp stable.

5) One of the pressurization nuts is tightened by 90 degrees, then the opposite pressurization nut by 90 degrees. To limit risk of damage to the pressurization nuts due to over torquing, the pressurization nuts need to be slowly tightened.

6) The pressurization nuts continue to be tightened, alternating between the two pressurization nuts in 90 degree steps, until the total cell compression is about 1 mm. For compression greater than about 1 mm, the pressurization nuts are rotated in small steps, 30 to 45 degrees being recommended. The maximum sample compression is about 2.0 mm. 90 degrees of pressurization nut rotation represents about 0.1 mm of compression.

Note: Over pressurizing the sample region needs to be avoided. If the sample is compressed by more than about 2.2mm, there is increased risk of damage to the pressure cell. If the pressure is found to be below 1 GPa with 2 mm of compression, this indicates a problem with the cell. The most probable sources of this reduced pressure are: i) too much pressure transmitting medium / not enough sample, ii) damaged Teflon tube or caps, resulting in a leak. For applied pressures above 1 GPa, the pistons will likely be deformed at higher pressures, so the pistons are considered a single-use consumable. The pistons are reusable for applied pressure under 1GPa.

C) Removing the sample

1) The cell is inserted into the cell clamp, and the two hex bolts are tightened to cinch the cell in the cell clamp.

2) The pressurization spanners are attached to the pressurization nuts.

3) The pressurization nuts are slowly unscrewed and removed from the cell.
4) The pressurization spanners are unthreaded to grip the side cylinders.

5) The piston backups and pistons are removed.

Note: If the applied pressure was greater than about 1 GPa, the pistons may be deformed and wedged in the centre cylinder. To remove the pistons in this case, with the centre cylinder cinched in one of the pressurization spanners, use some pliers to grip the pistons. Slowly twist the pistons to remove them from the centre cylinder. If the pistons are deformed and do not smoothly insert into the center cylinder, they should be discarded.

6) Using the proper diameter sample push rod, push out the sample from the centre cylinder.

**3.2.2.2 Determining applied sample pressure**

There are two ways to determine the applied sample pressure:

1) The transition temperature of a sample with a known pressure dependence is measured. The pressure cell kit comes with both Pb and Sn for this purpose. Either AC or DC magnetization can be measured to determine the applied sample pressure. AC susceptibility measurements will typically give a sharper critical temperature ($T_c$) transition and is recommended if available.

2) As the pressurization nuts are tightened, the compression of the cell can be measured. The following Fig. 3-12 shows a typical compression vs. sample pressure for the pressure ding along the sample Teflon tube and the filling factor of the sample. Assuming that these are kept constant, it is possible to approximate the applied pressure.
In the case of Fig. 3-12, the pressure was determined by measuring the $T_c$ of a Pb manometer. A 5 mm length of Teflon sample tube was used.

![Pressure vs cell compression](image)

Figure 3-12: Typical sample pressure vs. cell compression.

The sample pressure is a function of temperature. This temperature dependence is negligible for temperatures less than about 70 K (liquid N$_2$ temperature). For room temperature, samples that show ferromagnetic ordering can be used as a manometer. For example, Gadolinium shows ferromagnetic ordering near 300 K and has a pressure dependence on the $T_c$ of 12.2 K/GPa$^{-1}$. We have used this method to determine pressure.

### 3.2.2.3 Mounting pressure cell to VSM sample rod

The large diameter VSM coil is required to use the HMD pressure cell with the QD VSM system.

The same adapter can be used for both the PPMS and VersaLab measurement options. Fig. 3-13 shows the pressure cell mounted to the VSM sample rod.
Figure 3-13: Pressure cell threaded to VSM sample rod adapter.
**HYDROSTATIC PRESSURE: A VERY EFFECTIVE APPROACH TO SIGNIFICANTLY ENHANCE $J_c$ IN GRANULAR Sr$_2$V$_2$O$_6$ Fe$_2$As$_2$ SUPERCONDUCTORS**

### 4.1 Introduction

Iron based superconductors have revealed wonderful superconducting properties, including high values of critical temperature ($T_c$), critical current density ($J_c$), upper critical field ($H_{c2}$), and irreversibility field ($H_{irr}$). They also exhibit low anisotropy and very strong pinning, which gives rise to high $J_c$ ($\sim 10^6$ A/cm$^2$) in both single crystals and thin films at both low and high fields [1-10]. The $J_c$ and its field dependence in polycrystalline bulks and tapes/wires, however, are still lower than what is required for practical applications. Enhancement of $J_c$ or flux pinning using various approaches has always been a main focus of research with a view to large current and high field applications. So far, three main methods have been used to increase the $J_c$ in cuprates, MgB$_2$, and iron based superconductors: 1) texturing processes to reduce the mismatch angle between adjacent grains and thus overcome the weak-link problem in layer-structured superconductors; 2) introducing point pinning centres by chemical doping and 3) high energy ion implantation or irradiation to introduce point defect pinning centres. $J_c$ values achieved by the irradiation method have reached as high as $10^6$-$10^7$ A/cm$^2$ for both low and high fields in single crystals and thin films [11-13]. This method is not ideal, however, for $J_c$ enhancement in polycrystalline pnictide superconductors.

As is well known, the weak-link issue is the predominant factor causing low $J_c$, especially at high fields in pnictide polycrystalline samples, which must be overcome. In order to improve the $J_c$ and its field dependence in granular superconductors, the following prerequisites should be met: i) strong grain
We have taken into account that the following facts relating to flux pinning mechanisms must be addressed before an effective method is introduced for polycrystalline pnictide superconductors. The coherence length is very short ($\xi \approx$ a few nm), so elimination of weakly linked grain boundaries is important to achieve high $J_c$ [14]. The nature of the pinning mechanism plays a vital role in $J_c$ field dependence. It is noteworthy that a high pinning force can boost pinning strength and, in turn, leads to higher values of $J_c$. The ideal size of defects for pinning should be comparable to the coherence length [15]. Therefore, point defect pinning is more favourable than surface pinning, as its pinning force is larger than for surface pinning at high field, according to the Dew-Hughes model [16]. Therefore, it is very desirable to induce more point defects in superconductors. Although chemical doping and high energy particle irradiation can effectively induce point defects and enhance $J_c$ in high fields, $T_c$ and low field $J_c$ deteriorate greatly for various types of superconductors. Therefore, the ideal approach should be the one which can induce more point defects, with increased (or at least at no cost of) superconducting volume and $T_c$, as well as strongly linked grain boundaries.

Hydrostatic pressure has been revealed to have a positive effect on $T_c$ in cuprate and pnictide superconductors. For instance, high pressure of 150 kbars can raise $T_c$ of Hg-1223 significantly from 135 K to a record high 153 K [17]. The $T_c$ of hole doped (NdCeSr)CuO$_4$ was increased from 24 to 33 K at 3 GPa by changing the apical Cu-O distance [18]. The enhancement of $T_c$ for YBCO is more than 10 K at 2 GPa [19]. Excitingly, pressure also shows positive effects on $T_c$ for various pnictide
superconductors. Pressure can result in improvement of $T_c$ from 28 to 43 K at 4 GPa for LaOFFeAs [20]. For Co doped NaFeAs, the maximum $T_c$ can reach as high as 31 K from 16 K at 2.5 GPa [21]. Pressure can also enhance the $T_c$ of La doped Ba-122 epitaxial films up to 30.3 K from 22.5 K, due to the reduction of electron scattering and increased carrier density caused by lattice shrinkage [22]. A huge enhancement of $T_c$ from 13 to 27 K at 1.48 GPa was observed for FeSe, and it reached the high value of 37 K at 7 GPa [23, 24].

Beside the above-mentioned significant pressure effects on $T_c$ enhancement, pressure can have more advantages that are relevant to the flux pinning compared to other methods. 1) It always reduces the lattice parameters and causes the shrinkage of unit cells, giving rise to the reduction of anisotropy. 2) Grain connectivity improvement should also be expected, as pressure can compress both grains and grain boundaries. 3) The existence or formation of point defects can be more favourable under pressure, since it is well known that the formation energy of point defects decreases with increasing pressure [25-27]. 4) Pressure can cause low-angle grain boundaries to migrate in polycrystalline bulk samples, resulting in the emergence of giant grains, sacrificing surface pinning thereafter. Hence, a higher ratio of point pinning centres to surface pinning centres is expected due to the formation energy and migration of grain boundaries under pressure. 5) The significant enhancement of $T_c$, as above-mentioned, means that superconducting volumes should be increased greatly below or above the $T_c$ without pressure. Moreover, the $H_{c2}$, $H_{irr}$, and $J_c$ have to be enhanced along with the $T_c$ enhancement. These are the motivations of our present study on the pressure effects on flux pinning and $J_c$ enhancement in polycrystalline pnictide bulks. We anticipated that hydrostatic pressure would increase the superconducting volume,
$H_{\nu r}$, and $H_{c2}$ due to $T_c$ enhancement, increase the point defects, improve grain connectivity, and reduce the anisotropy in pnictide polycrystalline bulk samples. There is some evidence for $J_c$ enhancement under pressure in YBa$_2$Cu$_3$O$_{7-x}$ (YBCO) single crystal, which emphasizes the pressure effects on transport $J_c$ for different angle grain boundaries. A recent report also shows enhanced $J_c$ in a pnictide single crystal which is free of grain boundaries [28-30]. As mentioned earlier, polycrystalline superconducting materials are commonly used in practical applications, as they are easy to fabricate at low cost as compared to single crystals/thin films. Their superconducting performance is hindered by grain boundaries, however, due to granularity. Therefore, it is more important to use an efficient approach to enhance the $J_c$ in polycrystalline bulk samples. In this study, we chose a polycrystalline Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ sample to demonstrate the significant effects of the hydrostatic pressure on flux pinning and the significant enhancement of $J_c$ and $T_c$ in this granular sample. It has been reported that the $T_c$ for this compound can range from 15 - 30 K, depending on fabrication process and carrier concentration [31]. Generally, $T_c$ under pressure remains nearly constant (or little increase) for optimal doped superconductors and decreases linearly in the overdoped range. Under doped superconductors under pressure have dome-like plots for $T_c$ vs. pressure, so we chose a Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ sample with the low $T_c$ of 15 K for the proposed pressure effect investigation to ensure a clear pressure effect on $T_c$ [32]. Our results show that pressure can enhance the $J_c$ by more than 30 times at 6 K and high fields in polycrystalline Sr$_4$V$_2$O$_6$Fe$_2$As$_2$, along with $T_c$ enhancement from 15 to 22 K at 1.2 GPa and $H_{\nu r}$ enhancement by a factor of 4. Our analysis shows that pressure induced point defects inside the grains are mainly responsible for the flux pinning enhancement.
4.2 Results and Discussion

Figure 1 shows the temperature dependence of the zero-field-cooled (ZFC) and field-cooled (FC) moments for Sr$_4$V$_2$O$_8$Fe$_2$As$_2$ at different pressures. Pressure causes little change to the field-cooled branch, indicating that strong pinning is retained under pressure. The $T_c$ without pressure is about 15 K, very similar to that of underdoped samples reported for Sr$_4$V$_2$O$_8$Fe$_2$As$_2$ bulks [31]. Pressure enhances $T_c$ linearly from 15.3 K for $P = 0$ GPa to 22 K for $P = 1.2$ GPa, with the pressure coefficient, $dT_c/dP = 5.34$ K/GPa.

![Figure 4-1](image)

**Figure 4-1:** Temperature dependence of ZFC and FC moments at different pressures for Sr$_4$V$_2$O$_8$Fe$_2$As$_2$. The inset shows the pressure dependence of $T_c$.

The $M$-$H$ curves measured under different pressures indicate that the moment increases with increasing pressure. The field dependence of $J_c$ at different temperatures obtained from the $M$-$H$ curves by using Bean’s model under different pressures is shown in a double-logarithmic plot [i.e. Figure 2]. The remarkable effect of pressure towards the enhancement of $J_c$ can be clearly seen. For $P = 1.2$ GPa, the $J_c$ is significantly enhanced by more than one order of magnitude at high fields at 4 and 6 K, respectively, as shown in Figure 3.
Figure 4-2: Field dependence of $J_c$ under different pressures at 3, 4, 6, and 7 K.

The $J_c$ at 6 K as a function of pressure at different fields is plotted in Figure 4. The solid lines in Figure 4 show linear fits to the data, which give the slopes (i.e. $d(\ln J_c)/dP$) of 1.09, 1.69, and 2.30 GPa$^{-1}$ at 0, 2, and 4 T, respectively, indicating that the effects of pressure towards the enhancement of the $J_c$ are more significant at high fields.

Figure 4-3: Comparison of $J_c$ at 0 and 1.2 GPa at 4 and 6 K. The inset shows $d(\ln J_c)/dP$ versus temperature, indicating enhancement of $J_c$ at a rate of 1.08 GPa$^{-1}$ at zero field.
Figure 4-4: Pressure dependence of \( J_c \) (logarithmic scale) at 0, 2, and 4 T at the temperature of 6 K.

We also found that the \( H_{irr} \) of Sr\(_4\)V\(_2\)O\(_6\)Fe\(_2\)As\(_2\) is greatly increased by pressure. The \( H_{irr} \) is defined as a field where \( J_c \) reaches as low as 10 A/cm\(^2\) in \( J_c \) vs field curves for different pressures and temperatures. As shown in Figure 5, the \( H_{irr} \) increases gradually with pressure and rises to 13 T from 3.5 T at 7 K.

Figure 4-5: \( H_{irr} \) vs. \( T \) for different pressures.
The $J_c$ vs. reduced temperature ($1-T/T_c$) at zero field and different pressures is plotted in Figure 6, which shows a rough scaling behaviour as $J_c \propto (1 - T/T_c)^\beta$ at different pressures. The slope of the fitting line, $\beta$, depends on the magnetic field. The exponent $\beta$ (i.e. slope of the fitting line) is found to be 2.54, 2.73, 2.96, and 3.13 at 0, 0.25, 0.75, and 1.2 GPa, respectively. According to Ginzburg-Landau theory, the exponent “$\beta$” is used to identify different vortex pinning mechanisms at specific magnetic fields. It was found that $\beta = 1$ for non-interacting vortices, while $\beta \geq 1.5$ indicates the core pinning mechanism [33]. The different values of $\beta$ (i.e. 1.7, 2, and 2.5) were also reported for YBCO films which show the functioning of different core pinning mechanisms [34, 35]. In addition, the exponent $\beta$ values that we obtained are higher at higher pressures in our sample, indicating stronger improvement of $J_c$ with temperature at high pressures.

Figure 4-6: Logarithmic plot of $J_c$ as a function of reduced temperature at different pressures and fields.
For polycrystalline samples, high pressure can modify the grain boundaries through reducing the tunnelling barrier width and changing the tunnelling barrier height. The Wentzel-Kramers-Brillouin (WKB) approximation applied to a potential barrier gives the following simple expressions [36]:

\[
J_c = J_{c0} \exp \left( -2kW \right) \quad (1)
\]

Where \( W \) is the barrier width, \( k = (2mL)^{1/2}/\hbar \) is the decay constant, which depends on the barrier height \( L \), \( \hbar \) is the Planck constant, and \( J_{c0} \) is the critical current density for samples with no grain boundaries. The relative pressure dependence of \( J_c \) can be obtained from Eq. (1) as:

\[
\frac{d \ln J_c}{dP} = \frac{d \ln J_{c0}}{dP} - \left[ \left( \frac{d \ln W}{dP} \right) \ln \left( \frac{J_{c0}}{J_c} \right) \right] - \frac{1}{2} \left[ \left( \frac{d \ln L}{dP} \right) \ln \left( \frac{J_{c0}}{J_c} \right) \right]
\]

\[
= \frac{d \ln J_{c0}}{dP} + \kappa_{GB} \ln \left( \frac{J_{c0}}{J_c} \right) + \frac{1}{2} \kappa_L \ln \left( \frac{J_{c0}}{J_c} \right) \quad (2)
\]

Where the compressibility in the width and height of the grain boundary are defined by \( \kappa_{GB} = -d \ln W/dP \) and \( \kappa_L = -d \ln L/dP \), respectively. To estimate their contributions to the second and the third terms of Eq. (2) for \( J_c \) enhancement, we assume to a first approximation that \( \kappa_{GB} \) and \( \kappa_L \) are roughly comparable to the average linear compressibility values \( \kappa_a = -d \ln a/dP \) (\( \kappa_{GB} \approx \kappa_a \)) and \( \kappa_c = -d \ln c/dP \) (\( \kappa_L \approx \kappa_c \)) of \( \text{Sr}_4\text{V}_2\text{O}_6\text{Fe}_2\text{As}_2 \) in the FeAs plane, where \( a \) and \( c \) are the in-plane and out-of-plane lattice parameters, respectively. We assume to a first approximation that \( \kappa_a = -d \ln a/dP = -0.029 \text{ GPa}^{-1}, \kappa_c = -d \ln c/dP = -0.065 \text{ GPa}^{-1} \) [24], and the in-plane \( J_{c0} \approx 10^5 \text{ A/cm}^2 \) for a sample with no grain boundaries (single crystal) [37]. Setting \( J_c \approx 2 \times 10^3 \text{ A/cm}^2 \) at the temperature of 6 K and ambient pressure, we find that \( (-d \ln W/dP) \ln (J_{c0}/J_c) \approx 0.11 \) and \(-0.5(d \ln L/dP) \ln (J_{c0}/J_c) \) \approx 0.13, with both values adding up to 75% less than the above experimental value \( d \ln J_c/dP = 1.08 \text{ GPa}^{-1} \). This result suggests that the origin of the significant increase in \( J_c(T) \) under pressure does
not arise from the compression of the grain boundaries. Therefore, Eq. (2) suggests that the main reason for the rapid increase of $J_c$ with pressure is through point defects induced under pressure, i.e., $d\ln J_c/dP$ is responsible for approximately 75% of the total increase in the $J_c$ with pressure.

![Figure 4-7: Plots of $f_p$ vs. $H/H_{Ir}$ at different pressures (0, 0.75, and 1.2 GPa) for 4 (left) and 6 K (right) temperature curves. The experimental data is fitted through the Dew-Hughes model, and the parameters are shown.](image)

In order to further understand the $J_c$ enhancement under pressure, the pinning force $F_p = B \times J_c$ is calculated, and the scaling behaviour for the normalized pinning force $f_p = F_p/F_{p,max}$, is analysed for $h = H/H_{Ir}$. The results are shown in Figure 7 at 4 and 6 K under 0, 0.75, and 1.2 GPa. For the scaling, we can use the Dew-Hughes formula, i.e. $f_p(h) = Ah^p(1-h)^q$, where $p$ and $q$ are parameters describing the pinning mechanism [16]. In this model, $p = 1/2$ and $q = 2$ describes surface pinning while $p = 1$ and $q = 2$ describes point pinning, as was predicted by Kramer [38]. At ambient pressure in the temperature range of 3-7 K, the best fits of the curves are obtained.
with $p = 0.51 \pm 0.03$, $q = 1.86 \pm 0.03$, which suggests that surface pinning is the dominant pinning mechanism in our sample. At 1.2 GPa, the best obtained values for $p$ and $q$ were $0.9 \pm 0.1$ and $2.0 \pm 0.1$, respectively, within the studied temperature range. This means that the dominant pinning mechanism is normal core point pinning for high pressures. Therefore, our results show that the pressure has induced a clear transformation from surface to point pinning.

Moreover, it is noteworthy that pressure can induce a reduction in anisotropy. The anisotropy is defined as $\gamma = \xi_{ab}/\xi_c$ where $\xi_{ab}$ is a coherence length along ab plane and $\xi_c$ along c plane. At high temperatures, the pressure dependence of $T_c$, unit cell volume ($V$), and anisotropy ($\gamma$) are interconnected through the following relation [39]:

$$\frac{-\Delta T_c}{T_c} = \frac{\Delta V(T_c)}{V(T_c)} + F(\gamma)$$  \hspace{1cm} (3)

Where $F(\gamma) = [\gamma(P) - \gamma(0)/\gamma(0)]$. Although, no report for bulk modulus of Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ is yet available, we have tentatively used the bulk modulus ($K= 62$ GPa) of a similar superconductor i.e., SrFe$_2$As$_2$, to estimate $\Delta V(T_c)/V(T_c)$, which is found to be $\sim -0.016$ at $\Delta P = 1$ GPa, as it can be related to the bulk modulus as $\Delta V/V = -\Delta P/K$ [40]. Our experimental results yield a value of 0.486 for $\Delta T_c/T_c = [T_c(P) - T_c(0)]/T_c(0)$. By using these results, we can obtain $\gamma(P) \approx 0.53 \gamma(0)$. Thus, we can conclude that the anisotropy has been reduced by almost half at high temperature by pressure. The decrease in the unit cell parameters suppresses its volume, leading to an increase in the Fermi vector $k_F = (3\pi^2 N/V)^{1/3}$, where $N$ is the total number of electrons in the system. The increase in the Fermi vector promotes enhancement of the coherence length along the $c$-axis ($\xi_c = \hbar^2 K_F / \pi m \Delta$) where $\Delta$ is the uniform energy gap which, in turn, leads to the suppression of anisotropy.
4.3 Conclusion

In summary, hydrostatic pressure is a very effective means to significantly enhance $T_c$, $J_c$, $H_{irr}$, and flux pinning in the granular pnictide superconductor Sr$_4$V$_2$O$_8$Fe$_2$As$_2$. We demonstrate that the hydrostatic pressure can significantly increase $T_c$ from 15 to 22 K, as well as increasing $J_c$ by up to 30 times at both low and high field and increasing $H_{irr}$ by a factor of 4 at $P=1.2$ GPa. Pressure introduces more point defects inside grains, so that it is mainly responsible for $J_c$ enhancement. In addition, we found that the transformation from surface pinning to point pinning induced by pressure was accompanied by a reduction of anisotropy at high temperatures. Our findings provide an effective method to significantly enhance $T_c$, $J_c$, $H_{irr}$, and $H_{c2}$ for other families of Fe-based superconductors in the forms of wires/tapes, films, and single and polycrystalline bulks.
4.4 Experimental

For the polycrystalline Sr$_4$V$_2$O$_8$Fe$_2$As$_2$ sample synthesis, the Fe (Alfa Aesar, 99.2%) and As (Alfa Aesar, 99%) chips were sealed in evacuated quartz tube and heat treated for 12 hours at 700°C. Later, the stoichiometric amounts of V$_2$O$_5$ (Aldrich, 99.6%) + 1/2 × SrO$_2$ (Aldrich) + 7/2 × Sr (Alfa Aesar, 99%) + 2 × FeAs were weighed, mixed, ground thoroughly and palletized in rectangular form in a glove box in a high purity Ar atmosphere. The pellet was further wrapped in tantalum foil and then sealed in an evacuated (10$^{-5}$ torr) quartz tube and put for heat treatments at 750 and 1150°C in a single step for 12 and 36 hours respectively. Finally, the quartz ampoule was allowed to cool naturally to room temperature.

The temperature dependence of the magnetic moments and the M-H loops at different temperatures and pressures were performed on Quantum Design Physical Property Measurement System (QD PPMS 14T) by using Vibrating Sample Magnetometer (VSM). We have used HMD High Pressure cell and Daphne 7373 oil as a pressure transmitting medium to apply hydrostatic pressure on a sample. The critical current density was calculated by using the Bean approximation.
4.5 References


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GIANT ENHANCEMENT IN $J_c$, UP TO A HUNDREDFOLD, IN
SUPERCONDUCTING NaFe$_{0.97}$Co$_{0.03}$As SINGLE CRYSTALS UNDER
HYDROSTATIC PRESSURE

5.1 Introduction

It is already anticipated in our previous case study that the most significant approach
to enhancing $J_c$, particularly at high fields and temperatures, without degradation of
$T_c$, is the use of hydrostatic pressure [1]. Most recent research regarding $J_c$
enhancement and pinning mechanisms through doping and high energy ion
irradiation is mainly focused on the 1111 system (RFeAsO, where R is a rare earth
element), the 122 system (BaFe$_2$As$_2$, Ba$_{0.5}$K$_{0.5}$Fe$_2$As$_2$) and the iron chalcogenide 11
system. Only one report has revealed the nature of the pinning mechanism in LiFeAs
(111 type FeSCs) so far, despite its simple structure and reasonable $T_c$ value as
compared to the 1111 and 122 types [2]. NaFeAs ($T_c \approx$10K) experi-
ences three successive phase transitions around 52, 41, and 23 K, which can be related to
structural, magnetic, and superconducting transitions, respectively [3-5]. Bulk
superconductivity in NaFeAs with $T_c$ of $\sim$20K can be achieved by the substitution of
Co on Fe sites, which can suppress both magnetism and structural distortion [6, 7].
The $T_c$ of NaFe$_{0.97}$Co$_{0.03}$As single crystal is more sensitive to hydrostatic pressure as
compared to other 11 and 111 Fe-based superconductors, and it has a large positive
pressure coefficient [7]. In addition, $J_c$ values for Co-doped NaFeAs compounds
have not been reported so far. Therefore, it is very interesting to see if the hydrostatic
pressure can significantly improve the flux pinning for this compounds. High-quality
NaFe$_{0.97}$Co$_{0.03}$As single crystals were grown by the conventional high temperature
solution growth method using the NaAs self-flux technique [7]. In this chapter, we
report that hydrostatic pressure can enhance the $J_c$ by more than 100 times at high
fields at 12 K and 14K in NaFe$_{0.97}$Co$_{0.03}$As single crystal. This is a giant enhancement of $J_c$ and a record high to the best of our knowledge. The $H_{irr}$ is improved by roughly 6 times at 14K under $P=1$GPa.

### 5.2 Results and Discussion

The temperature dependence of the magnetic moment for zero-field-cooled (ZFC) and field-cooled (FC) curves at different pressures are shown in Fig.1. The $T_c$ increases with pressure, from 17.95K for $P = 0$ GPa to 24.33K for $P = 1$ GPa, with a huge pressure coefficient, i.e. $dT_c/dP \approx 6.36$K/GPa, which is nearly same to what we have already reported for NaFe$_{0.97}$Co$_{0.03}$As single crystal i.e. $dT_c/dP = 7.06$K·GPa$^{-1}$[7]. Interestingly, this pressure coefficient is more than two times greater than that of FeSe (3.2K·GPa$^{-1}$) [8]. The pressure-induced enhancement of $T_c$ in NaFe$_{0.97}$Co$_{0.03}$As can be associated with the optimization of the structural parameters of the FeAs layers, including the As–Fe–As bond angle and anion height [7, 9].

![Figure 5-1: Temperature dependence of magnetic moment at different applied pressures in both ZFC and FC runs for NaFe$_{0.97}$Co$_{0.03}$As.](image_url)

The field dependence of $J_c$ at different temperatures obtained from the $M-H$ curves by using Bean’s model, at $P=0$GPa, $P=0.45$GPa and $P=1$GPa are shown in Fig.2. Remarkably, $J_c$ is increased significantly at both low and high fields, especially with
enhancement of more than 10 times and up to more than 100 times for low and high fields at both 12 and 14 K, respectively. The significant positive effect of hydrostatic pressure on the $J_c$ at high fields and temperatures is further reflected in Fig.3, which shows the $J_c$ enhancement ratio (i.e. $J_c^{1GPa}/J_c^{0GPa}$) at 12 and 14K over a wide range of fields. We have taken the $J_c$ value at $P=0$GPa as a reference. The $J_c$ ratio values at both temperatures show significant improvements at low and high fields. Although this result also suggests that hydrostatic pressure is more effective at high fields and temperatures, it is worth mentioning that $J_c$ values are well improved at zero field at a significant rate, i.e. $d(ln J_c)/dP = 1.6$ and $2.9 \text{ GPa}^{-1}$ at 12 and 14K, respectively, as can be seen from the inset of Fig. 3. The $d(ln J_c)/dP$ values that have been found are more significant than for yttrium barium copper oxide (YBCO) [10].

Figure 5-2: Field dependence of $J_c$ at different pressures (0, 0.45 and 1GPa) at different temperatures
Figure 5-3: Plot of $J_{c1GPa}/J_{c0GPa}$ versus field at 12K and 14K. There is a giant enhancement in $J_c$ values at $P = 1$ GPa. The inset shows the plot of $d(\ln J_c)/dP$ versus temperature, which demonstrates enhancement in $\ln J_c$ at a rate of nearly $3 \text{GPa}^{-1}$ at 14K.

We also found that $H_{irr}$ of NaFe$_{0.97}$Co$_{0.03}$As is significantly increased by pressure. As shown in Fig.4, the $H_{irr}$ values improve gradually with pressure, and the $H_{irr}$ value at 14K is increased from 2.6 T at $P=0$ GPa up to 8.67T at $P=0.45$GPa and roughly more than 13 T at $P=1$GPa (by nearly six times).

Figure 5-4: Plot of $H_{irr}$ versus temperature at different pressures. Inset shows $H_{irr}$ as a function of reduced temperature.
In Fig. 5, we show the temperature dependence of $J_c$ at 0 and 12T under different pressures. It follows power law [$J_c \propto (1-T/T_c)^\beta$] behaviour at different pressures. According to the Ginzburg-Landau theory, the exponent $\beta$ is used to identify different vortex pinning mechanisms at specified fields. It was found that $\beta = 1$ refers to non-interacting vortices and $\beta \geq 1.5$ corresponds to the core pinning mechanism [11]. The exponent $\beta$ (i.e. slope of the fitting line) is found to be 1.79 and 1.85 for zero field, and 2.73 and 4.28 at 12T, at 0 and 1GPa, respectively, which shows strong $J_c$ dependence on pressure. The low values of $\beta$ at $P=1$GPa indicate that the $J_c$ decays rather slowly in comparison to its values at $P=0$GPa. In addition, the differences between $P=0$GPa and $P=1$GPa scaling show a real pressure effect, the factor is roughly 2, which corresponds nicely to the low-T data in inset Fig. 3.

Figure 5-5: Logarithmic plot of critical current density as a function of reduced temperature at different pressures and magnetic fields.

For single crystals, high pressure can modify an average linear compressibility value which is similar to those in polycrystalline and textured samples through reduction of the tunnelling barrier width and the tunnelling barrier height of grain boundaries. The
Wentzel-Kramers-Brillouin (WKB) approximation applied to a potential barrier gives the following simple expression [12-14]:

\[
J_c = J_{c0} \exp(-2kW) \ldots (1)
\]

Where \( W \) is the barrier width, \( k=(2mL)^{1/2}/\hbar \) is the decay constant, which depends on the barrier height \( L \), \( \hbar \) is the Planck constant, and \( J_{c0} \) is the critical current density at 0K and 0T, and its value is roughly \( 10^5 \text{A/cm}^2 \) in our case. The relative pressure dependence of \( J_c \) can be obtained from Eq. (1) as[15]:

\[
\frac{d \ln J_c}{dP} = \frac{d \ln J_{c0}}{dP} - \left[ (\frac{d \ln W}{dP}) \ln \left( \frac{J_{c0}}{J_c} \right) \right] - \frac{1}{2} \left[ (\frac{d \ln L}{dP}) \ln \left( \frac{J_{c0}}{J_c} \right) \right] \\
= \frac{d \ln J_{c0}}{dP} + \kappa_{GB} \ln \frac{J_{c0}}{J_c} + \frac{1}{2} \kappa_L \ln \frac{J_{c0}}{J_c} \ldots \ldots (2)
\]

Where the compressibility in the width and height of the grain boundary are defined by \( \kappa_{GB} = -d \ln W/dP \) and \( \kappa_L = -d \ln L/dP \), respectively. For single crystals, we assume to a first approximation that \( \kappa_{GB} \) and \( \kappa_L \) are roughly comparable, respectively, to the average linear compressibility values \( \kappa_a=-dlna/dP \) (\( \kappa_a \approx -0.029 \text{GPa}^{-1} \)) and \( \kappa_c=-dln\ c/dP \) (\( \kappa_c \approx -0.065 \text{GPa}^{-1} \)) of NaFe\(_{0.97}\)Co\(_{0.03}\)As in the FeAs plane, where \( a \) and \( c \) are the in-plane and out-of-plane lattice parameters, respectively [11]. Therefore, we can write Eq. (2) as

\[
\frac{d \ln J_c}{dP} \approx \frac{d \ln J_{c0}}{dP} + \kappa_a \ln \frac{J_{c0}}{J_c} + \frac{1}{2} \kappa_c \ln \frac{J_{c0}}{J_c} \ldots \ldots (3)
\]

By using \( J_c \approx 1.3 \times 10^3 \text{A/cm}^2 \) at 14 K, we find that \((\kappa_a \ln(J_{c0}/J_c)) \approx 0.12\text{GPa}^{-1} \) and \((1/2\kappa_c \ln(J_{c0}/J_c)) \approx 0.14\text{GPa}^{-1} \), so that both of them together only contribute less than 10% to the already mentioned experimental value \( d\ln J_c/dP = 2.09 \text{GPa}^{-1} \) inset of Fig. 2b). This result suggests that the origin of the significant increase in \( J_c(T) \) under pressure does not arise from the reduction of volume but mainly due to the pressure induced pinning centre phenomenon.
Figure 5-6: Top panel shows normalized temperature dependence ($t = T/T_c$) of normalized measured $J_c$ at 0.1T and 0T, in good agreement with $\delta l$ pinning. Lower panel shows plots of $F_p$ vs. $H/H_{irr}$ at $P=0$GPa and $P=0.45$GPa at 14K. The experimental data is fitted through D. Dew Hughes model.

To gain further insight into the pressure effect on the pinning mechanism in NaFe$_{0.97}$Co$_{0.03}$As, the experimental results have been analysed by using collective pinning theory. There are two predominant mechanisms of core pinning, i.e. $\delta l$ pinning, which comes from spatial variation in the charge carrier mean free path,$\ell$, and $\delta T_c$ pinning due to randomly distributed spatial variation in $T_c$. According to the theoretical approach proposed by Griessen et al. [16], $J_c(t)/J_c(0) \propto (1 - t^2)^{5/2}(1 + t^2)^{-1/2}$ in case of $\delta l$ pinning, whereas $J_c(t)/J_c(0) \propto (1 - t^2)^{7/6}(1 + t^2)^{5/6}$ corresponds to $\delta T_c$ pinning, where $t = T/T_c$. Fig.6 (Top Panel) shows a comparison
between the experimental $J_c$ values and the theoretically expected variation within the $\delta \ell$ and $\delta T_c$ pinning mechanisms at 0.1T and 0 T (the so-called remanent state shown by the solid symbols). The $J_c(t)$ values have been obtained from the $J_c(B)$ curves at various temperatures. It is found that the experimental data at $P=0$ and $P=1$GPa are in good agreement with theoretical $\delta \ell$ pinning. It is more likely that pinning in NaFe$_{0.97}$Co$_{0.03}$As originates from spatial variation of the mean free path “$\ell$”. We observed similar results in BaFe$_{1.9}$Ni$_{0.1}$As$_2$ and SiCl$_4$ doped MgB$_2$ at low fields. In addition, $\delta \ell$ pinning has also been reported in FeTe$_{0.7}$Se$_{0.3}$ crystal [17-19].

In order to understand the nature of the pinning mechanisms in more detail, it is useful to study the variation of the vortex pinning force density, $F_p = J_c \times B$ with the field. The normalized pinning force density ($F_p/F_{p\text{ max}}$) as a function of reduced field ($H/H_{\text{irr}}$) at $P=0$GPa and $P=0.45$GPa at 14K is plotted in lower panel of Fig.6. $H_{\text{irr}}$ is estimated by using the criterion of $J_c \approx 100$A/cm$^2$. We can use the Dew-Hughes formula, i.e. $F_p \propto h^m(1-h^n)$ to fit our experimental data, where $m$ and $n$ are fitting parameters to describe the nature of the pinning mechanism. We found that $m=1.15$ and $n=2$ at 0GPa, and $m =1.1$ and $n=2.1$ at 0.45GPa. According to the Dew-Hughes model, in the case of $\delta \ell$ pinning for a system dominated just by point pinning, the values of the fitting parameters are $m=1$ and $n=2$, with the $F_p\text{ normalized maxima}$ maximum occurring at $h_{\text{max}} = 0.33$, while $h_{\text{max}}$ occurs at 0.20 for surface/grain boundary pinning with $m=0.5$ and $n=2$. In case of $\delta T_c$ pinning, $h_{\text{max}}$ shifts to higher values, and the fitting parameters change accordingly. Further details can be found elsewhere [20]. The values of $m$ and $n$ that were found in the present study are almost the same at 0GPa and 0.45GPa, so normal core point pinning is dominant in our material.
Pressure can enhance the pinning force strength by a significant amount in NaFe$_{0.97}$Co$_{0.03}$As single crystal. The pinning force density as a function of field at 12K and 14K is plotted in Fig. 7. At high field and pressures, the $F_p$ is found to be over 20 and 80 times higher than at 0 GPa at 12 and 14K, respectively. Furthermore, pressure induces more point pinning centres at 12 K and 14 K, especially at $P=1$GPa, as can be seen in the inset of Fig. 7. The number density of randomly distributed effective pinning centres ($N_p$) can be calculated from the following relation:

$$\frac{\Sigma F_p}{\eta f_p^{\max}} = N_p \quad \ldots \ldots (4)$$

Where $\Sigma F_p$ is the aggregated pinning force density, $f_p^{\max}$ is the maximum normalized elementary pinning force ($f_p$), and $\eta$ is an efficiency factor. The $\eta$ value is 1 in the case of a plastic lattice, and the $\eta$ value is otherwise $f_p^{\max}/B$, where $B$ is the bulk modulus of the material [21]. We can assume that $\eta = 1$, as pressure can shrink lattice parameters. The inset of Fig. 7 shows the $N_p$ versus temperature plot at
$P=0$ and $P=1$GPa. The $N_p$ values are found to be much greater at 14 K at $P=1$GPa as compared to $N_p$ at $P=0$GPa (nearly six times as great). It is well known that hydrostatic pressure induces pinning centres which, in turn, leads to huge values of $J_c$ and increase in $N_p$ at $P=1$GPa is a direct evidence of that. This is further verified in Fig.8, which shows the plot of $J_c / J_c^{\max}$ versus reduced field (i.e. $h = H/H_{irr}$) at $P=0$GPa and $P=1$GPa for 14K and $P=0$GPa & $P=0.45$GPa for 12K. Obviously, the hump or secondary peak effect observed at high pressures suggests that the $J_c$ enhancement is due to induced pinning centres.

![Figure 5-8: Reduced field dependence of the normalized $J_c$ at 14K for different pressures. The inset shows the same plot for 12K at $P=0$GPa and $P=0.45$GPa](image)

Additionally, we found a pronounced reduction in the superconducting anisotropy at high temperatures, by almost 63% at $P=1$GPa. The pressure dependence of the $T_c$, volume ($V$) and anisotropy ($\gamma$) are interconnected through a relation [22]:

$$-\left[\frac{T_c^P - T_c^0}{T_c^0}\right] = \left[\frac{\Delta V}{V}\right] + \left[\frac{\gamma P - \gamma_0}{\gamma_0}\right] \ldots \ldots \ldots (5)$$
At $\Delta P=1\text{GPa}$, $\Delta V(T_c)/V(T_c)$ is estimated to be -0.02, as $\Delta V/V=-\Delta P/B$, where $K$ is the bulk modulus. We can use the bulk modulus ($B\approx 52.3\text{GPa}$) of a similar superconductor, i.e. Na$_{1-x}$FeAs [9]. The value of $\gamma$ at $P=1\text{GPa}$ is found to be 63% less than its value at $P=0\text{GPa}$.

5.3 Conclusion

Hydrostatic pressure can significantly enhance $J_c$ by up to $10^2$ times in NaFe$_{0.97}$Co$_{0.03}$As single crystals, which is a record high enhancement. The most significant enhancement in in-field performance of NaFe$_{0.97}$Co$_{0.03}$As in terms of pinning force density ($F_p$) is found at $P=1\text{GPa}$ in particular, where the $F_p$ at high fields is over 20 and 80 times higher at 12 and 14K, respectively, than at 0 GPa. The hydrostatic pressure induces more effective point pinning centres and $N_p$ at 1GPa is almost two times higher at 12K and over six times higher at 14K compared to the value at 0GPa. Moreover, a hump or secondary peak effect is found from the plot of the normalized $J_c$ as a function of reduced field. Therefore, this giant enhancement in $J_c$ values for NaFe$_{0.97}$Co$_{0.03}$As exists because of more pinning centres induced by pressure and the increase in pinning strength as well. The present study indicates that the supercurrent carrying ability in the Fe111 can be further and significantly increased by the proposed hydrostatic pressure technique. Our results were achieved in single crystal sample, which means that the enhancement is intrinsic, and more significant than other reported approaches. It gives us high expectations that the tapes or wires made by the same compounds should carry higher supercurrents using the hydrostatic pressure than those at ambient pressure.
5.4 Experimental

High-quality single crystals of NaFe$_{0.97}$Co$_{0.03}$As have been grown by use of the NaAs flux method. NaAs was obtained by reacting the mixture of the elemental Na and As in an evacuated quartz tube at 200$^\circ$C for 10 h. Then NaAs, Fe, and Co powders were carefully weighed according to the ratio of NaAs:Fe:Co = 4:0.972:0.028, and thoroughly ground. The mixtures were put into alumina crucibles and then sealed in iron crucibles under 1.5 atm of highly pure argon gas. The sealed crucibles were heated to 950$^\circ$C at a rate of 60$^\circ$C/h in the tube furnace filled with the inert atmosphere and kept at 950$^\circ$C for 10 h and then cooled slowly to 600$^\circ$C at 3$^\circ$C/h to grow single crystals.

The temperature dependence of the magnetic moments and the M-H loops at different temperatures and pressures were performed on Quantum Design Physical Property Measurement System (QD PPMS 14T) by using Vibrating Sample Magnetometer (VSM). We have used HMD High Pressure cell and Daphne 7373 oil as a pressure transmitting medium to apply hydrostatic pressure on a sample. The critical current density was calculated by using the Bean approximation.
5.5 References


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6 OBSERVATION OF STRONG FLUX PINNING AND POSSIBLE MECHANISM FOR CRITICAL CURRENT ENHANCEMENT UNDER HYDROSTATIC PRESSURE IN OPTIMALLY DOPED (Ba,K)Fe$_2$As$_2$ SINGLE CRYSTALS

6.1 Introduction

The previous results show that $J_c$ is enhanced significantly under hydrostatic pressure at high fields (i.e., over one order of magnitude) in comparison to low fields, along with enhancement of the closely related $T_c$ by more than 5 K in iron based superconductors [1, 2]. Until now, however, it has been unclear that the observed $J_c$ enhancement under pressure is correlated with improved $T_c$ or flux pinning. While the primary motivation for this experiment was to use an optimally doped single crystal material (i.e., no grain boundaries), which has an unchanged $T_c$ under hydrostatic pressure, in order to elucidate the contributions of flux pinning to $J_c$ enhancement. An important secondary motivation was to investigate further the possible contributions of $N_p$ and the pinning force strength to strong pinning.

Flux pinning has been a topic of much interest in field of superconductivity because of its importance for applications and fundamental aspects. This interest stems from the significance of flux pinning for high critical current density ($J_c$) in superconductors, which is the defining property of a superconductor. Generally, various types of random imperfections, such as cold-work-induced dislocations, secondary-phase precipitates, defects induced by high energy ion irradiation, etc., can be used to enhance flux pinning. Unfortunately, it is difficult to discern the maximum potential of a superconductor from these techniques, and the outcomes hold up only to a certain level. Furthermore, the principal result of hysteresis studies is that the critical current is only enhanced, in most of cases, either in low or high fields, with degradation of the superconducting critical temperature ($T_c$) another
drawback. For instance, proton irradiation can only enhance flux pinning in high fields by inducing point defects in Ba122:K. Similarly, light ion C$^{4+}$ irradiation of Ba122:Ni crystals can only enhance $J_c$ in low fields at high temperatures [3]. High energy particle irradiation can also decrease $T_c$ by more than 5 K for cobalt and nickel doped Ba-122 [4, 5].

As is well known, $J_c$ is mostly limited by weak links (in the case of polycrystalline bulks), and thermally activated flux creep (an intrinsic property) emerges from weak pinning [6]. Strong pinning can be achieved either by i) inducing new effective pinning centres, i.e., increasing the number density of pinning centres ($N_p$), or the vortex density, or ii) improving the pinning force.

The argument in detail is as follows: i) It is widely believed that hydrostatic pressure can induce pinning centres, which, in turn, enhance the pinning force. The total pinning force ($F_p$) and the pinning centres are correlated by $F_p = N_p f_p$, where $N_p$ is the number density of pinning centres and $f_p$ is the elementary pinning force, which is the maximum pinning strength of an individual pinning centre, with a value that depends on the interaction of the flux line with the defect. According to the conventional theory, strongly interacting defects can contribute to $F_p$ individually, provided that $F_p \propto N_p$, and weakly interacting defects can contribute only collectively; the collective theory leads to $F_p \propto (N_p)^2$ for small defect numbers [7].

ii) Secondly, an important contribution to the accumulated pinning force originates from the pinning strengths of individual pinning centres. The ability of a pin to trap a vortex depends on its location inside a material: a pin close to the centre traps more vortices than the same pin near the boundary, because the pinning strength of a pin is higher when it is near the centre [15]. Hydrostatic pressure can enhance the pinning strength of an individual pin by pushing it towards the sample’s centre. If vortices are
trapped by a strong potential, however, they can merge, forming a giant vortex, because repulsive vortex-vortex interaction usually vanishes at a very small distances when vortex cores strongly overlap [8]. The evidence for such vortex mergers can also be found elsewhere [9].

The discovery of iron based superconductors (FeSCs) has prompted many research groups to investigate their fundamental properties and also assess the suitability of these materials for applications. Generally, the Ba122:K compounds among the FeSCs seem to be the most technologically suitable because of their isotropic nature and high $T_c$, upper critical field ($H_{c2}$), and $J_c$ values ($J_c > 10^6$ A/cm$^2$ at 2 K and 0 T), and because they have possible room for flux pinning enhancement [10-14]. The depairing current density ($J_d$) is the maximum current density that superconducting electrons can support before de-pairing of Cooper pairs, and is given as

$$J_d = \frac{\Phi_0}{3\sqrt{3}\pi\mu_0\lambda^2\xi} \quad (1)$$

The $J_d$ value that is found is roughly 0.3 GA/cm$^2$ by using values of penetration depth, $\lambda = 105$ nm and coherence length, $\xi = 2.7$ nm [15, 16]. Our estimate indicates that there is a significant potential to further enhance flux pinning in (Ba,K)Fe$_2$As$_2$. Additionally, unchanged $T_c$ under hydrostatic pressure has been found for optimally doped (Ba,K)Fe$_2$As$_2$ [26]. Therefore, this is an ideal sample to use for investigating flux pinning by identifying the possible contributions of $N_p$ and the pinning force.

In this chapter, we extensively investigate the flux pinning of optimally doped (Ba,K)Fe$_2$As$_2$ under hydrostatic pressure. Strong pinning and therefore, critical current can be obtained by improving the pinning force strength. As a consequence, there are two significant implications that are beneficial to applications: i) There is strong pinning over a wide range of field, showing weak field dependency, and ii) $J_c$ enhancement occurs to the same extent in low and high fields. Under pressure, a
larger pinning force appears in (Ba,K)Fe$_2$As$_2$ crystal due to the increased pinning centre number density and the emergence of giant vortexes which show high pinning potentials. Our work is one of the very rare investigations where a nearly complete quantitative understanding of the flux pinning contributions to $J_c$ enhancement has been developed.

6.2 Results and Discussion

Figure 1 shows the temperature dependence of the magnetic moments for zero-field-cooled (ZFC) and field-cooled (FC) measurements at different pressures. $T_c$ remains almost unchanged at different pressures. $T_c \approx 37.95$ K was found at $P = 0$ GPa whereas, $T_c \approx 38.08$ K at $P = 1$ GPa. Similar results were also reported for Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ thin film [17]. Furthermore, a temperature independent magnetic moment at low temperatures was observed, along-with a small transition width, indicating the high quality of the crystals.

Figure 6-1: Magnetic moments versus temperature at $P = 0$ GPa and $P = 1.2$ GPa
The $M$-$H$ loops at different pressures and temperatures (4.1, 8, 12, 16, 20, 24, 28, and 32 K) can be seen in Figure 2. Larger $M$-$H$ Loops are observed at high pressure. For instance, the larger $M$-$H$ loops found at 8 K (Figure 3) for $P = 0.75$ and 1.2 GPa show significant enhancement of flux pinning in both low and high fields. The field dependence of $J_c$ at different temperatures (4.1, 16, and 24 K) and pressures (0 and 1.2 GPa), obtained from the $M$-$H$ curves by using Bean’s model, are shown in Figure 4. Nearly two-fold and five-fold $J_c$ enhancement can be seen at 16 K and 24 K, respectively. It is noteworthy that $J_c$ is enhanced for Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystal under pressure of 1.2 GPa in both low and high fields, which is not generally found with other approaches. At 16 K and self-field, the $J_c$ is remarkably high, approaching nearly 1 MA/cm$^2$, whereas $J_c \approx 2 \times 10^5$ A/cm$^2$ was obtained at 24 K and 12 T.

Figure 6-2: $M$-$H$ loops at 4.1, 8, 12, 16, 20, 24, 28, and 32 K for $P = 0$, 0.75, and 1.2 GPa.
Pressure can also improve the pinning force strength by a significant amount in Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ single crystal. The pinning force ($F_p = J_c \times B$) as a function of field at 8 K, 12 K, 24 K, and 28 K is plotted in Figure 5. At high fields and pressures, the $F_p$ is found to be nearly 5 times higher at 8, 12, 24, and 28 K as compared to $P = 0$ GPa, which corresponds nicely to $J_c$ enhancement. Figure 6 shows a comparison of $F_p$ obtained in our Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ under pressure with those of several other low and high temperature superconducting materials [10, 18-20]. The (Ba,K)Fe$_2$As$_2$ shows better in-field performance under pressure. Pressure can improve $F_p$ values to greater than 60 GN/m$^3$ at $H > 10$ T, which are even superior to those of Nb$_3$Sn and NbTi.

Figure 6-3: $J_c$ as a function of field at 0 GPa and 1.2 GPa at 4.1 K, 16 K, and 24 K.
Figure 6-4: $F_p$ versus field at 8 K, 12 K, 24 K, and 28 K at different pressures.

Figure 6-5: $F_p$ for different superconductors.

With respect to the $N_p$, pressure can also increase the number of point pinning centres, which can suppress thermally activated flux creep, leading to $J_c$ enhancement [1].
\( N_p \) can be calculated from the following relation:

\[
\frac{\Sigma F_p}{\eta f_p^{max}} = N_p \quad (1)
\]

Where \( \Sigma F_p \) is the accumulated pinning force density, \( f_p^{max} \) is the maximum elementary pinning force \( (f_p) \), which is the interaction between a flux line and a single defect, and \( \eta \) is an efficiency factor. \( \eta = 1 \) corresponds to a plastic lattice, and the \( \eta \) value is otherwise \( f_p^{max}/B \), where \( B \) is the bulk modulus of the material. We assume to a second order of approximation that the interaction between a flux line and a single defect is nearly same under pressure. Therefore, we can use \( f_p^{max} \approx 3 \times 10^{-13} \) N for a similar superconductor (i.e., Ba122:Co) to estimate \( N_p \) [21]. At 4.1 K, \( N_p \approx 7.3 \times 10^{24} \) m\(^{-3} \) at \( P = 0 \) Gpa, which increased to \( N_p \approx 1.2 \times 10^{25} \) m\(^{-3} \) for \( P = 1.2 \) GPa, while at 24 K, \( N_p \approx 6.6 \times 10^{23} \) m\(^{-3} \) at \( P = 0 \) Gpa, which increased to \( N_p \approx 3.8 \times 10^{24}/\)m\(^{3} \) for \( P = 1.2 \) GPa.

Figure 6-6: \( J_c/F_p \) ratios at \( P = 1.2 \) GPa and \( P = 0 \) GPa. The relative change of \( J_c \) with pressure as a function of \( T \) is given in the inset.
In order to investigate the possible contributions to strong flux pinning, we have shown a preliminary result in Figure 6 which describes the field dependence of $J_c^R - F_p^R$ at $P = 0$ GPa and $P = 1.2$ GPa, where $J_c^R = J_c^{1.2GPa}/J_c^{0GPa}$ and $F_p^R = F_p^{1.2GPa}/F_p^{0GPa}$. Analysis of the $J_c^R - F_p^R$ data, acquired at different temperatures, leads to minimal values, i.e., nearly zero, which shows that that pressure can increase the pinning force by increasing the number density and the formation of giant vortexes, and in this way, it can increase the critical current in high and low fields. The simplest way to show $N_p$ and $F_p$ dependence on pressure is illustrated in Figure 7, which demonstrates that pressure can enhance flux pinning.

![Figure 6-7: $F_p \propto N_p$ holds where $P < P_0$ (the threshold pressure value) and the size of defects, $L_0 \approx \xi$.](image)

High pressure can modify grain boundaries through reduction of the tunnelling barrier width and the tunnelling barrier height for polycrystalline bulks. The
Wentzel-Kramers-Brillouin (WKB) approximation, which is valid for such a potential barrier gives the following simple mathematical expression [22-24]:

\[ J_c = J_{c0} \exp(-2kW) \]  

(1)

Where \( W \) is the barrier width, \( k = (2mL)^{1/2}/\hbar \) is the decay constant, which is barrier height \( (L) \) dependent, \( \hbar \) is the reduced Planck constant, and \( J_{c0} \) is the critical current density at 0 K and 0 T. The relative pressure dependence of \( J_c \) can be determined from Eq. (1) as [25]:

\[
\frac{d \ln J_c}{dP} = \frac{d \ln J_{c0}}{dP} - \left[ \left( \frac{d \ln W}{dP} \right) \ln \left( \frac{J_{c0}}{J_c} \right) \right] - \frac{1}{2} \left[ \left( \frac{d \ln L}{dP} \right) \ln \left( \frac{J_{c0}}{J_c} \right) \right]
\]

\[ = \frac{d \ln J_{c0}}{dP} + \kappa_{GB} \ln \left( \frac{J_{c0}}{J_c} \right) + \frac{1}{2} \kappa_L \ln \left( \frac{J_{c0}}{J_c} \right) \]  

(2)

Where the compressibility in the width and height of the grain boundary are defined by \( \kappa_{GB} = -\frac{d \ln W}{dP} \) and \( \kappa_L = -\frac{d \ln L}{dP} \), respectively.

For (Ba,K)Fe\(_2\)As\(_2\) single crystals, we assume to a first approximation that \( \kappa_{GB} \) and \( \kappa_L \) are nearly comparable to the average linear compressibility values \( \kappa_a = -\frac{d \ln a}{dP} \) (\( \kappa_a \approx 0.00318 \) GPa\(^{-1}\)) and \( \kappa_c = -\frac{d \ln c}{dP} \) (\( \kappa_c \approx 0.00622 \) GPa\(^{-1}\)), respectively, in the FeAs plane, where \( a \) and \( c \) are the in-plane and out-of-plane lattice parameters [26]. Therefore, we can write Eq. (2) correspondingly as

\[
\frac{d \ln J_c}{dP} \approx \frac{d \ln J_{c0}}{dP} + \kappa_a \ln \left( \frac{J_{c0}}{J_c} \right) + \frac{1}{2} \kappa_c \ln \left( \frac{J_{c0}}{J_c} \right)
\]

(3)

By using \( J_c \approx 10^5 \) A/cm\(^2\) at 24 K and \( J_{c0} \approx 10^6 \) A/cm\(^2\), we find that \( (\kappa_a \ln(J_{c0}/J_c)) \approx 0.0073 \) GPa\(^{-1}\) and \( (1/2\kappa_c \ln(J_{c0}/J_c)) \approx 0.0071 \) GPa\(^{-1}\), so both only contributed less than 2% to the experimental value, i.e., \( d \ln J_c/dP = 0.92 \) GPa\(^{-1}\) from the inset of Figure 6. This estimate indicates that the origin of the strong flux pinning under pressure does not arise from the change in volume.
The $J_c$ value as a function of reduced temperature (i.e. $1-T/T_c$) at 0 and 10 T under different pressures is shown in Figure 8. The experimental data points in different fields and pressures follow a power law description [i.e. $J_c \propto (1-T/T_c)^\beta$], where $\beta$ is a critical exponent [27, 28]. Ginzburg-Landau theory predicts different vortex pinning mechanisms at specified fields, with different values of exponent $\beta$. It was found that $\beta = 1$ corresponds to non-interacting vortices and $\beta \geq 1.5$ refers to the core pinning mechanism. The exponent $\beta$ (i.e., slope of the fitting line) is found to be 1.74 and 1.85 for zero field, and 1.20 and 1.43 at 10 T, at 0 and 1.2 GPa, respectively, which reveals a strong $J_c$ dependence on pressure. The low values of $\beta$ at high pressure show that the $J_c$ decays rather slowly in comparison to its values at low pressure. Different values of exponent $\beta$ have also been observed in MgB$_2$ and yttrium barium copper oxide (YBCO) [29, 30].

![Graph](image)

*Figure 6-8: ln$J_c$ versus reduced temperature at different fields and pressures.*

The pinning mechanisms in Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ have been analysed by using collective pinning theory. Generally, core pinning comprises 1) $\delta\ell$ pinning, which comes from...
spatial variation in the charge carrier mean free path, $\ell$, and 2) $\delta T_c$ pinning due to randomly distributed spatial variation in $T_c$.

Figure 6-9: $J_c$ as a function of $T/T_c$. Experimental data points are in agreement with $\delta \ell$ pinning.

Referring to the theoretical approach proposed by Griessen et al.:

$$\frac{J_c(t)}{J_c(0)} \propto (1 - t^2)^{5/2} (1 + t^2)^{-1/2} \quad (2)$$

in the case of $\delta \ell$ pinning, while

$$\frac{J_c(t)}{J_c(0)} \propto (1 - t^2)^{7/6} (1 + t^2)^{5/6} \quad (3)$$

applies in the case of $\delta T_c$ pinning, where $t = \frac{T}{T_c}$. Figure 8 shows almost perfect overlapping of the experimentally obtained $J_c$ values and the theoretically expected variation for the $\delta \ell$ pinning mechanism at 0.1 T and 0 T (with the so-called remanent state shown by the solid symbols). The $J_c(t)$ values have been found from the $J_c(B)$ curves at different temperatures. The experimental data seems to be in good agreement with the theoretical $\delta \ell$ pinning curves at $P = 0$ and $P = 1$ GPa. Therefore,
pinning in Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ emerges from spatial variation of the mean free path $\ell$. We also observed similar results in BaFe$_{1.9}$Ni$_{0.1}$As$_2$ and SiCl$_4$ doped MgB$_2$ [31, 32]. Furthermore, $\delta\ell$ pinning has also been reported in FeTe$_{0.7}$Se$_{0.3}$ crystals [33].

6.3 Conclusion

We have studied the flux pinning in optimally doped Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystal under hydrostatic pressure, analysing the critical current density determined experimentally. We propose that strong flux pinning in a wide range of fields can be achieved by improving the pinning force under pressure. The pressure of 1.2 GPa improved the $F_p$ by nearly 5 times at 8, 12, 24, and 28 K, which can increase $J_c$ by nearly two-fold at 4.1 K and five-fold at 16 K and 24 K in both low and high fields, respectively. This study also demonstrates that such an optimally doped superconductor’s performance in both low and high fields can also be further enhanced by pressure.
6.4 Experimental

A High quality 122 crystals were grown by using a flux method. The pure elements Ba, K, Fe, As, and Sn were mixed in a mol ratio of $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2\text{Sn} = 1:45–50$ for the self-flux. A crucible with a lid was used to reduce the evaporation loss of K as well as that of As during growth. The crucible was sealed in a quartz ampoule filled with Ar and loaded into a box furnace [11]. The temperature dependence of the magnetic moments and the $M-H$ loops at different temperatures and pressures were measured on a Quantum Design Physical Property Measurement System (QD PPMS 14 T) by using the Vibrating Sample Magnetometer (VSM). We used an HMD high pressure cell and Daphne 7373 oil as a pressure transmitting medium to apply hydrostatic pressure on the samples.
6.5 References


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HYDROSTATIC PRESSURE INDUCED TRANSITION FROM $\delta T_c$ TO $\delta \ell$ PINNING MECHANISM IN MgB$_2$

7.1 Introduction

To generalize our hydrostatic pressure technique to other superconducting families, we have investigated the impact of hydrostatic pressure on $J_c$ and flux pinning mechanisms in MgB$_2$. Previous studies have shown that pressure of 1 GPa can reduce $T_c$, but only by less than 2 K in MgB$_2$ [1]. This is a very insignificant reduction as compared to the other approaches (i.e. chemical doping and irradiation) which are mainly used for $J_c$ enhancement [2]. For instance, chemical doping can significantly enhance $J_c$ in MgB$_2$, but with a considerable degradation of $T_c$; carbon doping can reduce $T_c$ from 39 K to nearly as low as 10 K, for carbon content up to 20% [3-8]. Similarly, the irradiation method can improve $J_c$ in MgB$_2$, but it reduces $T_c$ values significantly (by more than 20 K in some cases) [9-13]. Correspondingly, the chemical doping and irradiation methods can also change the nature of the pinning mechanism in MgB$_2$ [10, 14-16]. The determination of $J_c$ and the flux pinning mechanism under hydrostatic pressure is also an important step to probe the mechanism of superconductivity in more detail in MgB$_2$. It is very interesting to know whether hydrostatic pressure can increase the pinning and $J_c$ at both low and high fields.

Magnesium diboride (MgB$_2$) is a promising superconducting material which can replace conventional low critical temperature ($T_c$) superconductors in practical applications, due to its relatively high $T_c$ of 39 K, strongly linked grains, rich multiple band structure, low fabrication cost, and especially, its high critical current density ($J_c$) values of $10^5$-$10^6$ A/cm$^2$ [17-24]. Numerous studies have been carried out in order to understand the vortex-pinning mechanisms in more detail, which have led
to real progress regarding the improvement of $J_c$. There are two predominant mechanisms, $\delta T_c$ pinning, which is associated with spatial fluctuations of the $T_c$, and $\delta \ell$ pinning, associated with charge carrier mean free path ($\ell$) fluctuations [25-29]. In this chapter, we report our study on pressure effects on $T_c$, $J_c$, and the flux pinning mechanism in MgB$_2$. Hydrostatic pressure can induce a transition from the regime where pinning is controlled by spatial variation in the critical transition temperature ($\delta T_c$) to the regime controlled by spatial variation in the mean free path ($\delta \ell$). In addition, $T_c$ and low field $J_c$ are slightly reduced, although the $J_c$ drops more quickly at high fields than at ambient pressure. We found that the pressure increases the anisotropy and reduces the coherence length, resulting in weak interaction of the vortex cores with the pinning centres.

### 7.2 Results and Discussion

![XRD Pattern of MgB$_2$](image)

Figure 7-1: XRD Pattern of MgB$_2$.

Figure 7-1 illustrates XRD patterns for the MgB$_2$ sample. The peaks show that MgB$_2$ sample is a single phase. The zero-field-cooling (ZFC) and field-cooling (FC) curves at different applied pressures are plotted in figure 1. The $T_c$ drops from 39.7 K at $P = 0$ GPa to 37.7 K at $P = 1.2$ GPa, with a pressure coefficient of -1.37 K/GPa, as can be
seen in the inset of figure 1. It is well known that \( T_c \), the unit cell volume \( (V) \), and the anisotropy \( (\gamma) \) under pressure can be interrelated through a mathematical relation as in[30]

\[
\Delta T'_c(P) + \Delta V' + \Delta \gamma' = 0 \quad \text{………(1)}
\]

where \( \Delta T'_c(P) = \left[ \frac{T_c(P) - T_c(0)}{T_c(0)} \right] \), \( \Delta V' = \left[ \frac{V(P) - V(0)}{V(0)} \right] \) and \( \Delta \gamma' = \left[ \frac{\gamma(P) - \gamma(0)}{\gamma(0)} \right] \).

The \( \Delta V' \) found for MgB\(_2\) is 0.0065, as the pressure can reduce the unit cell volume of MgB\(_2\) from 29.0391 Å\(^3\) at \( P = 0 \) GPa to 28.8494 Å\(^3\) at \( P \approx 1.2 \) GPa [31]. A similar value for \( \Delta V' \) can also be obtained from \( \Delta V' = -\Delta P / B \), where \( B \) is the bulk modulus of the material [30]. We found \( \Delta T'_c(P) = 0.042 \) from figure 1. By using \( \Delta V' \) and \( \Delta T'_c(P) \), we can obtain from Equation (1):

\[
\Delta \gamma' = \left[ \frac{\gamma(P) - \gamma(0)}{\gamma(0)} \right] \approx 0.036 \quad \text{………(2)}
\]

Figure 7-2: Temperature dependence of magnetic moment under different applied pressures in both ZFC and FC runs. The inset shows the pressure dependence of the \( T_c \) for MgB\(_2\). \( T_c \) is found to decrease with a slope of \( dT_c/dP = -1.37 \) K/GPa.

This indicates that the anisotropy of MgB\(_2\) is increased by applying pressure, i.e., \( \gamma(P) > \gamma(0) \). Therefore, the coherence length \( (\xi) \) at \( P = 1.2 \) GPa is reduced as compared to its value at \( P = 0 \) GPa [i.e., \( (\xi)_P < (\xi)_0 \)]. The density of states in
Bardeen-Cooper-Schrieffer (BCS)-like superconductors such as MgB$_2$ is expressed as

$$N_s(E) = N_n(E_F) \left[ \frac{E}{\sqrt{E^2 - \Delta^2}} \right] \ldots \ldots \ldots (3)$$

Where $N_n(E_F)$ is the density of states at the Fermi level in the normal state and $\Delta$ is the superconductivity gap. Therefore, $N_s(E) \propto N_n(E_F)$ and

$$N_n(E_F) \propto V E_F^{1/2} \propto V k_F^2 \ldots \ldots \ldots (4)$$

where $V$ is the total volume and $k_F$ is the Fermi wave vector [32, 33],

$$k_F = \frac{2m\Delta \xi}{\hbar} \ldots \ldots \ldots (5)$$

Combining Equations (3), (4), and (5), we obtain

$$N_s(E) \propto V \xi \ldots \ldots \ldots (6)$$

It is important to mention that pressure has no significant impact on the unit cell volume of MgB$_2$ up to $P = 1.2$ GPa. Therefore, the density of states is mainly dependent on $\xi$. $(\xi)_P < (\xi)_0$ leads to a comparison regarding the density of states at $P = 1.2$ GPa and $P = 0$ GPa

$$i.e. [N_s(E)]_P < [N_s(E)]_0 \ldots \ldots \ldots (7)$$

given that hydrostatic pressure can decrease the density of states in MgB$_2$ and therefore contributes to a reduction in $T_c$. 

Figure 7-3: Field dependence of $J_c$ under different pressures measured at 5 K, 8 K, 20 K, and 25 K.
Figure 7-4: Comparison of $J_c$ at two pressures (0 GPa and 1.2 GPa) for 8 K and 20 K curves. The inset shows the plot of $\Delta J_c / J_{c0}$ versus field, illustrating the trend towards the suppression of $J_c$ with increasing field, nearly at a same rate of $-0.97 \, T^{-1}$ for 8 K and 20 K.

Figure 2 shows the field dependence of $J_c$ at different temperatures (i.e. 5, 8, 20, and 25 K) and pressures (i.e. 0, 0.7, and 1.2 GPa). We found that low field $J_c$ was reduced slightly under pressure. The $J_c$ drops more quickly at high fields, however, as compared to $P = 0$ GPa. This is further reflected in figure 3, which shows $J_c$ values at 8 and 20 K under pressure. The inset shows normalized $\Delta J_c$ (i.e., $\Delta J_c = J_c^P - J_c^0$) for both 8 K and 20 K, which indicates almost a similar decay trend. We also plotted irreversibility field ($H_{irr}$) as a function of temperature in figure 4, which shows that $H_{irr}$ decreases gradually from nearly 13 T to 11.8 T at $T = 5$ K for $P = 1.2$ GPa, which is ascribed to the observed $J_c$ suppression.
$J_c$ as a function of reduced temperature ($τ = 1 - T/T_c$, where $T$ is the temperature and $T_c$ is the critical temperature) is plotted in figure 5. The temperature dependence of $J_c$ follows a power law description in the form of $J_c \propto τ^μ$, where $μ$ is the slope of the fitted line and its value depends on the magnetic field [34-36]. The exponent $μ$ in our case is found to be nearly same at different pressures, and its values are 1.63, 2.22, and 2.65 at fields of 0, 2.5, and 5 T, respectively. Different values of exponent $μ = 1, 1.7, 2,$ and 2.5 are also reported for standard yttrium barium copper oxide (YBCO) films [37]. The larger exponent value at high field shows that pressure effects are more significant at high fields as compared to low fields.

Figure 7-6: $J_c$ as a function of reduced temperature ($τ = 1 - T/T_c$) at 0, 2.5, and 5 T for pressures of 0, 0.7, and 1.2 GPa. The solid lines are fitted well to the data according to the power law in the framework of Ginzburg-Landau theory.
A double logarithmic plot of $-\log\left[\frac{J_c(B)}{J_c(0)}\right]$ as a function of field at 12 K and 20 K for $P = 0$ GPa and $P = 1.2$ GPa is plotted in figure 6. This shows deviations at certain fields, denoted as $B_{SB}$ and $B_{th}$. According to the collective theory [10] the region below $B_{SB}$ is the regime where the single-vortex-pinning mechanism governs the vortex lattice in accordance with the following expression,

$$B_{SB} \propto J_{sv} B_{c2} \quad \ldots \ldots (8)$$

Where, $J_{sv}$ is the critical current density in the single vortex pinning regime and $B_{c2}$ is the upper critical field. At high fields (above the crossover field $B_{SB}$), $J_c(B)$ follows an exponential law

$$J_c(B) \approx J_c(0) \exp\{-\left(\frac{B}{B_0}\right)^{3/2}\} \quad \ldots \ldots (9)$$

Where, $B_0$ represents a normalization parameter on the order of $B_{SB}$. It is well known that the deviation observed at $B_{SB}$ is linked to the crossover from the single-vortex-pinning regime to the small-bundle-pinning regime, while the deviation at the thermal crossover field ($B_{th}$) can be connected to large thermal fluctuations [24]. The pinning behaviour can be obtained from the temperature dependence of the crossover.
field from the single vortex regime [38]. The temperature dependence of the crossover field can be expressed as

\[ B_{SB}(T) = B_{SB}(0) \left( \frac{1 - t^2}{1 + t^2} \right)^v \]  

(10)

Where \( v = 2/3 \) and 2 for \( \delta T_c \) and \( \delta \ell \), respectively.

The above-mentioned Equation (10) can be found by inserting the following expressions with \( t = T/T_c \) into Equation (8),

\[ J_{sv} \approx (1 - t^2)^{7/6} (1 + t^2)^{5/6} \text{ : for } \delta T_c \]  

(11)

and \( J_{sv} \approx (1 - t^2)^{5/2} (1 + t^2)^{-1/2} \text{ : for } \delta \ell \)  

(12)

Figure 7-8: Plots of \( B_{SB}(T)/B_{SB}(0) \) vs. \( T/T_c \) at different pressures (0, 0.7, and 1.2 GPa). The red fitted line is for \( \delta T_c \) pinning, the black fitted line is for \( \delta \ell \) pinning, and the green fitted line is for mixed \( \delta (T_c + \ell) \) pinning.
The crossover fields ($B_{SB}$) for reduced temperature ($T/T_c$) at $P = 0$ GPa, 0.7 GPa, and 1.2 GPa are plotted in figure 7. The experimental data points for $B_{SB}$ are scaled through Eq. (10) for $\delta\ell$ and $\delta T_c$. We found that hydrostatic pressure can induce the transition from the $\delta T_c$ to the $\delta\ell$ pinning mechanism. The $\delta T_c$ pinning mechanism is dominant in pure MgB$_2$ polycrystalline bulks, thin films, and single crystals [29, 39, 40]. The coherence length is proportional to the mean free path ($\ell$) of the carriers, and therefore, pressure can enhance $\delta\ell$ pinning in MgB$_2$. It is noteworthy that $J_c$ drops under pressure in MgB$_2$ due to the transition in the flux pinning mechanism.

### 7.3 Conclusion

In summary, the impact of hydrostatic pressure on the $J_c$ and the nature of the pinning mechanism in MgB$_2$, based on the collective theory, have been investigated. We found that the hydrostatic pressure can induce a transition from the $\delta T_c$ to the $\delta\ell$ pinning mechanism. Furthermore, pressure can slightly reduce low field $J_c$ and $T_c$, although pressure has a more pronounced effect on $J_c$ at high fields. Moreover, the pressure can also increase the anisotropy, along with causing reductions in the coherence length and $H_{irr}$, which, in turn, leads to a weak pinning interaction.
7.4 Experimental

The MgB₂ bulk sample used in the present work was prepared by the diffusion method. Firstly, crystalline boron powders (99.999%) with particle size of 0.2-2.4 µm were pressed into pellets. They were then put into iron tubes filled with Mg powder (325 mesh, 99%), and the iron tubes were sealed at both ends. Allowing for the loss of Mg during sintering, the atomic ratio between Mg and B was 1.2:2. The sample was sintered at 800°C for 10 h in a quartz tube under flowing high purity argon gas. Then, the sample was furnace cooled to room temperature. The size of bar shaped sample used for measurements is 3×2×1 mm³. The temperature dependence of the magnetic moments and the M-H loops at different temperatures and pressures were performed on Quantum Design Physical Property Measurement System (PPMS 14T) by using vibrating sample magnetometer (VSM). We used a Quantum Design High Pressure Cell with Daphne 7373 oil as a pressure transmission medium to apply hydrostatic pressure on a sample. The $J_c$ was calculated by using the Bean approximation.
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CONCLUSION

Hydrostatic pressure is a very effective approach to significantly enhance $J_c$ at both low and high fields in FeSCs. It also enhances $T_c$, $H_{irr}$, $H_{c2}$, and pinning number density by inducing more point defects, grain connectivity (in case of polycrystalline bulks and wires/tapes) and reduce the anisotropy. We have used this approach on polycrystalline bulks, single crystal, tape and MgB$_2$. Details are as follows;

A. We found that the hydrostatic pressure up to 1.2 GPa can not only significantly increase $T_c$ from 15 K (underdoped) to 22 K, but also significantly enhance the irreversibility field, $H_{irr}$, by a factor of 4 at 7 K, as well as the critical current density, $J_c$, by up to 30 times at both low and high fields for Sr$_4$V$_2$O$_6$Fe$_2$As$_2$ polycrystalline bulks. It was found that pressure can induce more point defects, which are mainly responsible for the $J_c$ enhancement. In addition, pressure can also transform surface pinning to point pinning in Sr$_4$V$_2$O$_6$Fe$_2$As$_2$.

B. Hydrostatic pressure can significantly enhance $J_c$ in NaFe$_{0.972}$Co$_{0.028}$As single crystals by at least tenfold at low field and more than a hundredfold at high fields. Significant enhancement in the in-field performance of NaFe$_{0.972}$Co$_{0.028}$As single crystal in terms of pinning force density ($F_p$) is found at high pressures. At high fields, the $F_p$ is over 20 and 80 times higher than under ambient pressure at 12K and 14K, respectively, at P=1GPa. We have discovered that the hydrostatic pressure induces more point pinning centres in a
sample, and the pinning centre number density found at 1GPa is almost twice as high as under ambient pressure at 12K and over six times as high at 14K.

C. We have studied the flux pinning in optimally doped Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ crystal under hydrostatic pressure, analysing the critical current density determined experimentally. We propose that strong flux pinning in a wide range of fields can be achieved by improving the pinning force under pressure. The pressure of 1.2 GPa improved the $F_p$ by nearly 5 times at 8, 12, 24, and 28 K, which can increase $J_c$ by nearly two-fold at 4.1 K and five-fold at 16 K and 24 K in both low and high fields, respectively. This study also demonstrates that such an optimally doped superconductor’s performance in both low and high fields can also be further enhanced by pressure.

D. The impact of hydrostatic pressure up to 1.2 GPa on the $J_c$ and the nature of the pinning mechanism in MgB$_2$ have been investigated within the framework of the collective theory. We found that the hydrostatic pressure can induce a transition from the regime where pinning is controlled by spatial variation in the critical transition temperature ($\delta T_c$) to the regime controlled by spatial variation in the mean free path ($\delta \ell$). Furthermore, $T_c$ and low field $J_c$ are slightly reduced, although the $J_c$ drops more quickly at high fields than at ambient pressure. The hydrostatic pressure can reduce the density of states [$N_s(E)$], which, in turn, leads to a reduction in the critical temperature from 39.7 K at P = 0 GPa to 37.7 K at P = 1.2 GPa.
PUBLICATION FROM THIS WORK


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